RCRA FACILITY INVESTIGATION (RFI)
WORK PLAN

GM POWERTRAIN BEDFORD FACILITY
105 GM DRIVE
BEDFORD, INDIANA

EPA ID# IND006036099

Volume I – Text, Figures, Tables, Appendix A, and Appendix B

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LIST OF ACRONYMS AND TERMS

Agreement:	Voluntary Corrective Action Agreement
AOIs:	Areas of Interest
AST:	Above Ground Storage Tank
Bedford Facility:	GM Powertrain Bedford Plant
CCR:	Current Conditions Report
CECOS:	CECOS International, Inc.
CRA:	Conestoga-Rovers & Associates
CRP:	Community Relations Plan
DMP:	Data Management Plan
FSP:	Field Sampling Plan
GM:	General Motors
HASP:	Health and Safety Plan
IDEM:	Indiana Department of Environmental Management
ISBH:	Indiana State Board of Health
ISPCB:	Indiana State Pollution Control Board
MPC:	Minerals Processing Corporation
NAPL:	Non-aqueous Phase Liquid
NPDES:	National Pollutant Discharge Elimination System
PCB:	Polychlorinated Biphenyls
PID:	Photoionization Detector
PMP:	Project Management Plan
POTW:	Publicly Owned Treatment Works
QA/QC:	Quality Assurance/Quality Control
QAPP:	Quality Assurance Project Plan
RCRA:	Resource Conversation and Recovery Act
RFI:	RCRA Facility Investigation
SVOC:	Semi-volatile Organic Compound
TAL:	Target Analyte List
TCL:	Target Compound List
TOC:	Total Organic Carbon
USDA:	United States Department of Agriculture
U.S.EPA:	United States Environmental Protection Agency
USGS:	United States Geological Survey
UST:	Underground Storage Tank
VOC:	Volatile Organic Compound
WWTP:	Wastewater Treatment Plant

LIST OF ACRONYMS AND TERMS

UNITS OF MEASURE

amsl:	above mean sea level
bgs:	below ground surface
cfs:	cubic feet per second
cm:	centimeter
°F:	degree Fahrenheit
gpd:	gallons per day
mgd:	million gallons per day

1.0 INTRODUCTION

This report presents a work plan for the Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) at the General Motors Corporation (GM) Powertrain Bedford Plant (Bedford Facility) in Bedford, Indiana (EPA ID# IND006036099).

1.1 GENERAL

Conestoga-Rovers & Associates (CRA) has been retained by GM to prepare this document entitled "RCRA Facility Investigation (RFI) Work Plan for GM Powertrain Bedford Plant (RFI Work Plan)".

The GM Powertrain Bedford Plant is located at 105 GM Drive, Bedford (Lawrence County), IN 47421 (Figure 1.1). The facility produces aluminum casting products, such as transmission cases, pistons, and engine blocks. Major aluminum production processes include die casting & permanent molding. The Bedford Facility has been operating as an aluminum foundry since 1942, with major facility modifications completed in 1950, 1953, 1966, 1971, 1974, 1977, 1979, and 1980.

The plant, located on 152.5 acres, contains approximately 915,000 square feet of floor space and employs approximately 1,200 people.

1.2 RFI APPROACH

General Motors signed a Voluntary Corrective Action Agreement (Agreement) with the United States Environmental Protection Agency (U.S.EPA) for the Bedford Facility on March 20, 2001. The signed Agreement states that GM will work voluntarily (with the U.S. EPA) to identify and define the nature and extent of releases of hazardous waste and/or hazardous constituents at or from the Bedford Facility.

To meet these requirements, GM will investigate those Areas of Interest (AOIs) that may be associated with a release of hazardous waste and/or hazardous constituents. These AOIs were identified in the Current Conditions Report (CCR) (CRA, May 25, 2001). Table 1.1 lists the AOIs at the Facility and identifies the investigation, if any, to be performed at each AOI.

In addition, GM is also currently evaluating the potential for off-site impact from Facility activities. These activities were previously detailed in two separate Preliminary Investigation Work Plans.

1.3 RFI GOALS AND OBJECTIVES

The overall goal of the RFI is to characterize the nature and extent of any releases of hazardous waste and/or hazardous constituents at or from the Facility which may pose an unacceptable risk to human health and the environment. The results of the RFI will be used to demonstrate that: (1) all current human exposures to media contaminated with hazardous waste and/or hazardous constituents above acceptable risk-based levels are under control, and (2) migration of groundwater contaminated with hazardous waste and/or hazardous constituents above acceptable levels is stabilized within any existing areas of contamination, and any discharge of contaminated groundwater to surface water is either insignificant or shown to be currently acceptable. This demonstration will be documented in an Environmental Indicators Report. The results of the RFI will be used to support the selection of final Corrective Measures necessary to protect human health and the environment from all current and future unacceptable risks, if any, due to releases of hazardous waste and/or hazardous constituents at or from the Facility.

To meet these goals, the objective of the RFI is to collect data of sufficient quality and quantity to support an assessment of potential current and future risks to human health and the environment associated with releases of hazardous waste and/or hazardous constituents. Specifically, the RFI will:

- Determine whether a release of hazardous waste/constituents to soil, groundwater, surface water, or sediment has occurred at AOIs identified in the CCR as requiring investigation;
- Characterize the source(s) of a release and determine the nature and extent of constituents in environmental media to the extent necessary to protect human health and the environment;
- Characterize actual and potential migration pathways, actual and potential human and environmental receptors, and current and reasonably expected future land and groundwater uses;

- Assess potential risk to human health and the environment associated with any significant releases of hazardous waste/constituents;
- Determine whether interim corrective measures are necessary to control current unacceptable human exposures, or to control migration of contaminated groundwater;
- Provide sufficient data to support a demonstration that current human exposures to contamination above risk-based levels are under control, and that the migration of groundwater contaminated above acceptable levels is stabilized; and
- Determine if final corrective measures are necessary to mitigate all current and future unacceptable risks, if any, to human health and the environment.

1.4 WORK PLAN ORGANIZATION

Though not included in this RFI Work Plan, two preliminary RFI activities are underway under separate Work Plans (Off-Site Well Survey and Sampling Work Plan and Stream Investigation Work Plan). These Work Plans are part of the overall RFI, but are not repeated in this Work Plan.

The RFI Work Plan is consistent with the U.S.EPA guidance. The RFI Work Plan is organized as follows:

Section 1.0 Introduction

This section presents an introduction to, and organization of, the RFI Work Plan.

Section 2.0 Description of Facility

This section summarizes general conditions at the Bedford Facility, including the Facility location, current and historical operations at the Bedford Facility, and current and historical waste management practices at the Bedford Facility (including historical RCRA regulatory activities).

Section 3.0 Regional Setting

This section summarizes local land use, climate, geology, hydrogeology, surface water hydrology, and ecology.

Section 4.0 Facility Setting

This section summarizes currently known geology, hydrogeology, and surface water hydrology within the vicinity of the Bedford Facility.

Section 5.0 Sampling and Analysis Plan

This section presents the current status of each AOI at the Facility, and describes the proposed investigation, if any, for each AOI.

Section 6.0 Data Evaluation

This section describes the proposed method for evaluating the RFI data. This will consist of initial data analysis, review of exposure information, and post-investigation evaluation.

Section 7.0 Reports

This section outlines the reporting requirements associated with the RFI, including progress reports, the RFI Report, and the Environmental Indicators Reports.

Section 8.0 RFI Schedule

This section presents the RFI schedule.

Section 9.0 References

This section presents a list of references used in the preparation of this RFI Work Plan. The background information in this RFI Work Plan has been taken from the Current Conditions Report (CRA, May 25, 2001), and therefore the reference list from that report has not been reproduced.

This document also includes the following appendices:

Appendix A Project Management Plan (PMP)

The PMP presents a discussion of the project team organization and the responsibilities of the project team members.

Appendix B Field Sampling Plan (FSP)

The FSP presents standard operating procedures for the collection of surface soil, subsurface soil, sediment, and groundwater samples.

Appendix C Quality Assurance Project Plan (QAPP)

Appendix C presents the RFI QAPP and reflects the activities outlined in this Work Plan and presents organization, objectives, planned activities and specific quality assurance/quality control (QA/QC) procedures, which will be utilized during implementation of the RFI Work Plan.

Appendix D Data Management Plan (DMP)

The DMP presents procedures to be employed for managing information, reports, and correspondence associated with the implementation of the RFI Work Plan.

Appendix E Site Health and Safety Plan (HASP)

The HASP presents the minimum health and safety standards to be met by all personnel during implementation of the RFI Work Plan.

Appendix F Community Relations Plan (CRP)

The CRP presents the mechanisms for dissemination of information to the public regarding investigation activities, results, and selection of a remedy. The CRP was submitted as a separate document on June 6, 2001, and is also included here.

1.5 INVESTIGATION SUMMARY

Table 1.1 presents a summary of the investigation plan for the AOIs, which is presented in greater detail in Section 5.0.

2.0 DESCRIPTION OF FACILITY

2.1 CURRENT OPERATIONS

Currently, the Bedford Facility operates within the GM Powertrain Division and produces aluminum casting products such as transmission casings, pistons, and engine blocks. Major aluminum production processes include die-casting and permanent molding. Descriptions of the manufacturing processes are summarized and additional information regarding the operational history of the Bedford Facility are provided in the Current Conditions Report dated May 25, 2001.

2.2 HISTORICAL OPERATIONS

The property was initially developed as a limestone-cutting mill circa 1890. Several mills once occupied this property (Struble, Walters, and Salem). Until the early 1940s, the property operated as a mill, cutting large limestone blocks for fine building stone.

Railroad maintenance shops and a turn-around yard (roundhouse) were built in the 1880's by the Evansville-Richmond Railroad. The railroad maintenance shops and roundhouse (AOI 1) were located to the south of the stone mills in the southern portion of the current Bedford Facility.

In 1942, GM leased the U.S. Government-owned property and the existing mill was converted to an aluminum foundry. Operations began in 1943 as an aluminum sand casting foundry to produce aircraft cylinder heads. From 1943 until 1945, GM produced cylinder heads for Allison and Packard Merlin aircraft engines for the US. Government. Additional land was later purchased and an additional building was erected to produce iron cylinder liners (these liners are no longer produced). Major Facility modifications occurred in 1950, 1953, 1966, 1971, 1974, 1977, 1979, and 1980.

In 1971, Minerals Processing Corporation (MPC) (an aluminum dross reclaimer) began operations on the portion of the property formerly occupied by the railroad maintenance operations. MPC leased the property from the railroad, which still owned the property in 1971. MPC's operations are included with the railroad maintenance operations identified as AOI 1, since they operated in the same area of the property. In 1977 GM purchased the property from the railroad that was formerly occupied by the railroad roundhouse and maintenance operations, and leased from the railroad by MPC. MPC

continued to operate the reclamation facility and leased the property from GM after GM assumed ownership until circa 1991.

2.3 WASTE MANAGEMENT PRACTICES AND RCRA REGULATORY HISTORY

2.3.1 RCRA REGULATORY HISTORY

In 1980, the Bedford Facility obtained a Resource Conservation Recovery Act (RCRA) Part A Permit for the storage of hazardous waste in excess of 90 days. GM obtained their Part B Permit for storage in 1983. The Hazardous Waste Storage Area was closed consistent with GM's approved closure plan in 1989, as it was no longer needed. A letter approving closure was received from the Indiana Department of Environmental Management (IDEM) on April 23, 1990.

2.3.2 CURRENT SOLID WASTE MANAGEMENT PRACTICES

The Waste Storage Area (also referred to as the Drum Storage Area and/or Oil House) is located to the north of the Piston Building. The Waste Storage Area (AOI 2) is located within a fenced area and is utilized for storing incoming liquid product, miscellaneous solid wastes, and hazardous waste materials. The entire Waste Storage Area is paved with secondary containment provided via a concrete curb and open drain leading to a sump.

2.3.3 HISTORICAL SOLID WASTE MANAGEMENT PRACTICES

Historically, between the late 1950's and 1984, solid wastes generated at the Facility were placed in the North Disposal Area (AOI 4). This area is located northeast of the former North Lagoon (see Figure 2.1).

In 1985, GM began placing excavated soil and demolition debris from a major on-site construction project, along with foundry sand fill, along the east edge of the salary parking lot in order to expand the parking lot. This area is referred to as the Former East Sand Disposal Area (AOI 5) as shown on Figure 2.1. Disposal in this area was discontinued in 1987 and the area was graded and covered with two feet of clay. This area was also used to stage sludges from the wastewater treatment lagoons in pits until

they dried. The sludge pits were then covered with clay. Currently, a portion of this area is used for fire training exercises.

2.3.4 CURRENT WASTEWATER MANAGEMENT PRACTICES

The nearest publicly owned treatment works (POTW) to the Bedford Facility is the City of Bedford POTW. The only existing discharge to the City of Bedford POTW is for sanitary wastewater (cafeteria and restrooms only). Currently all process water from the plant is discharged to the on site Bedford Facility wastewater treatment plant (WWTP) for treatment. The treated wastewater then is either recycled for process water for the Facility after primary and secondary treatment, or additionally passed through a tertiary treatment process (carbon filter) prior to discharge through the National Pollutant Discharge Elimination System (NPDES) permitted discharge Outfall 002. An average of 350,000 gallons per day (gpd) of treated water is discharged to Bailey's Branch of Pleasant Run Creek via an unnamed drainage originating at Outfall 002 (see Figure 1.1). The Bedford Facility currently reuses approximately 85 to 90 percent (2 to 3 million gallons per day [mgd]) of the treated wastewater.

Stormwater from the Bedford Facility flows through a stormwater sewer system to the Stormwater Lagoon. The stormwater in the Stormwater Lagoon is sent to the Bedford Facility WWTP. During rare high stormwater flow conditions, overflow from the lagoon is discharged through Outfall 003, by permit.

2.3.5 HISTORICAL WASTEWATER MANAGEMENT PRACTICES

GM has operated several forms of wastewater and storm water treatment facilities during the history of the Facility operations. Between 1952 and 1956, a sequence of five cascading lagoons were installed to treat wastewater and separate oil from the discharge.

Initially, some process water from the Bedford Facility operations was also discharged to the former North Lagoon (AOI 7), which primarily served to collect stormwater runoff from the northern portion of the Bedford Facility. Non-contact cooling water, roof drains, and floor drains located in several of the manufacturing departments within the Bedford Facility were also routed to the former North Lagoon and discharged to surface

water through Outfall 001. In 1974, the Bedford Facility received a NPDES permit for Outfall 001 (NPDES Process Discharge Permit No. IN 0003573).

Following approval from the Indiana State Pollution Control Board (ISPCB) and Indiana State Board of Health (ISBH) in January 1973, the Bedford Facility constructed a primary WWTP to reclaim hydraulic fluid from the wastewater originating from the Die Cast Department. The primary system consisted of a diversion chamber, two clarithickeners, a hydraulic fluid tank, and a heater and centrifuge. Construction and installation of the primary system took place at the location of South Lagoons #1, #2, and #3, thereby necessitating the draining, and cleaning out of sludges from the former South Lagoons #1, #2, and #3 (AOI 8).

In March 1974, both the ISPCB and the ISBH approved construction plans for a secondary wastewater treatment system with 90 percent recycle capacity of treated plant wastewater. The secondary water treatment system included a collection chamber, flash mix basin where chemicals were added, flume and splitter box, two clarifiers, sludge thickener, centrifuge, clearwell, filters, and a backwash reservoir. South Lagoon #5 was cleaned and closed in 1976 to make room for a new above ground concrete basin, as part of the decommissioning of the lagoons.

In June 1978, a Calgon carbon adsorption filter unit was installed to further reduce polychlorinated biphenyls (PCBs) in the discharge from Outfall 002. In June 1980, the Bedford Facility began removing sludges (via pumping) from the former South Lagoon #4. These sludges were dewatered on-site with a filter press and transported for disposal to the CECOS International, Inc.'s (CECOS) disposal facility (Secure Chemical Management Facility) near Williamsburg, Ohio.

Construction of the new Stormwater Lagoon (AOI 10) began in January 1981, pursuant to a construction permit issued by the ISPCB. The 14 million-gallon stormwater retention lagoon was constructed to replace the North Lagoon as Phase 4 of the WWTP expansion. It was designed to capture stormwater runoff from the entire Bedford Facility. The water in the Stormwater Lagoon is pumped to the aeration basins in the wastewater treatment system where it is combined with process wastewater and flows through the remainder of the treatment process. Emergency stormwater overflows are allowed under the NPDES permit via Outfall 003.

In late 1982, biological treatment was added to the process WWTP. The biological treatment system included aeration tanks, aerobic digesters and associated effluent

pump station, and return sludge pumps. The overall waste treatment facility consisted of primary separators, secondary aerobic biological treatment followed by clarification, gravity filtration, chlorination, and final carbon adsorption. Sludge digesters and filter presses were used to process the wastewater treatment sludge.

3.0 REGIONAL SETTING

3.1 LAND USE

The land in the vicinity of the Bedford Facility includes industrial, commercial and residential properties. The Bedford Facility is bordered to the north by residential and undeveloped areas, on the south by the Southern Indiana Railroad, IMCO, and residential property, on the east by residential and undeveloped areas, and on the west by the railway and residential properties, and a cemetery. A land use map is presented on Figure 3.1.

3.2 DEMOGRAPHICS

The Bedford Facility is located in Lawrence County in south central Indiana. Lawrence County is approximately 449 square miles in area with Bedford as its largest city. The City of Bedford has an estimated population of 13,800, which accounts for 30 percent of the approximately 45,752 residents of Lawrence County. Approximately 39.2 percent of the land area in Lawrence County is used for commercial and industrial; 36.5 percent used for residential; 17.6 percent used for agricultural; and, 6.7 percent used for utilities.

3.3 CLIMATE

The climate in Lawrence County is temperate with warm summers, cold winters, and no distinguishable wet or dry seasons. In winter, the average temperature is 34 degrees Fahrenheit (°F), and in the summer, the average temperature is 75°F. The average annual precipitation as rainfall is 28.4 inches and the average annual snowfall is 14.1 inches.

The average relative humidity for Bedford is 73 percent. The prevailing winds are generally from the southwest during most of the year. During the winter months they prevail from a northerly direction.

3.4 REGIONAL GEOLOGY

3.4.1 OVERBURDEN

The Bedford Facility lies within an area of Indiana that was not glaciated (driftless area) during the last glacial period on the North American continent. The maximum progression of the Illinoian Glacial advance (the furthest advance of the Laurentide Ice Sheet) lies to the west, north, and east of the immediate region surrounding the Bedford Facility. Consequently, the surficial geology of the area generally consists of a relatively thin layer of unconsolidated deposits of sand, clay, and chert produced by the weathering of limestone bedrock (regolith, which is more commonly referred to as terra rosa in this area due to its reddish color) and wind-deposited silty material, known as loess. Thicker deposits of proglacial outwash, lake sediment, and recent colluvium occurs along the major stream valleys. The surficial deposits range in thickness from zero feet along bedrock outcrops to approximately 100 feet thick along Salt Creek and the East Fork of the White River.

3.4.2 BEDROCK

The bedrock within the region is near the eastern margin of a structure known as the Illinois Basin. The bedrock formations in this area generally dip to the west at approximately 20 to 25 feet per mile. The Cincinnati Arch lies to the east of the Illinois Basin and covers much of Indiana.

Two regional structures are located within the vicinity of the Bedford Facility, the Leesward Anticline and the Mt. Carmel fault. The Leesward Anticline is located to the north and east of Bedford and plunges to the south-southeast. The Mt. Carmel fault is a normal fault with the downthrown side located to the west of the fault. This fault is located to the north and east of the Bedford Facility and truncates the Leesward Anticline on its western side. The Mt. Carmel fault generally acts as a hinge line, with gentler dips to the west of the fault and slightly steeper dips to the east.

Bedrock within the immediate vicinity of the Bedford Facility consists of the lower beds of the Middle Mississippian St. Louis Limestone (the oldest formation within the Blue River Group) and is only approximately 25 feet thick in the immediate vicinity of the Bedford Facility. Immediately underlying the St. Louis Limestone, and outcropping to the east of the Bedford Facility, are the Salem Limestone and the Harrodsburg

Limestone, respectively. These two Mississippian formations make up most of the Sanders Group. The Salem Limestone is approximately 70 to 80 feet thick, where preserved, and the Harrodsburg Limestone is approximately 80 to 90 feet thick in the area.

The Borden Group, which underlies the Sanders Group and outcrops further to the east, consists of approximately 500 to 800 feet of siltstone and shale, interbedded with some sandstone and minor limestone. The New Providence Shale formation makes up the bottom of the Borden Group, and is approximately 200 feet thick.

The Sanders and Blue River Groups have been described to consist mostly of carbonates, with minor amounts of chert, shale, siltstone, anhydrite, gypsum, and calcareous sandstone. A thin bed of brown dolomitic limestone commonly marks the bottom of the St. Louis Limestone. The Salem Limestone, which is more massively bedded limestone, is known as the Indiana Limestone, the Bedford Limestone, or the Oolitic Limestone and is quarried as fine building stone. However, some horizons may contain geodes, joints and solution fractures, which render the formation less suitable for quarrying.

Numerous joints and fractures are present in these formations with master sets trending east-west within the St. Louis Limestone, with minor sets 90 degrees to the master sets. Karst topography is abundantly present in the area. Numerous sinkholes can be observed on the United States Geological Survey (USGS) topographic quadrangles approximately 5 to 10 miles to the west of the Bedford Facility. Several caverns have been mapped in Lawrence County, including one of the largest mapped caverns in the United States, the Blue Springs Cavern, located approximately five miles southwest of the City of Bedford. Other mapped caverns in the area include the Shiloh Cave, the No Sweat Cave, the Dog Hill Cave, the Donnehue Cave, and the Salt Creek Cave. Other unmapped caverns within close proximity to the Bedford Facility include: Mouse Hole Cave, located one mile east-northeast; Eighteenth Street Cave, located one and one-half miles to the south-southeast; and Armstrong Caves I and II, located one and one-half miles to the west-southwest.

3.5 REGIONAL HYDROGEOLOGY

Groundwater resources are found in Lawrence County within unconsolidated deposits along the valleys of the major rivers or streams and within the thick Mississippian carbonate aquifer system (within the western portion of Lawrence County) and the

Silurian-Devonian carbonate bedrock aquifer (within the eastern portion of Lawrence County).

There are two basic types of aquifers: consolidated and unconsolidated. Unconsolidated aquifers in Lawrence County generally occur along the Salt Creek and the East Fork of the White River within the proglacial outwash deposits, glaciolacustrine deposits, and recent alluvium.

Groundwater flow within the consolidated (carbonate) aquifers takes place along the joints, fractures, and bedding planes that eventually may become enlarged by solution to cave passages or karst features. Recharge to a karst system occurs through surface openings that vary in scale from narrow, solutionally widened joints to large sinkholes. Discharge typically occurs through springs, which are solutionally widened joints or bedding planes, but may be enlarged, to sizable cave openings. Groundwater within this aquifer system discharges to surface water through fractures in the rock.

3.6 REGIONAL SURFACE WATER HYDROLOGY

Most of the rivers in the East Fork White River Basin drain to the southwest. According to USGS Water Resources Division, the current stream flow recorded at the East Fork White River gauging station, located 7.8 miles southeast of Bedford in Lawrence County, is 4,210 cubic feet per second (cfs).

Major tributaries to the East Fork White River include the Muscatatuck River, Salt Creek, Driftwood River, Flatrock River, and the Big Blue River. Drainages in the East Fork White River Basin include the Lost River, Sugar Creek, Graham Creek, Clifty Creek, Big Creek, Indian Creek, White Creek, Brandywine Creek, and the Little Blur River.

Rivers in the eastern half of the East Fork White River Basin have a subparallel drainage. Those rivers include the Sugar Creek, Big Blue River, Little Blur River, Flatrock River, Clifty Creek, Sand Creek, Vernon Forth, Graham Creek, and the East Fork White River from Medora to Jonesville.

Drainage of the Mitchell Plain in the central Lawrence County, northeast Orange County, and Monroe County is different from the rest of the East Fork White River Basin. In the streams that flow across the Mitchell Plain, surface water is intercepted by

swallow holes and diverted underground into the groundwater system or subterranean channels.

4.0 FACILITY SETTING

4.1 LOCAL TOPOGRAPHY

The Bedford Facility is located on the Mitchell Plain physiographic province, which is underlain by Mississippian limestones. The area is a low relief karst plain that is pitted in some areas by numerous sinkholes. This low-lying karst plateau is developed on soluble limestones of the Blue River Group, sloping approximately 25 to 30 feet per mile westward (Fenelon and Greeman, 1994).

Based on the USGS 7.5 minute topographical quadrangles (Oolitic, Bartlettsville, Bedford East, and Bedford West Quadrangles), elevations around the Facility in Lawrence County range from 550 feet above mean sea level (amsl) to 700 feet above amsl. The elevation of the Bailey's Branch and Pleasant Run Creek stream channels range from approximately 550 to 600 feet amsl.

4.2 GEOLOGY

The surficial geology of the area generally consists of a relatively thin layer of unconsolidated deposits of sand, clay, and chert produced by the weathering of limestone bedrock (regolith), and loess. Thicker deposits of proglacial outwash and lake sediment, and recent colluvium occurs along the major stream valleys.

Soil in the vicinity of the Bedford Facility has been mapped by the United Stated Department of Agriculture (USDA) as Udorthents (or manmade soils). Soil classification includes earth and broken rock materials for road fills and embankments. Fills include: rock, broken brick, concrete, steel, and glass. The natural soil in the immediate vicinity of the Bedford Facility is known as Crider. Crider soil is a fine-grained, silt loam to silty clay loam. Crider soil develops on 20 to 42 inches of silty loess over clayey material derived from limestone.

The ridge tops and high hill slopes in the vicinity of the Bedford Facility are layered with limestone and thin shales of the Mississippian St. Louis limestone, which is underlain by the Salem Limestone. The valley bottoms and walls along the Bailey's Branch and Pleasant Run Creek are developed within the upper part of the Borden Group, consisting of shales, siltstones, sandstones, and some limestone. No sink holes, caverns, or sinking streams have been observed on the Bedford Facility.

The gradient of bedrock and fracture patterns in the area can be observed in quarries and road cuts. The bedrock layers dip to the southwest at approximately 20 to 25 feet per mile in the Bedford area. Near the bedrock surface are identifiable sets of fractures or joints.

Other types of joints include sheet, vertical, and inclined. Sheet joints are horizontal fractures occurring at or near the bedrock surface and commonly found in porous, softer limestone beds. Based on data collected from adjacent abandoned quarries, and nearby road and railroad cuts, vertical joints that occur in the limestone bedrock are relatively planar as compared to other fractures. Inclined joints occur in the lower part of the St. Louis Limestone and the upper part of the Salem Limestone at an approximate 60-degree angle from the horizontal.

4.3 HYDROGEOLOGY

Much of the groundwater flow within the Mississippian carbonate aquifer near the Bedford Facility is subject to the influence of karst topography. The water flow is likely along solution-enlarged joints and fractures of the St. Louis limestone discharging into streams and to the surface through springs.

Several springs can be observed on the Bedford Facility property. Some springs originate as minor seeps, which occasionally disappear into the ground, only to reappear downstream. Some springs on the property provide flow and likely contribute to the baseflow of the unnamed ditches.

Groundwater at the Facility is expected to be encountered within the carbonate bedrock. However, Facility-specific information is generally not known and groundwater may potentially be encountered as perched water within the unconsolidated material, or within the epi-karst zone (weathered, partially dissolved portions of the upper carbonate bedrock surface).

Pre-Investigation, Hydrogeologic Conceptual Model

The following presents an analysis of the anticipated hydrogeologic conditions that may be encountered at the Bedford Facility during the initial subsurface investigation. This analysis has been developed based on existing literature, site observations, and physical properties of potential constituents of concern that may be present in the environment due to historical operations at the Facility. The presented conceptual model is not based on actual data collected (inadequate actual site data exist) from the Facility and will be modified based on information collected during the RFI, including hydrogeologic data from the monitoring wells proposed in Section 5.1.5.

Shallow, perched groundwater may be present in the unconsolidated deposits and/or the epi-karst zone (the upper, weathered/dissolved bedrock). Groundwater flow through this zone may be horizontal (e.g., discharge as springs), vertical into joints, fractures, and bedding planes of the underlying competent bedrock, or a combination of both. Potential groundwater flow in the epi-karst zone may be through granular matrix or through fracture flow similar to the bedrock. Given the apparent ephemeral nature of the springs in the vicinity of the Facility, the elevation of the shallow groundwater may fluctuate significantly throughout the year, and may fluctuate rapidly in response to rainfall events. Recharge of the epi-karst zone is likely directly from precipitation infiltration, through surface water streams and/or from the underlying bedrock.

The general direction of groundwater flow within the unconsolidated overburden, the epi-karst zone, and the bedrock is not known at this time. However, based on the surrounding topography and apparent base-flow observations of the unnamed ditches, springs, and streams, it is possible that the shallow groundwater flow could be in any direction from the plant (the plant being topographically higher than the surrounding area).

The majority of the groundwater flow within the carbonate bedrock is expected to occur through secondary porosity/permeability features such as fractures and joints. Much of the recharge of this system occurs through grikes (exposed fractures at the ground surface) and sinkholes (Palmer, 1969). Such fractures, joints, and bedding planes may be solutionally enlarged, especially within the expected upper bedrock formation (St. Louis). Groundwater flow within the solutionally enlarged features may be pipe-full (or conduit) or may behave similar to surface water flow within non pipe-full conditions. Either way, horizontal groundwater flow through the bedrock is expected to be primarily fracture controlled (dominated by the secondary porosity rather than through

the matrix of the limestone). Successful groundwater monitoring wells will need to intersect those fractures (exact locations of these fractures are not known).

Though not immediately adjacent to the Facility, a review of local well logs and published information indicates that shallow groundwater is also present nearby in unconsolidated, alluvial deposits. These deposits are generally located along the floodways of Salt Creek and along the East Fork of the White River, though these deposits may extend into portions of Pleasant Run Creek.

Potential constituents of concern at this Facility include the potential for both dissolved-phase (transport with groundwater flow) and non-aqueous phase contaminants (transport controlled by structural features). Dissolved phase transport could occur through the dissolution of separate phase liquid, or through contaminant loading from overlying soil.

4.4 SURFACE WATER HYDROLOGY

The Bedford Facility is situated high on a ridge such that the Facility is drained by surface runoff primarily to the east and northeast in small valleys (or unnamed ditches) which are tributaries of the Bailey's Branch of Pleasant Run Creek. The ridge top is approximately 150 to 185 feet higher than the valley bottom one-half mile northeast of the Bedford Facility. According to Facility personnel, surface water runoff from the Facility to the west is minimal.

Stormwater from the operations portions (e.g., improved surfaces) of the Bedford Facility is currently routed to the Stormwater Lagoon (which is part of the Facility's recycled water system). Stormwater from non-operational portions of the Facility (i.e., property located north and east of the Stormwater Lagoon) drains directly to several unnamed ditches and eventually to Bailey's Branch of Pleasant Run Creek.

5.0 SAMPLING AND ANALYSIS PLAN

5.1 OVERALL SAMPLING RATIONALE

The purpose of sampling activities will be to determine whether a significant release of hazardous waste and/or hazardous constituents has occurred at each AOI. The proposed investigation is designed to identify contaminants of concern, if any, and characterize exposure concentrations. Additional sampling to determine the extent of contamination or provide additional data for statistical analysis may be required to make risk-based decisions on the need for corrective measures. Sampling activities will generally be completed as follows, although sample collection methods may be varied as necessary to accommodate specific features of each AOI (e.g., Shelby tube vs. split-barrel, etc.).

A summary of the samples to be collected in each AOI is presented in Table 5.1. General sample locations are presented on Figure 5.0. Exact sample locations will be determined based on field conditions and biased to target areas, or intervals, of highest suspected concentrations based on knowledge of historic operations.

5.1.1 SOIL SAMPLING

Soil sample locations will be selected consistent with the purpose of the investigation for each AOI. Conservatively biased sampling schemes will generally be employed, and exact sample locations may be modified due to field conditions. Soil samples from up to four sample depths will generally be collected to provide data for assessing surface soil, soil at typical construction depths, and soil above groundwater. A fourth soil sample may be collected if visual and/or photoionization detector (PID) field information indicates a zone in between the other three locations that shows potential impact to soil. Any deviations from this sample strategy will be described under the discussion of each AOI proposed investigation.

1) The direct contact scenario refers to the potential for Facility workers to contact contaminated soil during the course of normal work activities. The 0 to 2-foot interval below ground surface (bgs) represents the surface soil most likely to be encountered by Facility workers. If the soil is overlain by concrete, asphalt, railway ballast, or other material, the direct contact sample will be collected from

the first soil interval beneath these materials and any undercourse material that may be present.

2) The construction worker scenario refers to the potential for construction workers to excavate into subsurface soil during the course of construction or maintenance activities. Generally, a two-foot sample will be collected from within the 6 to 10-foot depth interval to represent a typical construction depth. However, if the water table is encountered before the construction worker sample interval, this sample will be collected above the water table and will represent the second and third sample intervals as one sample.

3) The third sample (or second sample if the water table is encountered before the construction worker sample interval) evaluates the potential for surface contaminants to have infiltrated through contaminated soil to the water table and potentially impact groundwater. The interval immediately above the water table provides a potential means for determining whether contaminants in soil have migrated to groundwater. If groundwater is not encountered, the third soil sample will be collected from the soil directly overlying the bedrock surface. A fourth soil sample may be collected if visual and/or PID field information indicates a zone in between the other three locations that shows potential impact to soil.

4) To allow for evaluation of soil sample results from the AOIs, background soil samples will be collected from the surface soil, and from the St. Louis and Salem Formations. A total of nine surface soil samples will be collected from areas at or near the Facility with no known history of activities that may have caused contamination. In addition, samples at four separate locations will be collected from unconsolidated soil (6-10 feet bgs), from the St. Louis Formation (if encountered), and from the Salem Formation (if encountered) during the installation of the groundwater monitoring wells. The distance between sample locations will be maximized to obtain a representative data set. Background soil samples (and associated QA/QC samples) will be analyzed for the inorganic list of constituents presented in Appendix C, total organic carbon (TOC), and grain size.

5.1.2 GEOPHYSICAL DELINEATION

Historic information indicates that sludges from lagoons formerly in operation at the Facility were disposed of in AOI 6. In order to delineate the sludge disposal areas, a geophysical investigation will be conducted.

Initially, the proposed geophysical investigation will be completed utilizing electromagnetic techniques, consisting of EM31 and EM61 surveys. If these techniques do not provide the needed information, other geophysical techniques will be evaluated (e.g., resistivity, shallow seismic, ground penetrating radar, etc.). The sludges may be difficult to locate based on an intrinsic geophysical signature, however the disposal areas may be identifiable due to contrasts between the disposal pit cap material and surrounding soils.

The various geophysical methods proposed for the investigation can be briefly described as follows. The EM31 is characterized by a 14 foot long boom, with a transmitter-receiver coil separation yielding an approximate depth of penetration of 10 feet in the horizontal dipole mode and 20 feet in vertical dipole mode. The EM31 will measure values of terrain conductivity in order to determine if a contrast exists between the soil conductivity of the sludge pits and cap material, and the surrounding soils.

The EM61 has a stacked coil configuration and is primarily designed for detecting buried metal objects at depths up to 10 feet. This instrument has also been used to determine the limits of (metallic) conductive fill including foundry sand. In order to determine if sludge pits were excavated in the foundry sand and then capped with clay, an EM61 survey will be completed over area AOI 5. It is anticipated that if a clay cap exists within the foundry sand (which subsequently has not been covered by foundry sand), the EM61's comparatively small radius of influence will delineate the location of a former sludge pit.

Upon completion of the geophysical investigation, the survey results will be presented as colored, contoured plots, for ease of interpretation. Any anomalous areas detected by the geophysical surveys which may represent locations of sludge pits will be outlined on the plots. Recommendations for intrusive activities including soil borings and soil sampling locations will be provided, based on a review of this information.

5.1.3 SURFACE SOIL/SEDIMENT SAMPLING

Surface soil samples are proposed at each soil sampling location. However, due to the nature of the materials managed or physical features in the area, certain AOIs are not expected to contribute to subsurface contamination. Therefore, only surficial samples will be collected in these areas. Surface soil and/or sediment sample locations will be selected consistent with the purpose of the investigation for each of these AOIs. Conservatively biased sampling schemes will generally be employed. Sample locations will also be governed by physical constraints.

1) Sediment samples will be collected of the fine-grained sediment, if possible, from the upper four inches (or 10 centimeters) of the sediment column.
2) Surface soil samples will be collected from the 0 to 2 feet bgs interval (excluding pavement and undercourse).

5.1.4 GROUNDWATER SAMPLING

Nine groundwater monitoring wells are proposed to be installed around the perimeter of the Facility based on the review of initial file information and the potential for off-site migration of contaminants (refer to Section 5.4). The wells are intended to give an initial understanding of site-wide hydrogeological conditions at the Facility. In addition, two groundwater collection sumps will be sampled for characterization of the groundwater quality at these locations (Section 5.4).

5.1.5 PROPOSED MONITORING WELL INSTALLATION

An initial set of nine groundwater monitoring wells are proposed around the perimeter of the property. These monitoring wells are anticipated to provide preliminary hydrogeologic information sufficient to design an appropriate future monitoring well network, where required. In order to gain the most information out of a single bedrock borehole, multi-level packers will be used to separate water-bearing fracture zones within a single borehole.

Each boring intended for the installation of the groundwater monitoring wells will be advanced utilizing conventional drilling methods. Continuous cores will be collected of the competent bedrock for the total depth of the borings. The overburden (Section 5.1.1)

will be continuously sampled using a standard split-barrel sampler, Shelby tube, or continuous core sampler and will be visually classified (described) in the field at the time of collection. If cohesive soil is present, a minimum of one Shelby tube at each of the monitoring well locations will be submitted to a geotechnical laboratory for analysis of grain size and vertical permeability. If groundwater is encountered in the overburden, a shallow monitoring well will be installed to monitor that zone. In this case, the bedrock drilling will be completed after setting an outside casing to prevent the vertical migration of overburden groundwater to the bedrock.

The competent bedrock will be logged in 20 foot vertical increments during advancement (this is proposed for the first well boring and may be revised based on field conditions encountered). This logging will consist of visual inspection of the collected cores, borehole diameter (caliper), and optical acoustic televiewer at a minimum. Additional geophysical logging (i.e., temperature, resistivity, natural-gamma, and spontaneous potential) will be performed on the borehole upon completion to final depth. Boreholes will not be advanced vertically past any detected separate-phase liquids. If a non-aqueous phase liquid (NAPL) is not encountered, it is expected that the boreholes will be advanced to a maximum of 50 feet into the Salem Formation. If groundwater is not encountered, the borehole will be left open to observe if groundwater will enter the borehole. If groundwater does not enter the borehole within a reasonable amount of time (e.g., through a precipitation event), the borehole will be abandoned using a cement/bentonite slurry grout.

Groundwater samples will be collected using low-flow purging/sampling techniques. Groundwater samples collected for metals analyses will be filtered in the field upon collection using a 0.45 micron filter. Groundwater samples collected for PCB analysis will be both filtered and unfiltered. All other groundwater samples collected will be unfiltered.

5.1.6 CONCRETE INSPECTION/SAMPLING

Concrete inspection has been completed for two AOIs described below (AOI 3 and 24). AOI 3 consists of a former PCB storage area within the Sludge Building and a former PCB storage area within the Die Cast Building. AOI 24 is an area where a spill occurred to the concrete and was subsequently cleaned. There was no known release to the environment at any of these locations.

However, the concrete floor at the former PCB storage area (within the Die Cast Building) was observed to be pitted during the concrete inspection. In order to assess the current potential exposure from this area, the concrete floor (for the former PCB storage area) will be sampled as described below. The former PCB storage area (within the WWTP Sludge Building) and AOI 24 were inspected and the results did not reveal any deterioration, observable cracks, or any visual evidence of a release. The concrete from these two areas will not be further sampled.

Concrete wipe samples will be conducted at the former PCB storage area AOI 3 (located in the Die Cast Building). The wipe samples will consist of swabbing a 10 centimeter (cm) by 10 cm area with a prepared, hexane-saturated gauze pad or glass wool.

5.1.7 SURFACE WATER SAMPLING

Surface water samples will be collected at several locations across the Facility during the RFI. If sediment samples are proposed to be collected at the same locations as surface water samples, the surface water sample will be collected prior to sediment samples to represent normal undisturbed conditions.
Surface water samples collected for metals analyses will be filtered in the field upon collection using a 0.45 micron filter, as well as, unfiltered. Surface water samples collected for PCB analysis will be both filtered and unfiltered. All other surface water samples collected will be unfiltered

5.2 OFF-SITE WELL SURVEY AND SAMPLING

This scope of work was included in the Off-Site Well Survey and Sampling Work Plan (June 6, 2001).

5.3 STREAM INVESTIGATION

This scope of work was included in the Stream Investigation Work Plan (June 6, 2001).

5.4 INVESTIGATION IN AREAS OF INTEREST (AOIs)

The following sections provide descriptions of the current conditions of each AOI and outline the proposed investigation in each AOI. Figures 2.1 and 5.0 present the locations of these AOIs.

5.4.1 AOI 1 – FORMER RAILROAD OPERATIONS AND MINERALS PROCESSING FACILITY

The portion of land southeast of the Die Cast Building was the location of the former railroad maintenance operations and the Minerals Processing Facility. The area formerly utilized by MPC is currently used by GM to temporarily store aluminum sows (a product produced by a secondary aluminum smelter, that is remelted by a manufacturer and which requires less adjustment of the metal chemistry by the manufacturer). The former railroad maintenance operations and MPC are no longer present (the buildings have been removed). Two springs, which discharge near the WWTP, appear to emanate from this area. These springs, when water is present, each discharge to a concrete sump, both located behind the current WWTP. The water collected in these sumps is gravity-drained to the pump house (located near Outfall 002) and ultimately pumped to the current Stormwater Lagoon.

Proposed Soil Investigation

Seven soil borings will be advanced within AOI 1 to assess the current soil quality of this area, including two borings each immediately below the areas previously utilized by Minerals Processing Facility and the former railroad turntable (Figure 5.1). Up to four soil samples (and associated QA/QC) from each boring will be collected in accordance with Section 5.1.1 and samples will be submitted to the laboratory for analysis of Target Compound Lists (TCL) for volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), PCBs, inorganics (Target Analyte List (TAL) minus the earth metals), and chloride. Each boring will be advanced to a maximum of three feet into the underlying bedrock, if possible (this area is suspected to have been filled in with numerous milled limestone blocks, which may create problems with drilling).

Proposed Spring/Seep Investigation

In addition to the soil samples described above, one surface water sample will be collected from each of the two concrete sumps which collect spring water emanating from this area and draining near the GM WWTP (Figure 5.1), if accessible and if water is present. The water samples (and associated QA/QC) will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, inorganics (TAL minus the earth metals), and chloride.

5.4.2 AOI 2 – WASTE STORAGE AREA

The Waste Storage Area (also referred to as the Drum Storage Pad) is a secure fenced area located north of the Piston Building. The Former RCRA Hazardous Waste Storage Area was located in the northeast corner of the Waste Storage Area.

The entire Waste Storage Area, along with the drum storage, is paved with secondary containment provided via a concrete curb. A sump, which gravity drains to the WWTP, is located in the southeast corner of the area. The Former RCRA Hazardous Waste Storage Area located within the Waste Storage Area received closure from IDEM in April 1990.

Proposed Soil Investigation

Four soil borings are proposed to be advanced within this AOI; one boring at each of the four corners (Figure 5.2). Up to four soil samples from each borehole (and associated QA/QC) will be collected in accordance with Section 5.1.1 and will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals). Each boring will be advanced to a maximum of three feet into the bedrock surface, if possible (this area is suspected to have been a filled-in bedrock valley, which may create problems for drilling).

5.4.3 AOI 3 – PCB STORAGE AREAS

There have been two PCB storage areas utilized at the Facility. One former PCB storage area consisting of a secure room was located in the west side of the Die Cast Building and one former PCB storage area was located in the WWTP Sludge Building. The

curbing in the former PCB Storage Area at the Die Cast Building has been removed and the area is used for general storage. The former PCB Storage Area at the Sludge Building is curbed on a concrete floor with a blind sump (this structure remains, though, is no longer utilized for the storage of PCBs). A 55-gallon drum, used for the storage of PCB material (e.g., miscellaneous rags, gloves and cleanup materials), was formerly located in the curbed area.

Concrete Inspection/Sampling

No known releases have occurred from either of the former PCB Storage Areas. On August 28, 2001, GM, U.S.EPA, and IDEM conducted a visual inspection of both PCB Storage Areas. Based on that inspection and correspondence received from the U.S.EPA, three concrete wipe samples will be collected to assess the current condition of the former PCB Storage Area located within the Die Cast Building (refer to Figure 5.3).

Based on the visual inspection conducted on August 28, 2001, samples will not be collected from the former PCB Storage Area located in the Sludge Building because there has been no documented release at this location and the visual inspection did not reveal any evidence of a historical release.

5.4.4 AOI 4 – FORMER NORTH DISPOSAL AREA

Beginning in the mid 1950s, the Bedford Facility began disposing of waste materials northeast of the former North Lagoon. This area has been referred to as the Sand Fill Area, Sand Landfill, Foundry Sand Storage Area, Landfill Storage Area, North Landfill, and Landfill. This area is currently covered with grass and is inactive. Several springs, located along the north toe of the disposal area, were observed during a February 2001 site walkover completed for the development of the Current Conditions Report (May 25, 2001).

Proposed Soil Investigation

Five soil borings are proposed in this area to characterize the nature of the surficial soil, fill materials, and underlying materials. Two additional soil borings, and associated sampling, will be completed as part of the investigation of AOI 15, which is located atop the North Disposal Area (Figure 5.4). Up to four soil samples (and associated QA/QC)

will be collected at each boring. Soil samples will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals).

Proposed Spring/Seep Investigation

One surface water (if water is present) and one sediment sample will be collected at each of three separate locations where springs/seeps have been observed near, or emanating directly from, the toe of the slope of the fill material (Figure 5.4). Each sediment and surface water sample (and associated QA/QC) will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals).

5.4.5 AOI 5 – FORMER EAST SAND DISPOSAL AREA

In 1985, GM began disposing of foundry sand east of the salary parking lot. The foundry sand was used as fill to expand the existing parking lot. The Former East Sand Disposal Area is no longer used for disposal purposes and is partially covered by asphalt from the salary parking lot and by grass. No other operations have been, or are currently considered in this area.

Proposed Soil Investigation

Four soil borings are proposed in this area to characterize the nature of the surficial soil, fill materials, and underlying materials (Figure 5.5). Additional soil borings, and associated sampling, will be completed as part of the investigation of AOI 6, which are also located in the East Sand Disposal Area. Up to four soil samples (and associated QA/QC) will be collected at each boring. Soil samples will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals).

5.4.6 AOI 6 – FORMER SLUDGE DISPOSAL AND FIRE TRAINING AREA

The former Sludge Disposal Area was identified from historical aerial photographs and from interviews with Facility personnel. This disposal area was located within the East Sand Disposal Area on Facility property to the east and south of the salary parking lot east of GM Drive. The Facility's fire-training area is located south of the salary parking lot and is situated on top of the former Sludge Disposal Area. The former Sludge

Disposal Area east of the salary parking lot is currently covered with grass and some trees. The disposal area and fire training area south of the salary lot is also covered with grass and contains several fire-training pits for the Facility.

Geophysical Delineation

As discussed in Section 5.1.2, a geophysical survey will be completed in an attempt to determine the actual locations of the sludge pits within the fill material.

Proposed Soil Investigation

Based on the results of the geophysical delineation, it is anticipated that up to seven soil borings will be advanced in this area to characterize the nature of the surficial soil, fill materials, and underlying materials (actual locations depicted on Figure 5.6 may change based on the results of the geophysical survey). Up to four soil samples (and associated QA/QC) will be collected at each boring. Soil samples will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals).

5.4.7 AOI 7 – FORMER NORTH LAGOON AND OUTFALL 001

The Stone City Construction Company originally constructed the Former North Lagoon (also referred to as the North Pond, Old Lagoon, and North Stormwater Lagoon) sometime between 1938 and 1942. GM continued to use the Former North Lagoon after taking over in 1942 for stormwater collection and utilized a portion of the stormwater in the Facility's manufacturing processes on an as-needed basis until 1981. GM completed closure activities (soil and sediment removal and disposal) of the Former North Lagoon in 1987.

The Former North Lagoon is currently a grass field, with the only visible feature being a manhole collection sump. Outfall 001 no longer exists; however, the unnamed ditch that carried the discharge is still present. There is no current surface water flow from this former outfall.

Proposed Soil Investigation

Two soil borings will be advanced (within the area that was previously excavated and backfilled) at locations where the highest levels of PCBs remained after excavation

(Figure 5.7). Soil samples (and associated QA/QC) will be collected in accordance with Section 5.1.1 (up to four samples per boring) and one additional sample per boring will be collected of the bedrock surface. These samples will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals). It is anticipated that both soil borings will be advanced to a maximum of three feet into the bedrock.

Additional soil, sediment, and/or surface water samples will be collected downstream of this former outfall during implementation of the Stream Investigation Work Plan (June 6, 2001) and through the investigation at AOI 4.

Proposed Groundwater Sample

One groundwater sample is proposed to be collected from the existing collection sump at this AOI (Figure 5.7). This sump currently collects groundwater from a drainage system installed prior to backfilling the former excavation which is then routed through the Plant's WWTP. The water sample (and associated QA/QC) will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals).

5.4.8 AOI 8 – FORMER SOUTH LAGOONS AND OUTFALL 002

GM utilized five lagoons (South Lagoons #1 through #5) at the Bedford Facility for the purpose of treating process wastewater prior to discharge to Outfall 002, pursuant to a NPDES permit (IN 0003573), originally issued in 1974. These former South Lagoons (also referred to as the Process Lagoons; South Lagoon #4 also referred to as the Sludge Storage Lagoon) operated in a cascading-type fashion in order to separate and reclaim the heavier hydraulic oils (settled to the bottom) contained in the process wastewater. The process wastewater was primarily from the existing service tunnels beneath the die cast machines. GM constructed the former South Lagoons in 1959. The five South Lagoons have been removed. The existing WWTP occupies the location of Former South Lagoons #1, #2, #3 and #5. Although there have been no documented releases from Former South Lagoons #1, #2, #3, and #5, there is a potential that subsurface conditions may have been impacted due to the nature of the operations of the lagoons.

GM completed closure activities (soil and sediment removal and disposal) at the Former South Lagoon #4 in 1983. The location of Former South Lagoon #4 is currently a grass

field with a collection sump that transfers any groundwater in the sump to the WWTP. Based on historic operations of South Lagoon #4 and the presence of water and oil entering the excavation during closure of the lagoon, there is a potential that subsurface conditions may have been impacted.

The Facility currently discharges treated wastewater through Outfall 002.

Proposed Soil Investigation

Two soil borings will be advanced within this AOI; one will be located near the north sidewall of the former excavation of South Lagoon #4 and one will be located at the location of the former South Lagoon #5 (See Figure 5.8). Soil samples (and associated QA/QC) will be collected in accordance with Section 5.1.1 (up to four samples per boring) and one additional sample from the boring at #4 South Lagoon will be collected of the bedrock surface. These samples and will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals). Additional soil samples will be collected in the area during the investigation of AOI 11. It is anticipated that both soil borings will be advanced to a maximum of three feet into the bedrock.

No boreholes are proposed at former South Lagoons #1, #2, and #3 since the area is currently occupied by the existing WWTP. It is believed these were cleaned out to install the WWTP, which extends to a greater depth than the lagoons were believed to be, and access is very limited by underground utilities. Additional soil, sediment, and/or surface water samples will be collected downstream of Outfall 002 during implementation of the Stream Investigation Work Plan (June 6, 2001).

Proposed Groundwater Sample

One groundwater sample is proposed to be collected from the existing collection sump at this AOI (Figure 5.8). This sump currently collects groundwater from a drainage system, installed prior to backfilling the former excavation of South Lagoon #4, and routes the water through the Plant's WWTP. The water sample (and associated QA/QC) will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals).

5.4.9 AOI 9 – SERVICE TUNNELS

Six service tunnels are located in the Die Cast Building at the Bedford Facility. The service tunnels are located under the die cast machines and are identified as: North Make #'s 1 through #5 and South Make #1. Portions of the six service tunnels in the Die Cast Building are currently being utilized for maintaining the die cast machines and to convey hydraulic fluid from the machines to the WWTP. All of the die cast service tunnels are constructed of concrete with portions of the ceiling open to the die cast machines on the operating floor.

The western and eastern ends of the service tunnel for North Make #1 and the eastern two-thirds of the service tunnel for South Make #1 have been closed. Portions were closed pre-1976 and mid-1980 by breaking up the tunnel floors and filling with fill material and unspecified construction debris. Additional portions of the North Make #1 tunnel were closed in 1990 by leaving the tunnel floor intact and filling in the tunnel.

Proposed Soil /Rock Investigation

Nine soil borings are proposed to be advanced inside of the Die Cast Building at the eastern and western ends of the existing and former service tunnels (Figure 5.9 shows the currently proposed locations). Actual boring locations may be adjusted to accommodate for equipment that cannot be moved and to locate the borings at the ends of the service tunnels, where adequate ceiling height exists for drilling.

Soil/rock samples are anticipated to be collected from two depths at each boring location; one from the approximate elevation of the tunnel floor, and one sample from approximately ten to twenty feet below the floor elevation. Additional sample intervals may be selected if evidence of contamination is observed. All samples (and associated QA/QC) submitted to the laboratory will be analyzed for TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals).

Access to the tunnel areas within the plant is severely limited due to operating machinery and molten metal transfer activities. Therefore, the completion of these proposed borings will be coordinated with the production schedules, and may need to be completed during times when production operations are temporarily shut down (i.e., holidays, equipment changeovers, etc.).

5.4.10 AOI 10 – EXISTING STORMWATER LAGOON AND OUTFALL 003

The Existing Stormwater Lagoon is located northeast of the salary parking lot, east of GM Drive. The Stormwater Lagoon, which discharges to Outfall 003, is permitted emergency stormwater overflows and is designed to contain stormwater runoff from improved surfaces expected from a 25-year, 24-hour storm event. The emergency overflow from Outfall 003 enters an unnamed ditch and flows to the northeast until the ditch joins with Bailey's Branch of Pleasant Run Creek. This lagoon is still in operation and serves to collect stormwater. It is also part of the Facility's water recycle system. The water in the Stormwater Lagoon during normal, non-overflow conditions is pumped to the aeration basins in the waste treatment system, where it combines with process wastewater and flows through the remainder of the treatment process.

Proposed Sediment Investigation

Three sediment samples are proposed to be collected at this AOI. Two sediment samples will be collected from within the current lagoon (one of these will be collected near where the stormwater is pumped into the lagoon) and one sediment sample will be collected from the location of the spring near Outfall 003 (Figure 5.10). All samples (and associated QA/QC) will be collected in accordance with Section 5.1.3 and will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs and inorganics (TAL minus the earth metals).

Proposed Surface Water Investigation

One surface water sample will be collected from the existing lagoon and one surface water sample will be collected from the spring located near Outfall 003 (see Figure 5.10). Collected surface water samples (and associated QA/QC) will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals).

5.4.11 AOI 11 – ABOVEGROUND STORAGE TANKS

The Bedford Facility has historically utilized several above ground storage tanks (ASTs) for storage of various materials including vehicle fuel and PCB-containing fluids (hydraulic fluids or oils). ASTs with documented or suspected releases have been classified as AOIs. These include: Former Used Hydraulic Fluid ASTs; Used Mineral

Oil AST Release; and, Temporary Diesel AST Release (the location of these AOIs are shown on Figure 5.11). The former used hydraulic fluid ASTs were located in three bermed areas east of GM Drive. The WWTP concrete holding basin currently covers one of the former bermed areas; the second former bermed AST area is covered with gravel west of the WWTP concrete treatment tank; and, the third former bermed AST area is located within the bermed containment for the one-million gallons ASTs. The one million-gallon ASTs remain active and are used as surge tanks for the WWTP. The used mineral oil hydraulic fluid AST which had a release in 1977 and the surrounding asphalt were removed between March and June 1977. The location of the October 2000 diesel fuel release is currently covered with gravel.

Proposed Soil Investigation For 29 Former Used Oil ASTs

Three soil borings will be advanced at this AOI; one at each of the three former bermed areas that had contained the ASTs (Figure 5.11). Soil samples (and associated QA/QC) from each of these borings will be collected in accordance with Section 5.1.1 (up to four samples per boring) and will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals). It is anticipated that each soil boring will be advanced to a maximum of three feet into the bedrock.

Proposed Soil Investigation For The Mineral Oil AST Release

One surficial soil sample will be collected in accordance with Section 5.1.3 from the location of the 1977 mineral oil AST release to assess whether or not impacted materials remain at this location (see Figure 5.11). The soil sample collected from the mineral oil release will be analyzed for TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals).

Proposed Soil Investigation Diesel Fuel AST Release

One surficial soil sample will be collected in accordance with Section 5.1.3 from the location of the diesel fuel AST release to assess whether or not impacted materials remain at this location (see Figure 5.11). The soil sample collected from the diesel fuel release will be analyzed for TCL VOCs, SVOCs, and PCBs.

5.4.12 AOI 12 – AREA AFFECTED BY THE RECLAIMED HYDRAULIC FLUID RELEASE

A release of reclaimed hydraulic fluid occurred when a tanker ruptured adjacent to the Die Cast Building. Two storm drains are located in the area where the release occurred and the area is covered with concrete. During a February 2001 site walkover completed for the development of the CCR, the condition of the concrete was noted to be deteriorated from truck traffic and facility use.

Proposed Soil Investigation

Two surficial soil samples (beneath the pavement and undercourse) will be collected from within this AOI (one near where the release occurred and one near the stormwater drain) to assess whether or not impacted materials remain at this location (Figure 5.12). Each soil sample (and associated QA/QC) will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals).

5.4.13 AOI 13 – UNDERGROUND STORAGE TANKS

Seven underground storage tanks (USTs) have been utilized at the Facility for the storage of vehicle fuel and ethyl silicate. These USTs were installed between 1950 and 1977 and were removed between 1991 and 1994. The seven USTs located at the Facility have been removed and closure reports have been submitted to the IDEM.

Closure Verification

Based on review of the file information, it appears these areas were adequately remediated. No additional sampling is proposed for either UST location (Figure 5.13). A formal request will be submitted to IDEM regarding the State's concurrence with the findings of the Closure Reports.

5.4.14 AOI 14 – MCBRIDE COWS DISPOSAL AREA

In April 1978, GM purchased and euthanized 91 head of cattle from the McBride dairy farm located along Bailey's Branch of Pleasant Run Creek, into which GM discharged its treated wastewater. Documentation indicated that the cattle were properly disposed of in accordance with Indiana Code IC-2.1-16-20. Euthanized cattle were placed in a

prepared pit where the sidewalls and bottom were plastic-lined and the cattle were covered with four to six feet of clay. This area is currently inactive and covered with grass. No other facility activity has occurred in this area.

Proposed Soil Investigation

Soil borings will be advanced within the disposal pit to determine the approximate dimensions (Figure 5.14). One soil sample will be collected from within the pit at the cow remnants level and one sample will be collected from outside the pit at a depth expected to be below the pit depth for analysis of PCBs.

5.4.15 AOI 15 – FORMER EQUIPMENT STORAGE AREA

A review of historical photographs, aerials, drawings and interviews with Facility personnel identified a former Equipment Storage Area located on the east side of GM Drive. The former Equipment Storage Area is located north of the former North Lagoon and is believed to overlie a portion of the Former North Disposal Area (AOI 4). It is currently covered with gravel, and has a fence on the north, south and west (with a gate) sides. The east side of this area is open. GM currently leases this area to a trucking company for trailer parking.

Proposed Soil Investigation

Two soil borings are proposed to be advanced within this AOI (see Figure 5.15). Up to four soil samples (and associated QA/QC) will be collected at each boring. Soil samples will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals).

5.4.16 AOI 16 – FORMER EAST ELECTRICAL SUBSTATION

An electrical substation was located west of the aeration basin on the east side of GM Drive (see Figure 5.16). The transformer and visually stained surficial soil was removed by Cinergy Corporation (the local electric utility).

It has been determined that GM does not own this property. Cinergy owns and operated the property where the former electrical substation was located. Therefore,

this area is no longer considered an AOI. Former operations by GM near this location will be assessed through the investigation at AOI 6.

5.4.17 AOI 17 – PISTON BUILDING OIL ACCUMULATIONS

In late 1977, oil accumulations were identified in a ditch along the west side of the current Piston Building. The area west of the Piston Building is currently paved and there is no evidence of a former ditch.

Proposed Soil Investigation

Two soil borings are proposed to be advanced at this AOI (Figure 5.17). Up to four soil samples (and associated QA/QC) will be collected from each boring in accordance with Section 5.1.1 and will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals). It is anticipated that each boring will be advanced to a maximum of three feet into the bedrock, if possible.

5.4.18 AOI 18 – AREA AFFECTED BY THE HENRY SYSTEM DISCHARGE

On April 7, 1986, wastewater from the Henry System located in the Piston Building was accidentally discharged onto a concrete slab south of the building, entered a storm sewer and flowed down the steps leading into the WWTP (normally this discharge would have gone through a small service tunnel into the WWTP). The Henry System is located in the southern portion of the Piston Building. The Facility has made recent improvements to the Henry System and now reclaims the oil for reuse prior to sending the final discharge to the WWTP via a small service tunnel.

Proposed Soil Investigation

Two surficial soil samples (beneath the pavement and undercourse) are proposed to be collected to assess whether or not impacted materials are present (Figure 5.18). Each sample (and associated QA/QC) will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals).

5.4.19 AOI 19 – AREA AFFECTED BY THE PAINT AND THINNER SPILL

In May 1990, spent thinner and paint was spilled on the ground surface north of the concrete pad on the outside of the Paint and Thinner Storage Area at the Oil House. The Facility currently utilizes the Oil House for storing incoming virgin oils and paint materials. Waste paint and paint solvents are now stored in a metal, fire resistant cabinet located within the Waste Storage Area (AOI 2).

Proposed Soil Investigation

One soil boring will be advanced in this area (Figure 5.19). Soil samples will be collected in accordance with Section 5.1.1 (up to four samples per boring) and will be submitted to the laboratory for analysis of TCL VOCs and PCBs. It is anticipated that this boring will be advanced to a maximum of three feet into the bedrock, if possible.

5.4.20 AOI 20 – NORTHERN PORTION OF THE PISTON BUILDING

The Manifold Department was located in the northern portion of the current Piston Building. The Robotic Piston Department currently occupies this area.

Proposed Soil Investigation

One surficial soil sample (beneath the pavement and undercourse) is proposed to be collected to assess whether or not impacted materials remain (see Figure 5.20). This sample (and associated QA/QC) will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals).

5.4.21 AOI 21 – FILLED RAVINE NORTH OF THE DIE CAST BUILDING

Reviews of historic photographs (1945) indicate that a ravine existed north of the current Die Cast Building. The photograph also showed manufacturing activities, including product storage in this area. This area has been filled in to the same approximate elevation of the main plant and is currently paved or otherwise occupied by the Bulk Storage, Maintenance Sheet Metal and Fabrication Building, Chip Processing and Crusher Buildings, and the Alloy Storage Building. Chip system residue is still generated from the chip drying process. The residue is currently being disposed at a

licensed hazardous waste facility. The existing Equipment Storage Area is paved and is utilized for the storage of equipment.

Proposed Soil Investigation

Four soil borings will be advanced to assess the conditions of this AOI (see Figure 5.21). Soil samples will be collected in accordance with Section 5.1.1 (up to four samples per boring, exclusive of background soil samples) and will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals). It is anticipated that this boring will be advanced to a maximum of three feet into the bedrock, if possible.

5.4.22 AOI 22 – TOOL ROOM ANNEX DOCK RELEASE

A release of cleaner (Spray Kleen) to the Tool Room Annex Dock and adjacent unpaved rail bed occurred on March 21, 2000, when a tote containing the cleaner fell off of a fork truck and spilled in this area.

Proposed Soil Investigation

One surficial soil sample will be collected from the existing tracks area (Figure 5.22). This sample will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals).

5.4.23 AOI 23 – AREA AFFECTED BY THE 1996 WASTEWATER TREATMENT FILTER CAKE RELEASE

In October 1996, a release of filter cake waste liquid occurred on a gravel surface outside of the Sludge Building.

Proposed Soil Investigation

One surficial soil sample (beneath the pavement and undercourse) will be collected in the vicinity of the October 1996 release from this AOI (Figure 5.23). This sample will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals).

5.4.24 AOI 24 – AREA AFFECTED BY THE JUNE 2000 DIE LUBE 5150 RELEASE

A release of Die Lube 5150 (used on the die cast forms to allow for release of the parts) occurred near the Receiving Office in the Die Cast Building. The Facility continues to utilize the area near the Receiving Office in the Die Cast Building to store new totes awaiting usage.

Concrete Inspection

In order to assess whether or not the spill in this area may have entered the underlying soil, a visual inspection was completed to determine the integrity of the floors. The results of this inspection did not reveal the presence of any significant cracks, however, obvious saw cuts and a replacement section of concrete were observed.

Proposed Soil Sampling

Based on the results of the visual inspection, two soil samples (immediately below the concrete floor and undercourse) (Figure 5.24) will be collected and will be submitted to the laboratory for analysis of TCL VOCs, SVOCs, PCBs, and inorganics (TAL minus the earth metals).

6.0 DATA EVALUATION

6.1 OBJECTIVES

The purpose of the investigation is to determine whether potential risk to human health and the environment associated with hazardous waste or constituents released at AOIs identified for investigation warrants interim or corrective measures. Data collected during the investigation will be used for the following:

- Determine whether a significant release of hazardous constituents to soil, groundwater, surface water, or sediment has occurred at the AOIs that have not been previously investigated;
- Determine the nature and extent of constituents in soil and groundwater as necessary to support a baseline risk assessment, where a significant release of hazardous constituents has been confirmed;
- Support the RCRA corrective action Environmental Indicator Determinations; and
- Support the identification of AOIs that warrant interim or corrective measures, and evaluation of interim or corrective measures alternatives for these AOIs.

During the investigation, data collection will be conducted in phases. After each phase, adequacy of the data for these data uses will be evaluated as discussed below in Section 6.2 to determine whether additional data collection is warranted. When data of sufficient quality and quantity have been collected, the data will be used to support decisions regarding the need for interim or corrective measures as discussed in Section 6.3.

6.2 EVALUATION OF NEED FOR ADDITIONAL INVESTIGATION

The primary purpose of data collection during the investigation is to provide sufficient characterization of the nature and extent of any releases of constituents to allow a reliable quantification of potential exposures from AOI-related constituent concentrations. That is, the analytical data collected during the investigation must be adequate for:

- Determining whether the concentration of a constituent in an environmental medium at an AOI is significantly above background levels (i.e., levels not attributable to the Facility's operations) in that medium;
- Estimating the exposure concentration of a constituent in the medium in which the constituent was measured; and
- Estimating the exposure concentration of a constituent in a medium to which the constituent may migrate (e.g., volatilization of a soil constituent into ambient air).

To ensure adequacy for these intended uses, the analytical data will be evaluated in accordance with the procedures outlined in U.S.EPA guidance on baseline risk assessments (U.S.EPA 1989).

One element in the evaluation of the analytical data will be a qualitative review of the data with respect to adequacy of the samples in characterizing the average concentrations of constituents for each exposure pathway identified as potentially relevant during the investigation. The qualitative review, using professional judgment, will include an examination of the following:

- Consistency in the types of constituents found in all sampled media at each AOI vis-a-vis expectations based on history of operations and chemical properties of the constituents, which may indicate potential for false negative or false positive identification of constituents
- Lateral and vertical distribution of constituent concentrations to detect any obvious spatial trends, which may indicate that concentrations significantly higher than the measured concentrations may be likely in unsampled areas or depths
- Presence of unusually high constituent concentrations, which may indicate the presence of nonaqueous-phase liquids

If the qualitative review identifies conditions that are likely to cause risk-based assessments of the data to provide unreliable conclusions regarding the need for interim or corrective measure, further sampling or other actions (e.g., checking for laboratory errors) will be taken to address such conditions.

In conjunction with the qualitative review, the data will be evaluated using appropriate, conservative human health and ecological risk-based screening levels to identify potentially significant concentrations. This screening will be conducted on each data

point generated during the investigation. The presence of concentrations higher than screening levels will not necessarily mean that additional investigation is warranted. Similarly, the absence of concentrations higher than screening levels will not necessarily mean that additional investigation is unnecessary. Rather, decisions regarding the need for further investigation will be made based on professional judgment considering the screening results and results of the qualitative review discussed above, including the magnitude of the concentrations, their spatial distribution, and other factors (e.g., background levels, as discussed below).

The analytical data also will be reviewed to identify constituents that are present at concentrations in an environmental medium that are within background levels in that medium.

- For constituent concentrations measured in soil, the constituent concentrations will be compared with site-specific background levels in soil using a statistical prediction interval at a 0.01 level of significance. If a soil constituent is determined to be within background levels in soil, then it would not be considered for quantitative exposure assessment for soil-related exposure pathways.
- For constituents in groundwater, the concentration of a constituent in a monitoring well downgradient of an AOI will be compared with the maximum concentration measured in the monitoring wells upgradient of the Facility. If a groundwater constituent is determined to be within site-specific background levels in groundwater, then it would not be considered for quantitative exposure assessment for groundwater-related exposure pathways.
- For constituents in surface water, the concentration of a constituent in a surface water sample collected downstream of the Facility will be compared with the maximum concentration in the surface water samples collected upstream of the Facility. If a surface water constituent is determined to be within site-specific background levels in surface water, then it would not be considered for quantitative exposure assessment for surface-water-related exposure pathways.
- For constituents in sediment, the concentration of a constituent in a sediment sample collected downstream of the Facility will be compared with the maximum concentration in the sediment samples collected upstream of the Facility. If a sediment constituent is determined to be within site-specific background levels in sediment, then it would not be considered for quantitative exposure assessment for sediment-related exposure pathways.

Although all laboratory analytical data collected during the investigation will be validated as discussed in the QAPP, the evaluation of data after each phase of investigation will be conducted without waiting for data validation to be completed. This will allow for more timely decisions regarding the need for further field investigations. However, the use of data to support decisions regarding the need for interim or corrective measures will be based on validated data.

6.3 USE OF INVESTIGATION DATA

The objective of the investigation is to determine whether potential risk to human health and the environment associated with hazardous waste or constituents released from the AOIs identified for investigation warrants interim or corrective measures. The determination will rely on a risk-based assessment (which could be a baseline risk assessment) that will characterize the potential human health risk associated with each AOI from reasonable maximum exposures under current and reasonably expected future land and groundwater uses at and near the Facility. The ecological risk assessment will be conducted on a facility-wide basis taking into account the potential presence of threatened and endangered species, the presence of ecological populations and communities of valued ecological resources, habitat restrictions, and pathways of exposure for ecological receptors.

The risk assessments will include development of exposure scenarios, consistent with current and reasonably expected future land and groundwater uses, that describe potential exposure pathways by which on-site and off-site human and ecological populations may become exposed to constituents released from an AOI. Documentation to confirm reasonably expected future land and groundwater uses, the potential presence of threatened or endangered species, and the existing habitats will be developed during the investigation for the baseline risk assessments. The physical characteristics of the Facility, including topography, hydrology, hydrogeology, geology and ecology will be evaluated in conjunction with chemical data to assess chemical fate and transport mechanisms. This information will be used to assess the current and potential future impact, if any, of the releases identified at an AOI.

7.0 REPORTS

7.1 PROGRESS REPORTS

The following documents will be submitted, as indicated in the Agreement:

- the Current Conditions Report has already been submitted;
- the Progress Reports will continue to be submitted quarterly;
- the RFI Report will be submitted in sufficient time to support completion of other RFI documents;
- the Human Health Environmental Indicators Determination Form will be submitted by January 30, 2005; and
- the Migration of Contaminated Groundwater Environmental Indicators Determination Form will be submitted by April 7, 2007.

7.2 PROGRESS REPORTS

Progress reports will detail work performed to date, data collected, problems encountered, project schedule, and percent completed. Progress reports will be submitted to U.S.EPA by the 15th day of each month following a quarter.

Periodic meetings and/or conference calls will be scheduled with U.S.EPA to review progress and discuss findings to date

7.2 RFI REPORT

The objective of the RFI Report, as stated in the Agreement, is to:

"*Identify the nature and extent of any releases of hazardous waste and/or hazardous constituents at or from the Facility which may pose an unacceptable risk to human health and the environment. The report will also describe the nature and extent of any releases of hazardous waste and/or hazardous constituents at or from the Facility which do not pose an unacceptable risk to human health and the environment, and provide the basis for those conclusions, including an evaluation of the risks.*"

The RFI Report will present all data that have been gathered as part of the investigation, an analysis of the data, and conclusions about the status of the Facility and the need for corrective measures. The RFI Report will include the following information:

- a description of the various field activities performed during the course of the investigation;
- a description of the physical characteristics of the Facility, including topography, hydrology, hydrogeology, geology, and ecology;
- an identification of potential receptors;
- a description of the nature and extent of contamination, if any, associated with each AOI and the impact, if any, of the AOI on various environmental media at the Facility;
- a discussion of potential routes of migration for the contamination, if any, associated with each AOI and factors expected to affect contaminant migration;
- an evaluation of current and potential future risk, if any, to human health and the environment associated with each AOI under current and reasonably expected future land use scenarios;
- an identification of AOIs where interim measures are required to address current unacceptable risks to human health and the environment, if any;
- an identification of AOIs where final corrective measures are required to address any potential unacceptable future risk under reasonably expected future land use scenarios; and
- additional relevant information, including but not limited to field forms, analytical data, and risk assessment methods.

The data analysis and conclusions presented in the RFI Report will form the basis for the Environmental Indicators Report and Corrective Measures Proposal. Any data deficiencies identified in the RFI that are necessary to complete these reports will be identified and filled by the collection of additional data.

7.3 ENVIRONMENTAL INDICATORS REPORT

The objectives of the Environmental Indicators Report, as stated in the Agreement, are to demonstrate that:

"All current human exposures to contamination at or from the Facility are under control. That is, for all media known or reasonably suspected to be contaminated with hazardous wastes or hazardous constituents above risk-based levels, for which there are complete pathways between contamination and human receptors, significant or unacceptable exposures do not exist."

and

"Migration of contaminated groundwater at or from the Facility is stabilized. That is, the migration of all groundwater known or reasonably suspected to be contaminated with hazardous wastes or hazardous constituents above acceptable levels is stabilized to remain within any existing areas of contamination as defined by monitoring locations designated at the time of the demonstration. In addition, any discharge of groundwater to surface water is either insignificant or shown to be currently acceptable according to an appropriate interim assessment."

The Environmental Indicators Report will consist of two parts: Documentation of Environmental Indicator Determination on Current Human Exposures Under Control (CA725) and Documentation of Environmental Indicator Determination for Migration of Contaminated Groundwater Under Control (CA750). A Supplemental RFI Report will be submitted as necessary to provide any data gathered subsequent to preparation of the RFI Report to support these demonstrations and/or update the risk assessment to incorporate additional findings and evaluate the effects of any interim measures implemented. Any interim measures implemented will be documented in a separate report.

8.0 RFI SCHEDULE

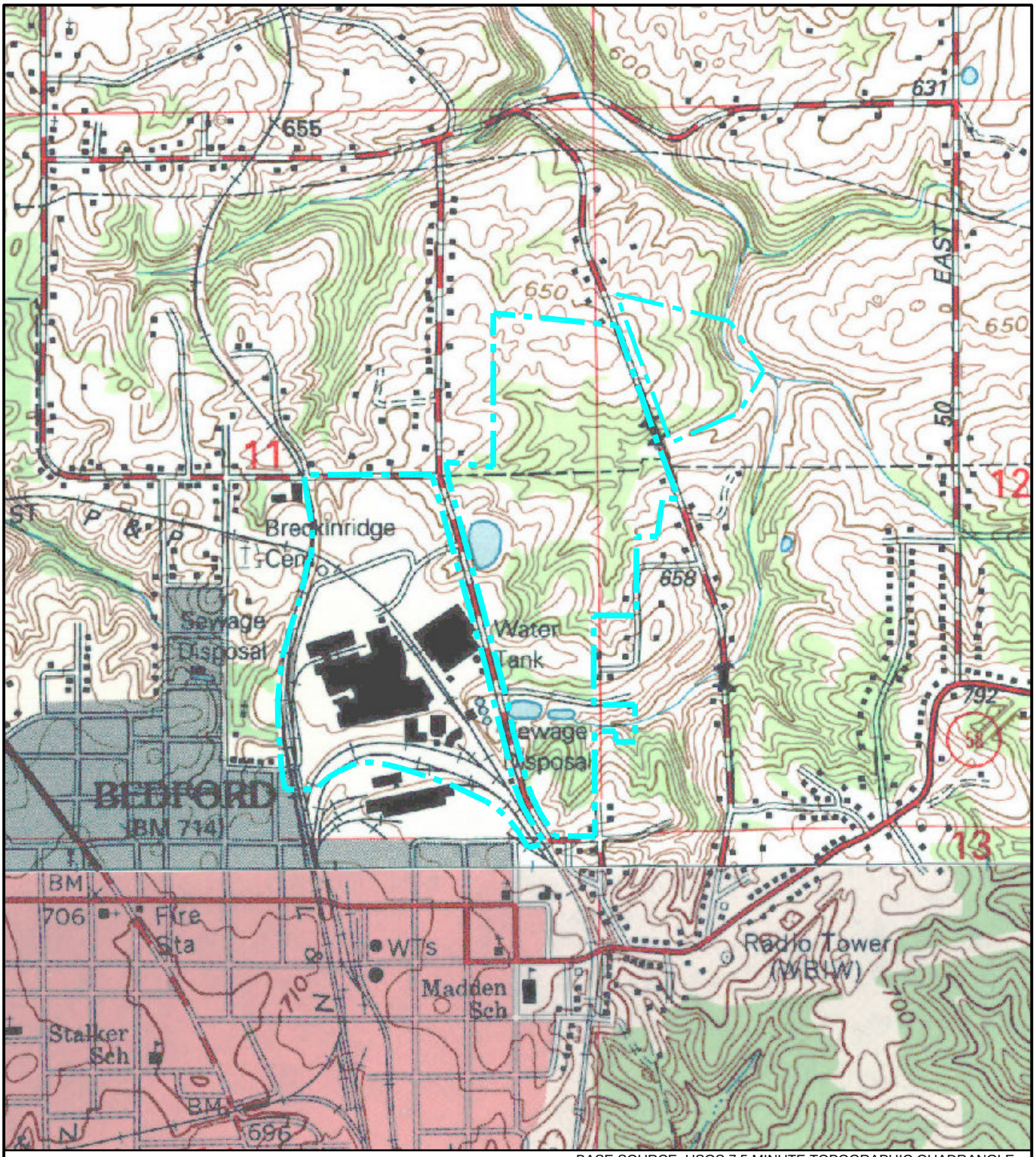
This RFI Work Plan will be completed in accordance with the schedule presented on Figure 8.1. Figure 8.2 presents the overall schedule for the RFI.

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LEGEND:

APPROXIMATE PROPERTY BOUNDARY



BASE SOURCE: USGS 7.5 MINUTE TOPOGRAPHIC QUADRANGLE;
BARTLETTSVILLE, INDIANA 1994
BEDFORD EAST, INDIANA 1978
BEDFORD WEST, INDIANA 1993
OOLITIC, INDIANA 1987

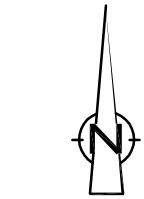
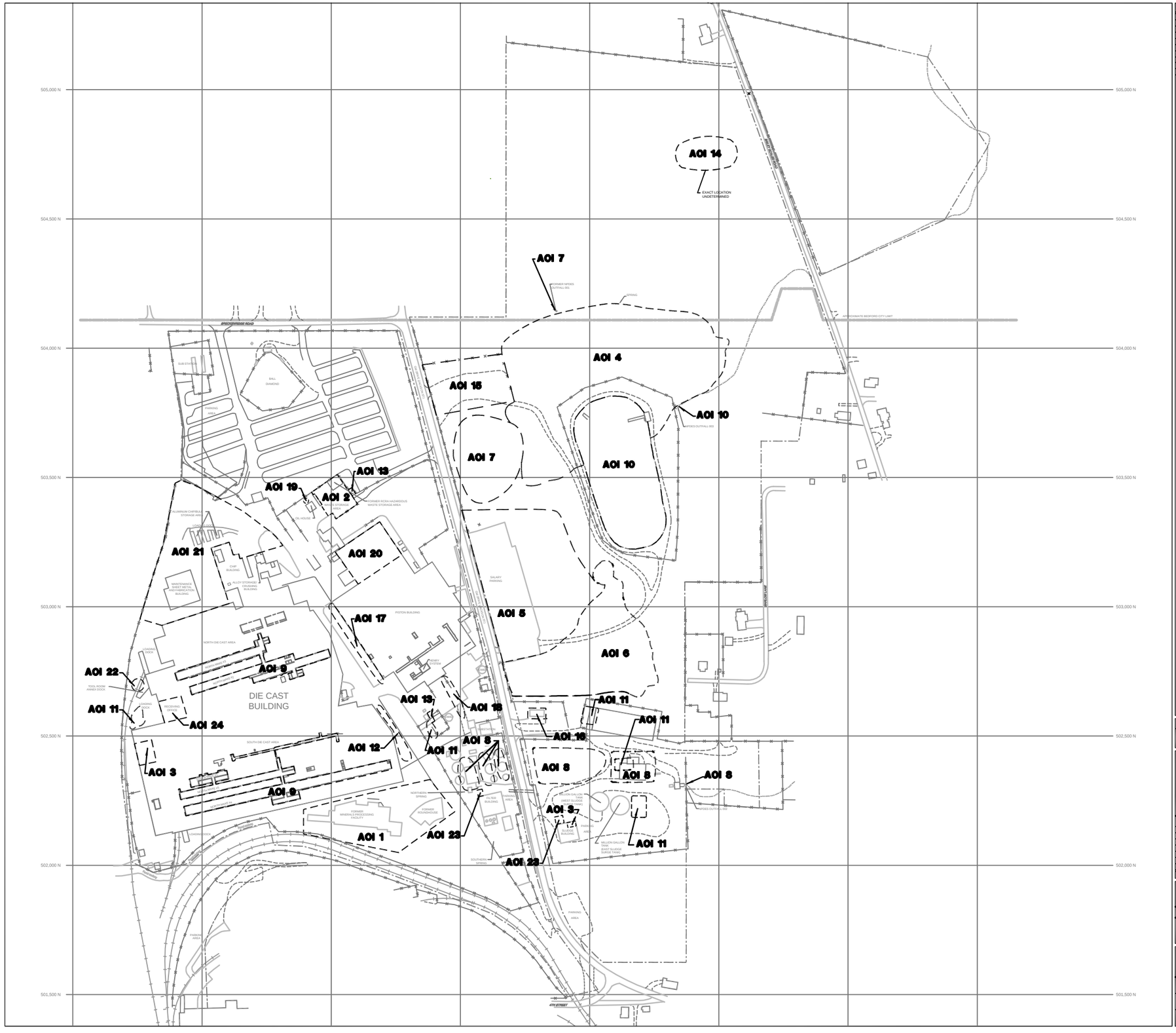


figure 1.1

SITE LOCATION
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO026 OCT 26/2001



No.	Revision	Date	Initial

0 50 100ft

LEGEND:

- APPROXIMATE AOI BOUNDARY
- APPROXIMATE PROPERTY BOUNDARY
- APPROXIMATE BEDFORD CITY LIMIT
- STREAMS
- FENCE LINE
- RAILROAD TRACKS
- DIRT ROADS
- ROADS / PAVED AREAS

AOI Summary

AOI ID Description

AOI 1 Former Railroad Operations and Minerals Processing Facility
AOI 2 Waste Storage Area
AOI 3 PCB Storage Areas
AOI 4 Former Sludge Disposal Area
AOI 5 Former East Sand Disposal Area
AOI 6 Former Sludge Disposal and Fire Training Area
AOI 7 Former North Lagoon and Outfall 001
AOI 8 Former South Lagoons and Outfall 002
AOI 9 Service Tunnels
AOI 10 Existing Stormwater Lagoon and Outfall 003
AOI 11 Aboveground Storage Tanks
AOI 12 Area Affected by the Reclaimed Hydraulic Fluid Release
AOI 13 Underground Storage Tanks
AOI 14 McBride Cows Disposal Area
AOI 15 Former Sludge Disposal Area
AOI 16 Former East Electrical Substation
AOI 17 Piston Building Oil Accumulations
AOI 18 Area Affected by the Henry System Discharge
AOI 19 Area Affected by Paint and Thinner Spill
AOI 20 Northern Portion of the Piston Building
AOI 21 Filled Ravine North of Die Cast Building
AOI 22 Tool Room Annex Dock Release
AOI 23 Area Affected by the 1996 Wastewater Treatment Filter Cake Release
AOI 24 Area Affected by the June 2000 Die Lube S150 Release

SCALE VERIFICATION

THIS BAR MEASURES 1" ON ORIGINAL. ADJUST SCALE ACCORDINGLY.

Approved

DRAWING STATUS

Status	Date	Initial
DRAFT FOR REVIEW		

GM POWERTRAIN BEDFORD PLANT
BEDFORD, INDIANA

GENERAL MOTORS POWERTRAIN DIVISION

SITE PLAN AND AOI LOCATIONS

CONESTOGA-ROVERS & ASSOCIATES

Source Reference:

Project Manager:	Reviewed By:	Date:
	DLR/AKS	AUGUST 21, 2001
Scale:	Project N.	Report N.	Drawing N.
as shown	13968-00	008	Figure 2.1

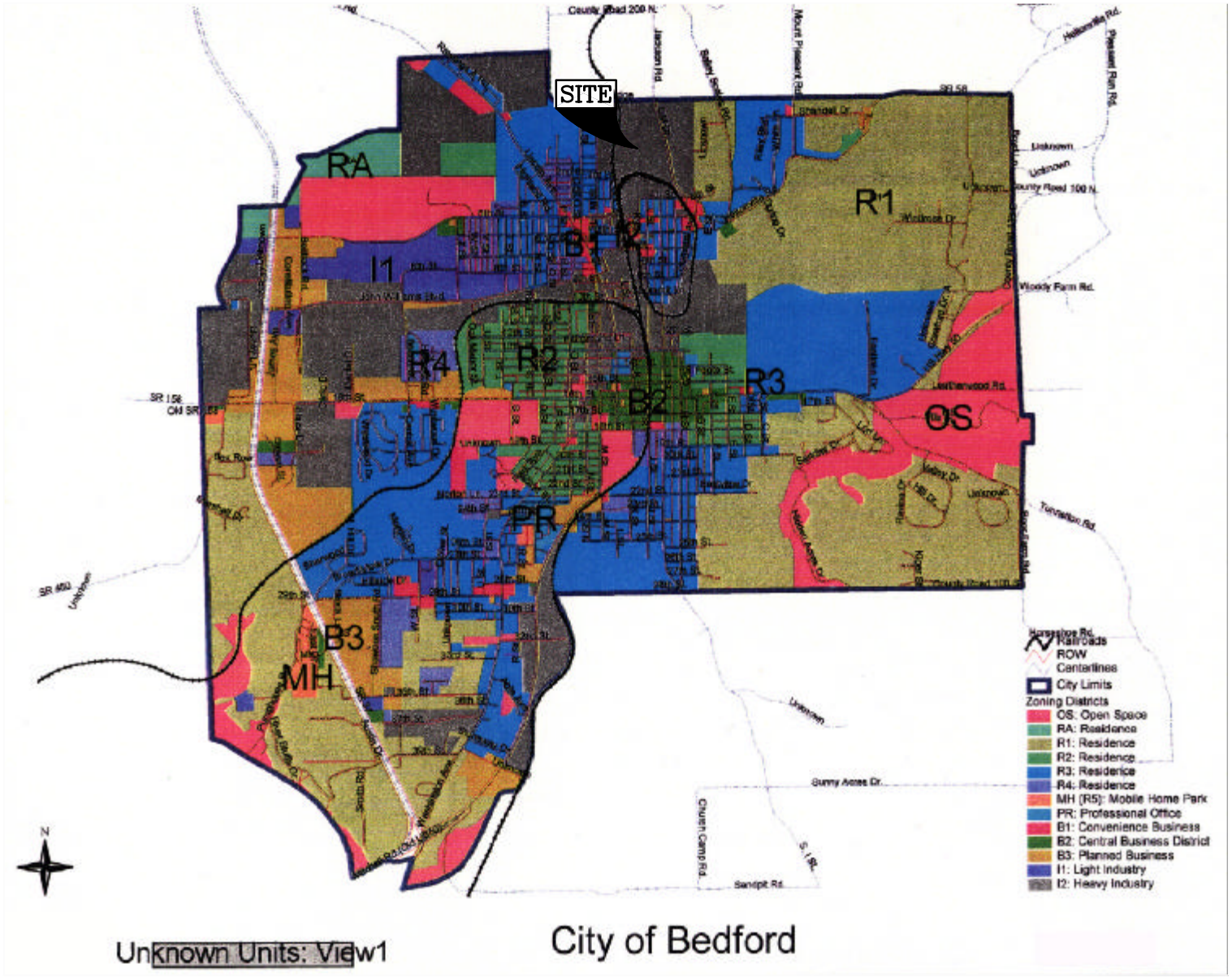


figure 3.1

LAND USE MAP
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

CRA

CITY OF BEDFORD, JULY 14, 2000



No.	Revision	Date	Initial

N

0 40 120ft

LEGEND:

- FORMER PLANT STRUCTURES
- PROPERTY BOUNDARY
- BEDFORD CITY LIMIT
- STREAMS
- FENCE LINE
- RAILROAD TRACKS
- DIRT ROADS
- ROADS / PAVED AREAS
- PROPOSED MONITORING WELL LOCATION
- ADDITIONAL MONITORING WELL LOCATION
- PROPOSED BACKGROUND SOIL SAMPLE LOCATION
- PROPOSED BORING LOCATION
- ADDITIONAL BORING LOCATION
- PROPOSED SURFACE SOIL SAMPLE LOCATION
- PROPOSED SUMP WATER SAMPLE LOCATION
- PROPOSED SURFACE WATER SAMPLE LOCATION
- PROPOSED SEDIMENT SAMPLE LOCATION
- PROPOSED CONCRETE WIPE SAMPLE LOCATION

AOI Summary

AOI ID Description

AOI 1 Former Railroad Operations and Minerals Processing Facility
AOI 2 Waste Storage Area
AOI 3 PCB Storage Areas
AOI 4 Former North Disposal Area
AOI 5 Former East Sand Disposal Area
AOI 6 Former Sludge Disposal and Fire Training Area
AOI 7 Former North Lagoon and Outfall 001
AOI 8 Former South Lagoons and Outfall 002
AOI 9 Service Tunnels
AOI 10 Former Wastewater Lagoon and Outfall 003
AOI 11 Aboveground Storage Tanks
AOI 12 Area Affected by the Reclaimed Hydraulic Fluid Release
AOI 13 Underground Storage Tanks
AOI 14 McBride Cows Disposal Area
AOI 15 Former Equipment Storage Area
AOI 16 Former East Electrical Substation
AOI 17 Piston Building Oil Accumulations
AOI 18 Area Affected by the Henry System Discharge
AOI 19 Area Affected by Paint and Thinner Spill
AOI 20 Northern Portion of the Piston Building
AOI 21 Filled Ravine North of Die Cast Building
AOI 22 Tool Room Annex Dock Release
AOI 23 Area Affected by the 1996 Wastewater Treatment Filter Cake Release
AOI 24 Area Affected by the June 2000 Die Lube 5150 Release

SCALE VERIFICATION

THIS BAR MEASURES 1" ON ORIGINAL. ADJUST SCALE ACCORDINGLY.

Approved

DRAWING STATUS

Status	Date	Initial
DRAFT FOR REVIEW		

GM POWERTRAIN
BEDFORD PLANT

RFI INVESTIGATION

PROPOSED SAMPLE LOCATIONS

CONESTOGA-ROVERS & ASSOCIATES

Source Reference:

Project Manager:	Reviewed By:	Date:
D.V.	S.V.	APRIL 2002
Scale:	Project N.°:	Report N.°: / Drawing N.°:
as shown	13968-00	PRES006 / 2

13968-00(PRES006)(GN-WA)02 APR 10/2002

REA

DIE CAST AREA

NORTH MAKE #4

AOI 9

AOI 8

CHEMICAL BUILDING

NORTHERN SPRING

FILTER BUILDING

FORMER ROUNDHOUSE

FORMER MINERALS PROCESSING FACILITY

AOI 1

SOUTHERN SPRING

LEGEND

APPROXIMATE AOI BOUNDARY

PROPERTY BOUNDARY

FENCE LINE

RAILROAD TRACKS

PROPOSED MONITORING WELL LOCATION

PROPOSED BACKGROUND SOIL SAMPLE LOCATION

PROPOSED BORING LOCATION

PROPOSED SURFACE WATER SAMPLE LOCATION

PROPOSED SURFACE SOIL SAMPLE LOCATION

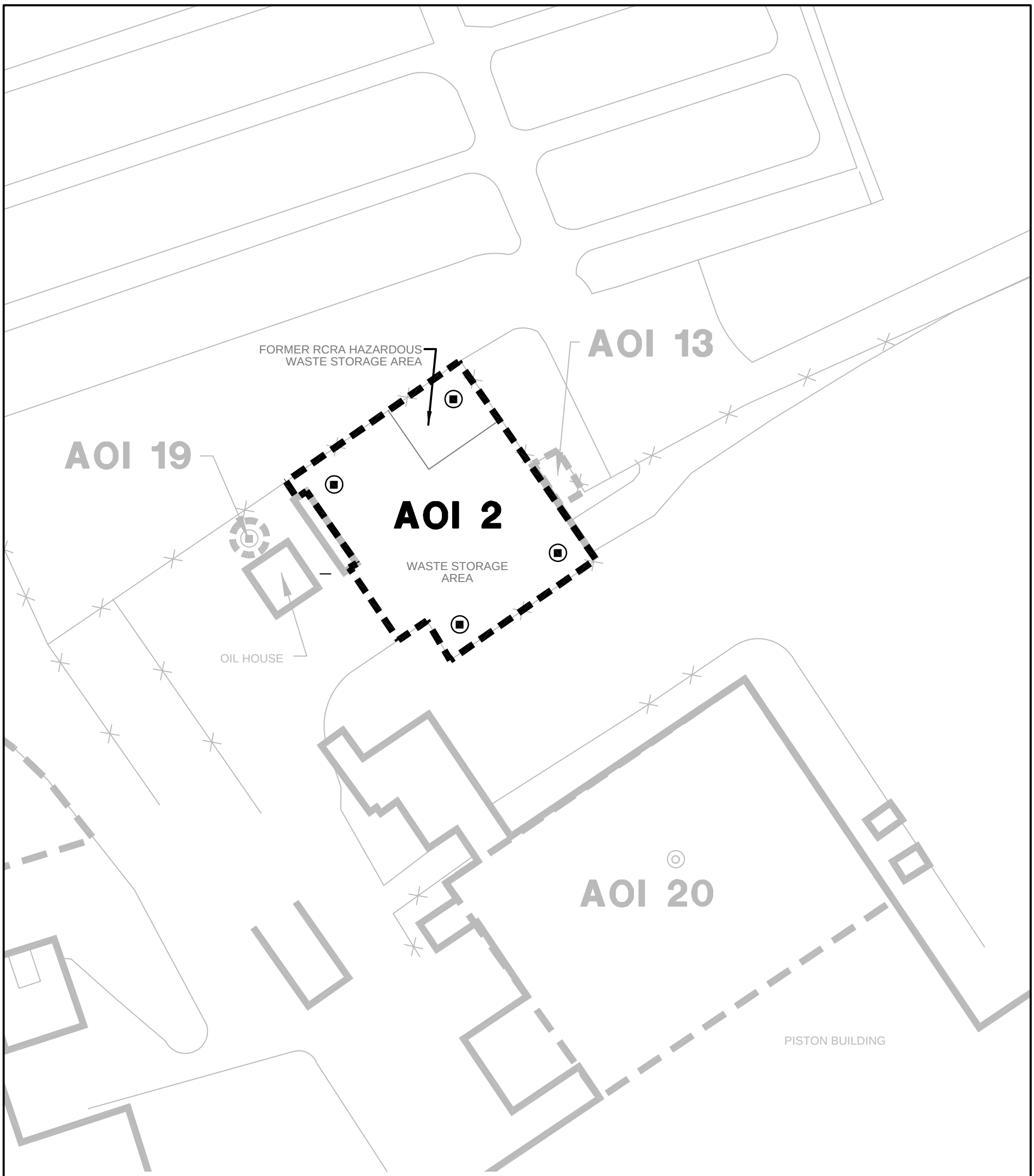
N

0 20 60ft

figure 5.1

AOI 1 - FORMER RAILROAD OPERATIONS AND MINERALS PROCESSING FACILITY
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO001 OCT 24/2001



LEGEND

- APPROXIMATE AOI BOUNDARY
- FENCE LINE
- ROADS / PAVED AREAS
- PROPOSED BORING LOCATION
- PROPOSED SURFACE SOIL SAMPLE LOCATION

N

0 20 60ft

figure 5.2

AOI 2 - WASTE STORAGE AREA
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

CRA

13968-00(008)GN-CO002 OCT 25/2001

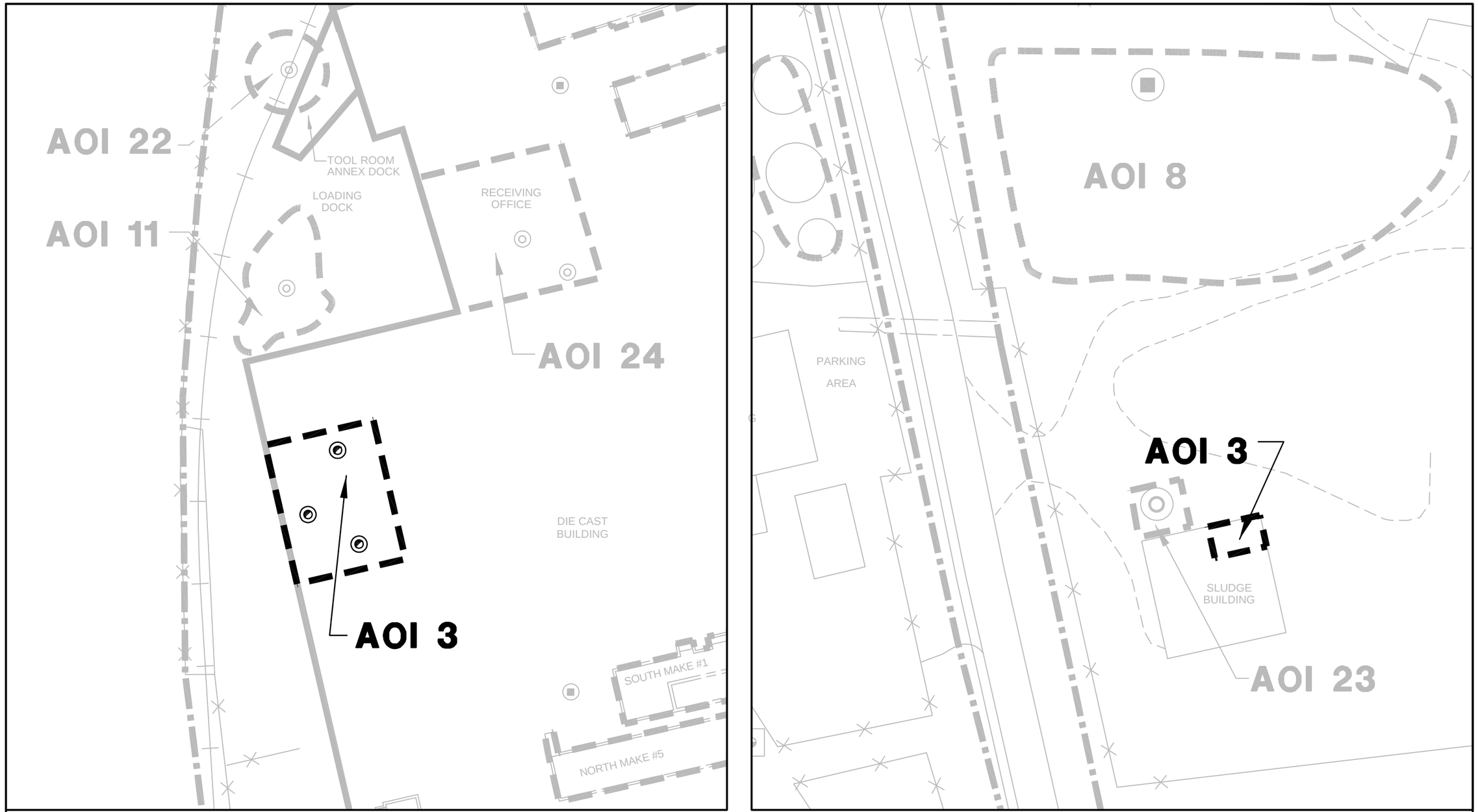


figure 5.3

AOI 3 - PCB STORAGE AREAS
PROPOSED CONCRETE SAMPLING
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO003 OCT 25/2001

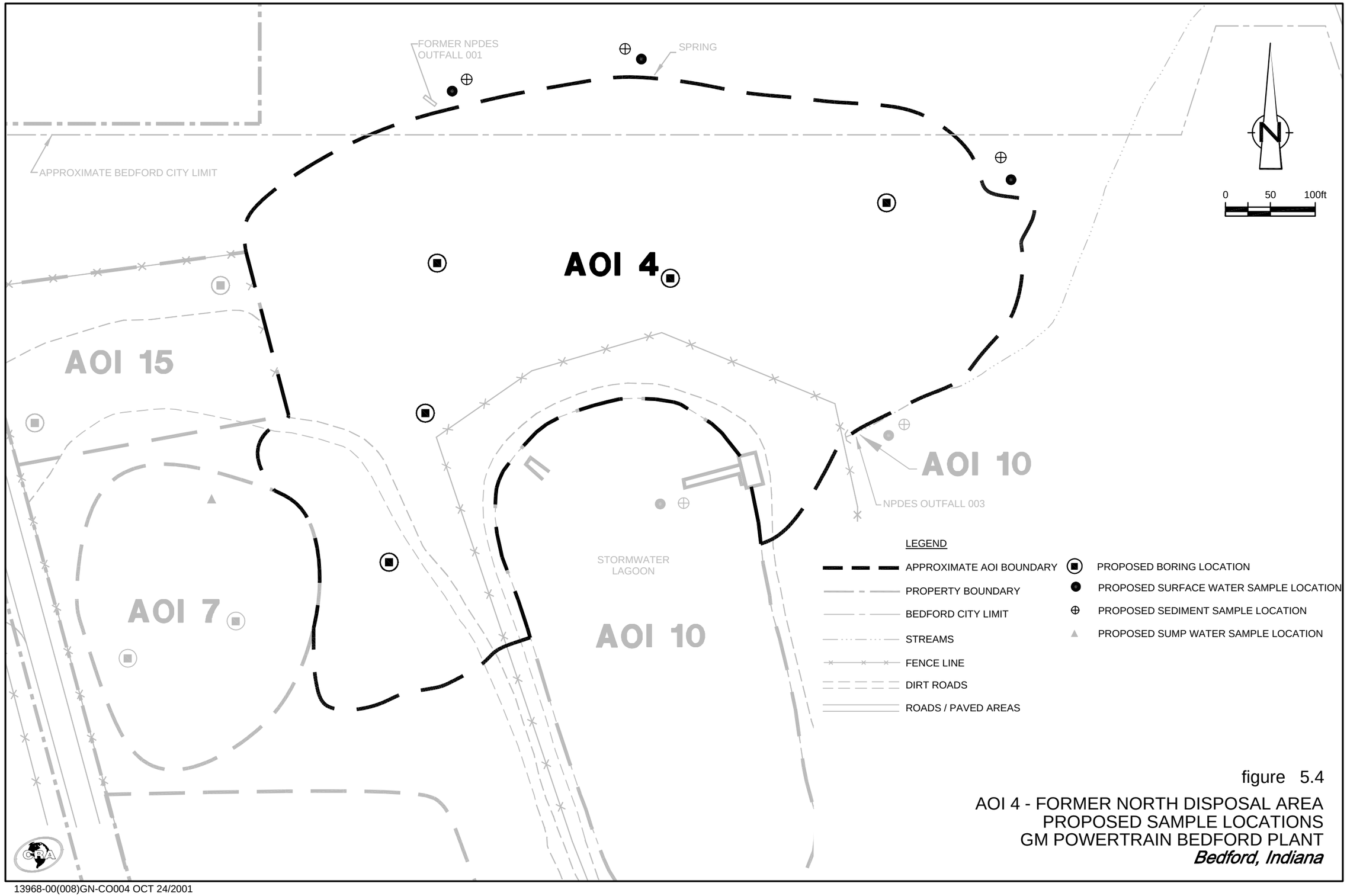


figure 5.4

AOI 4 - FORMER NORTH DISPOSAL AREA
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO004 OCT 24/2001



figure 5.5

AOI 5 - FORMER EAST SAND DISPOSAL AREA
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO005 OCT 15/2001

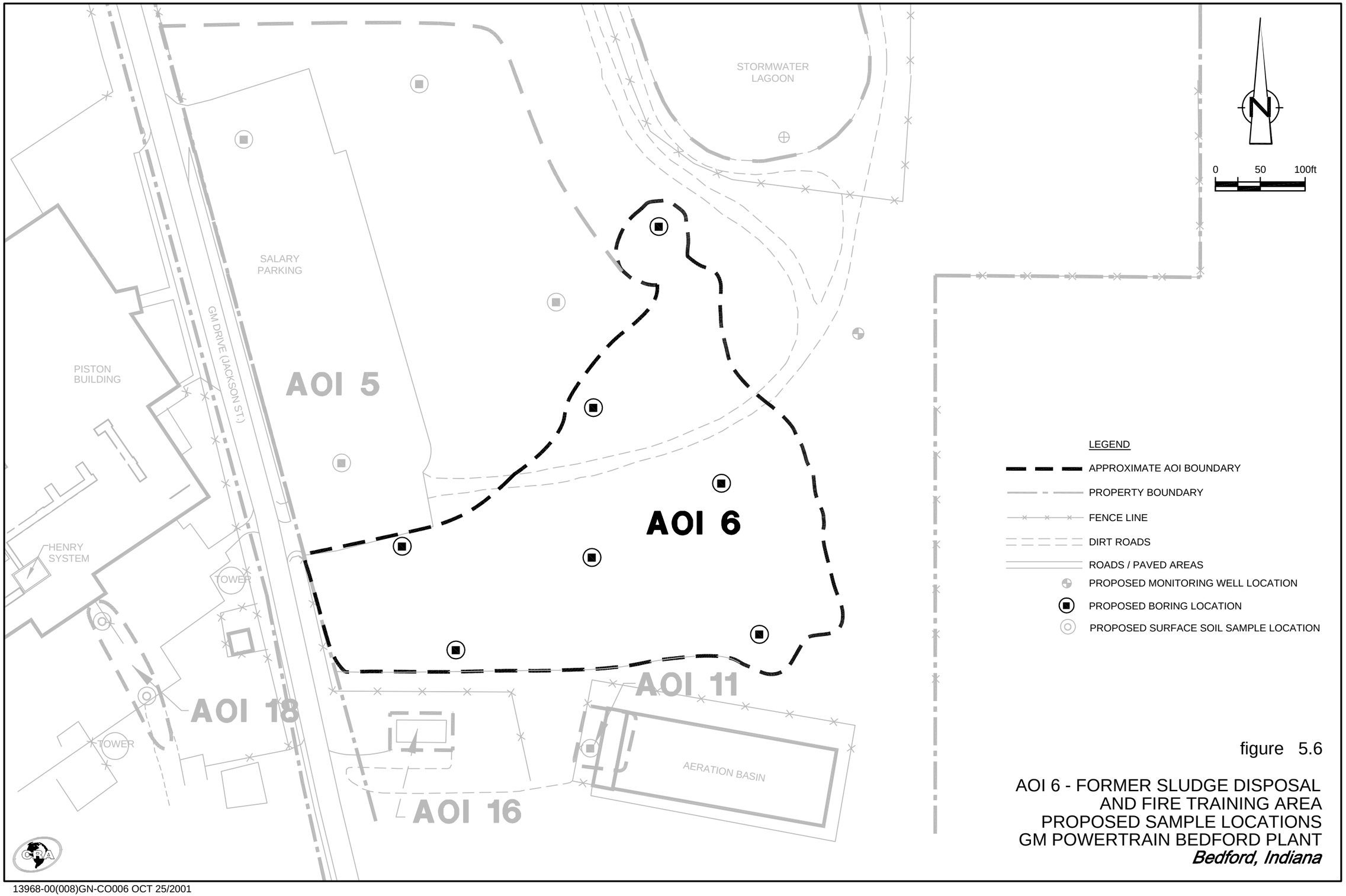


figure 5.6

AOI 6 - FORMER SLUDGE DISPOSAL AND FIRE TRAINING AREA
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO006 OCT 25/2001

AOI 7

FORMER NPDES
OUTFALL 001

AOI 4

GM DRIVE (JACKSON ST.)

AOI 15

AOI 7

AOI 10

STORMWATER
LAGOON

AOI 5

LEGEND

- APPROXIMATE AOI BOUNDARY
- PROPERTY BOUNDARY
- BEDFORD CITY LIMIT
- FENCE LINE
- DIRT ROADS
- ROADS / PAVED AREAS
- PROPOSED MONITORING WELL LOCATION
- PROPOSED BACKGROUND SOIL SAMPLE LOCATION
- PROPOSED BORING LOCATION
- PROPOSED SUMP WATER SAMPLE LOCATION
- PROPOSED SURFACE WATER SAMPLE LOCATION
- PROPOSED SEDIMENT SAMPLE LOCATION

N

0 50 100ft

figure 5.7

AOI 7 - FORMER NORTH LAGOON AND OUTFALL 001
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO007 OCT 25/2001

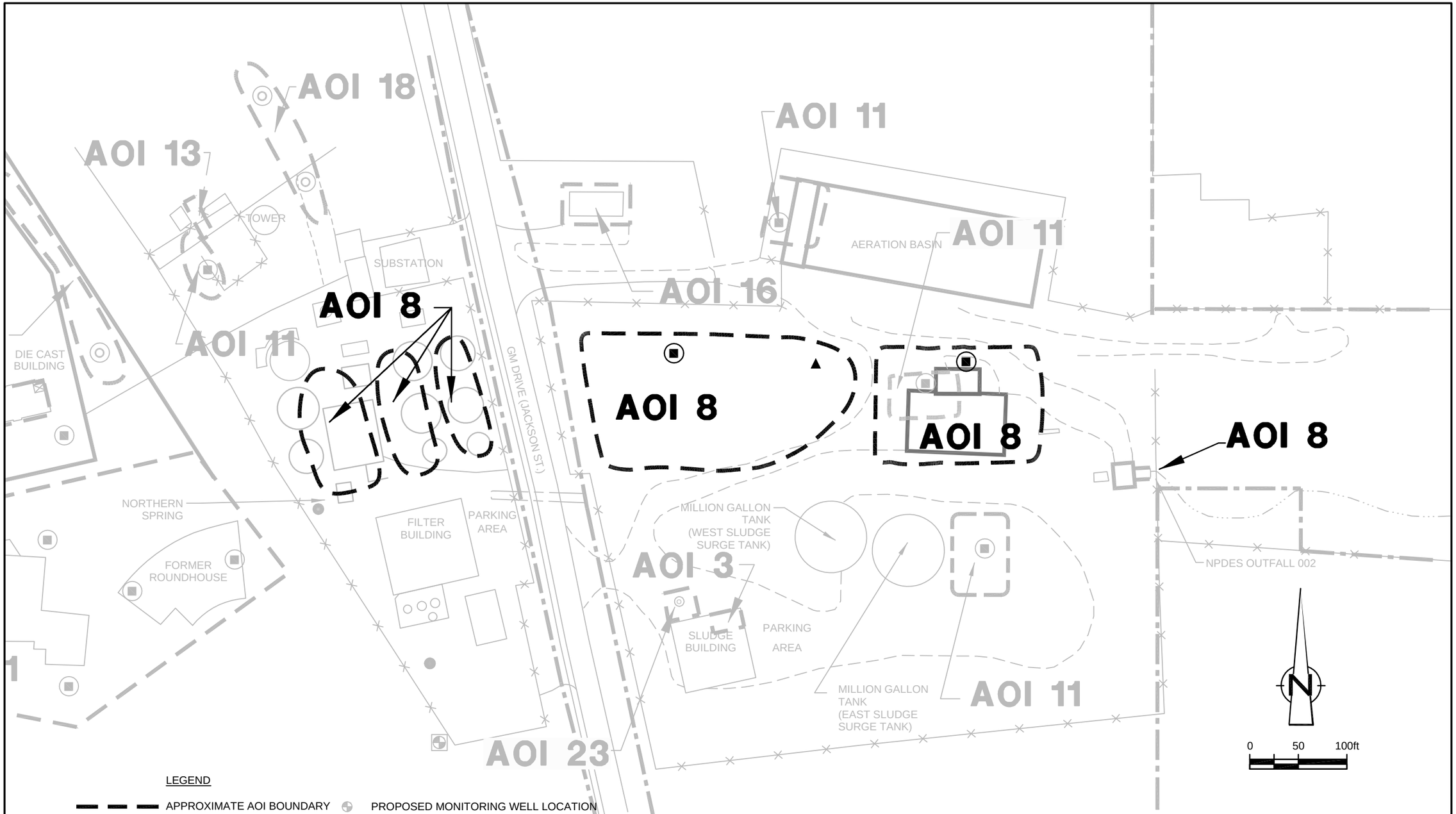


figure 5.8

AOI 8 - FORMER SOUTH LAGOONS AND OUTFALL 002
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO008 OCT 25/2001

MAINTENANCE SHEET METAL AND FABRICATION BUILDING

AOI 17

PISTON BUILDING

NORTH DIE CAST AREA

NORTH MAKE #3

NORTH MAKE #2

AOI 9

DIE CAST BUILDING

RECEIVING OFFICE

AOI 24

SOUTH DIE CAST AREA

NORTH MAKE #1

AOI 12

SOUTH MAKE #1

NORTH MAKE #5

AOI 9

NORTH MAKE #4

FORMER MINERALS PROCESSING FACILITY

LOADING DOCK

Paved roadway

Paved roadway

AOI 1

LEGEND

APPROXIMATE AOI BOUNDARY

PROPERTY BOUNDARY

FENCE LINE

RAILROAD TRACKS

ROADS / PAVED AREAS

PROPOSED MONITORING WELL LOCATION

PROPOSED BORING LOCATION

PROPOSED SURFACE SOIL SAMPLE LOCATION

N

0 50 100ft

figure 5.9

AOI 9 - SERVICE TUNNELS
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO009 OCT 25/2001

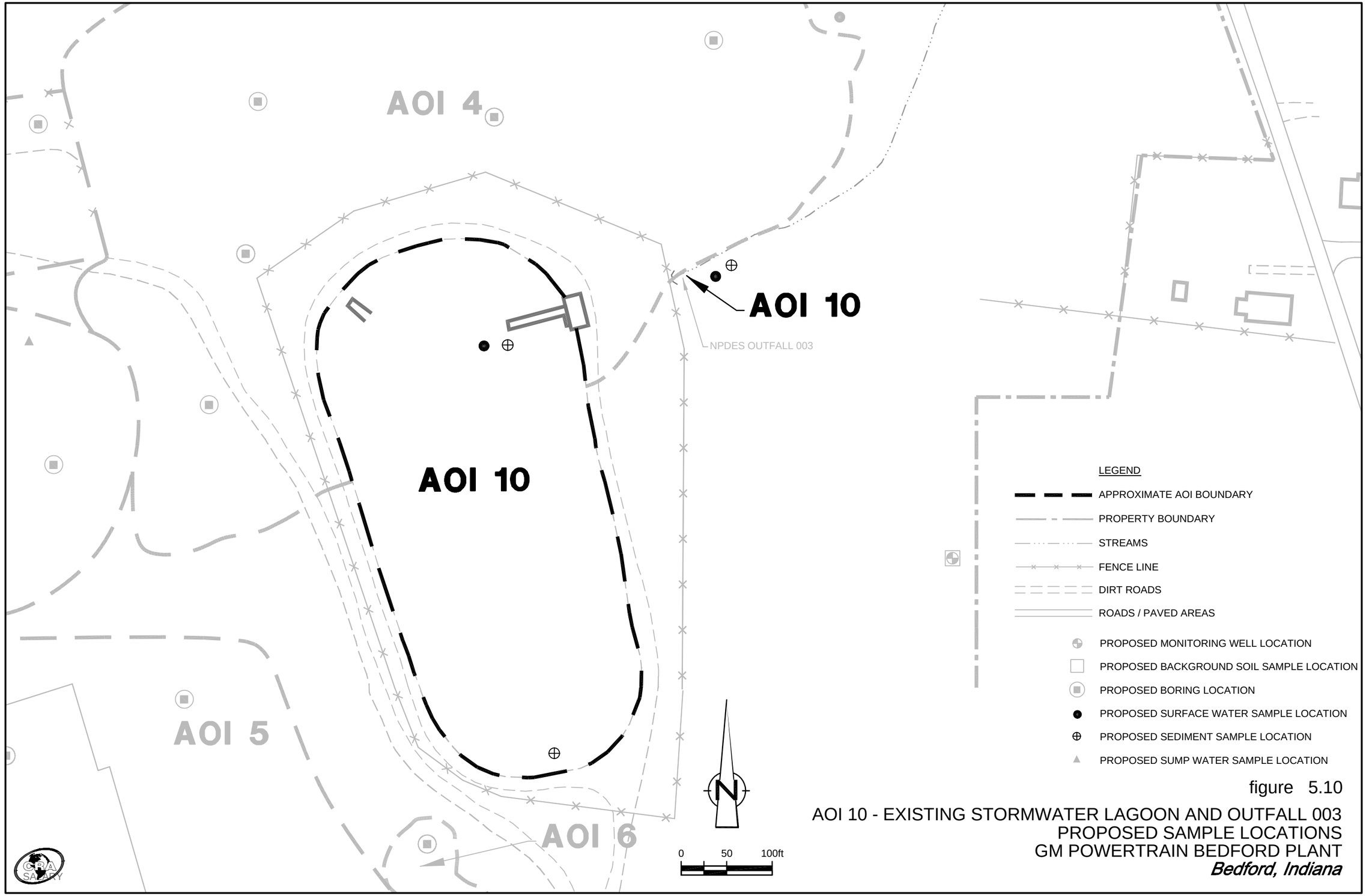
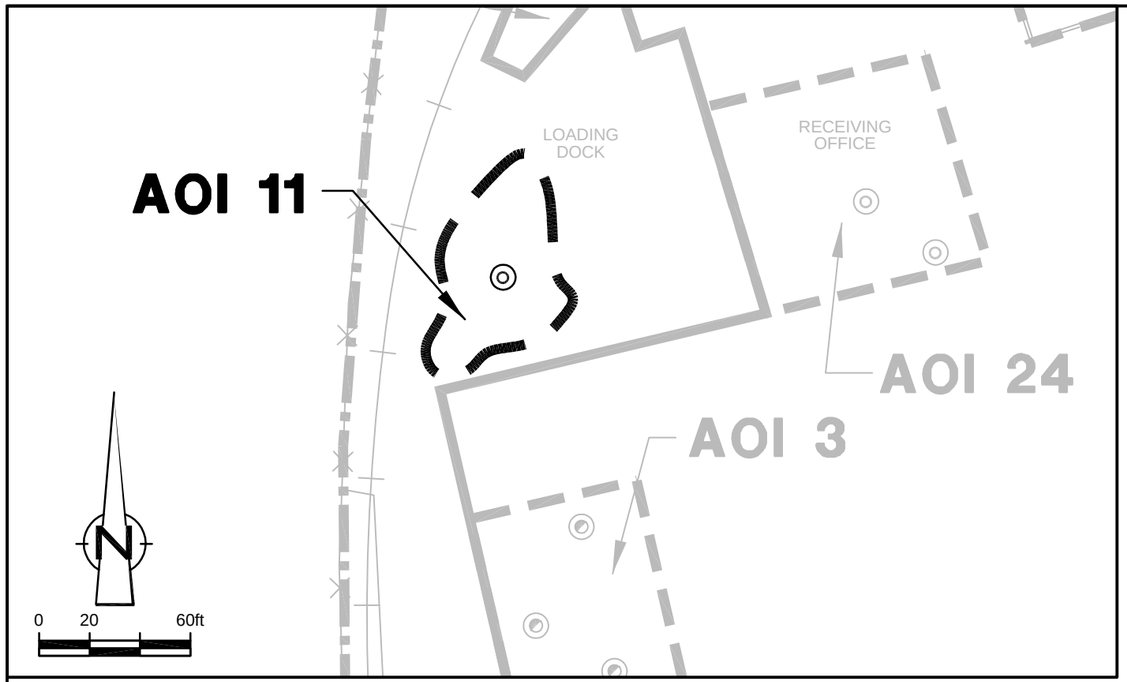


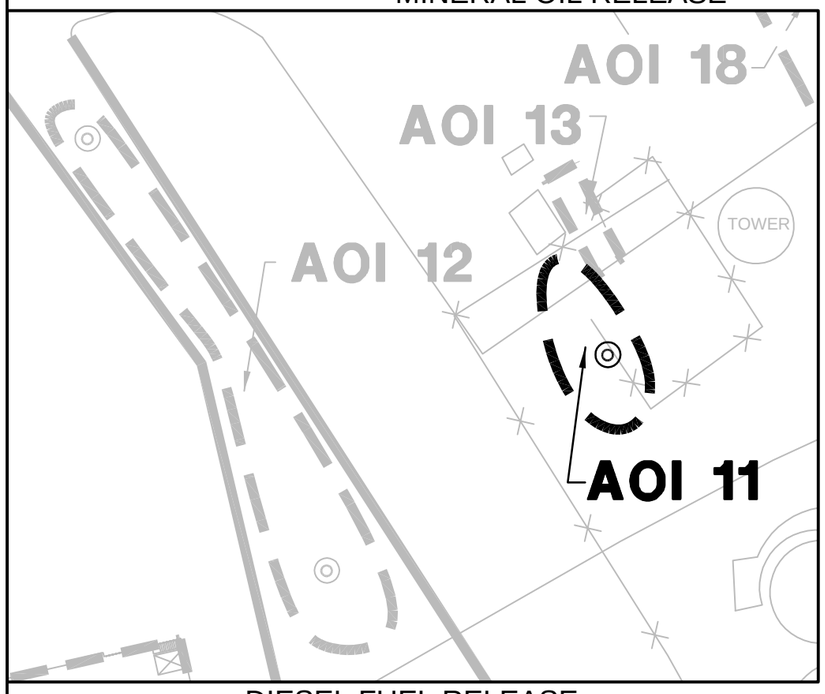
figure 5.10

AOI 10 - EXISTING STORMWATER LAGOON AND OUTFALL 003
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

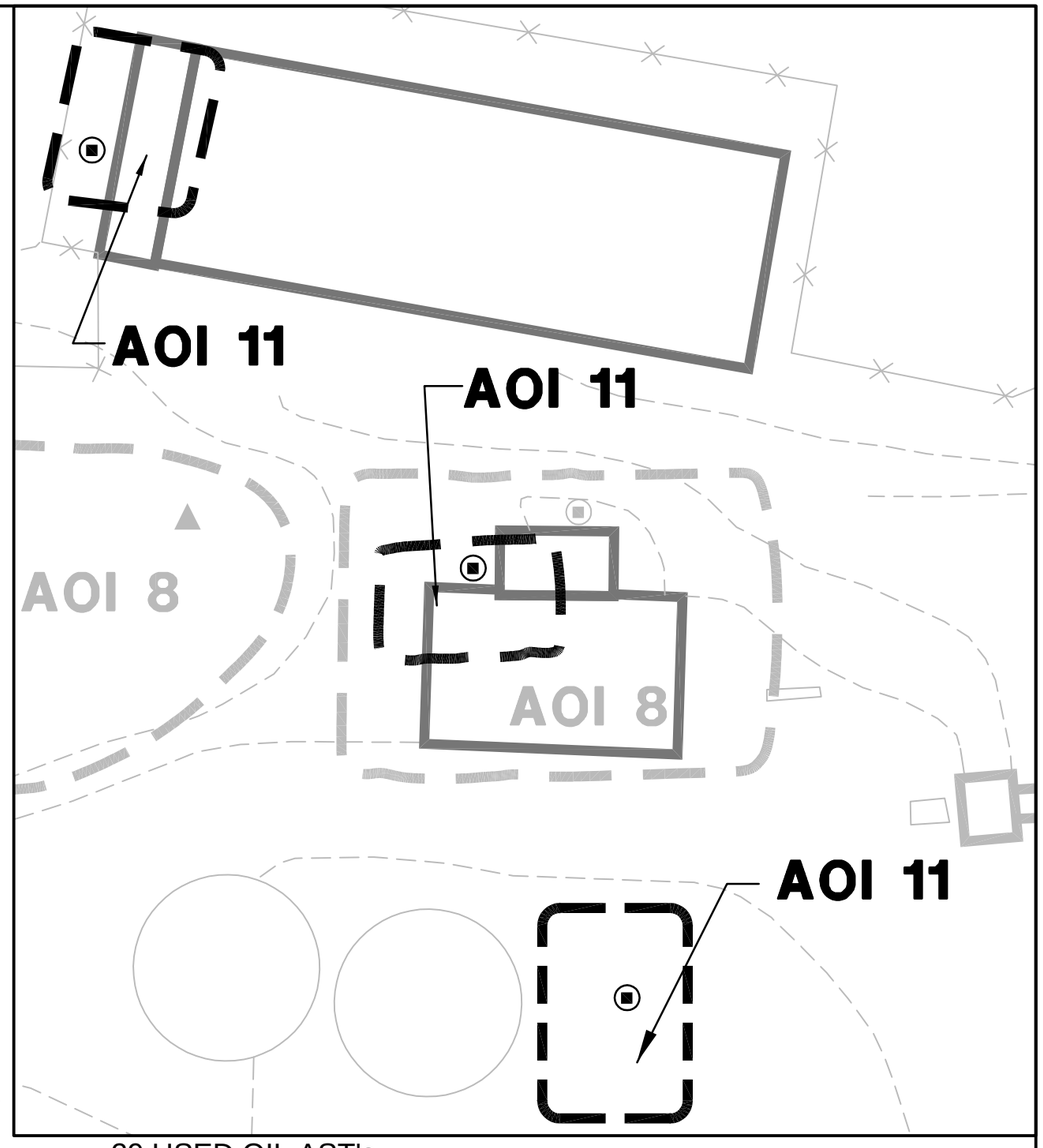
13968-00(008)GN-CO010 OCT 25/2001



MINERAL OIL RELEASE



DIESEL FUEL RELEASE



29 USED OIL AST's

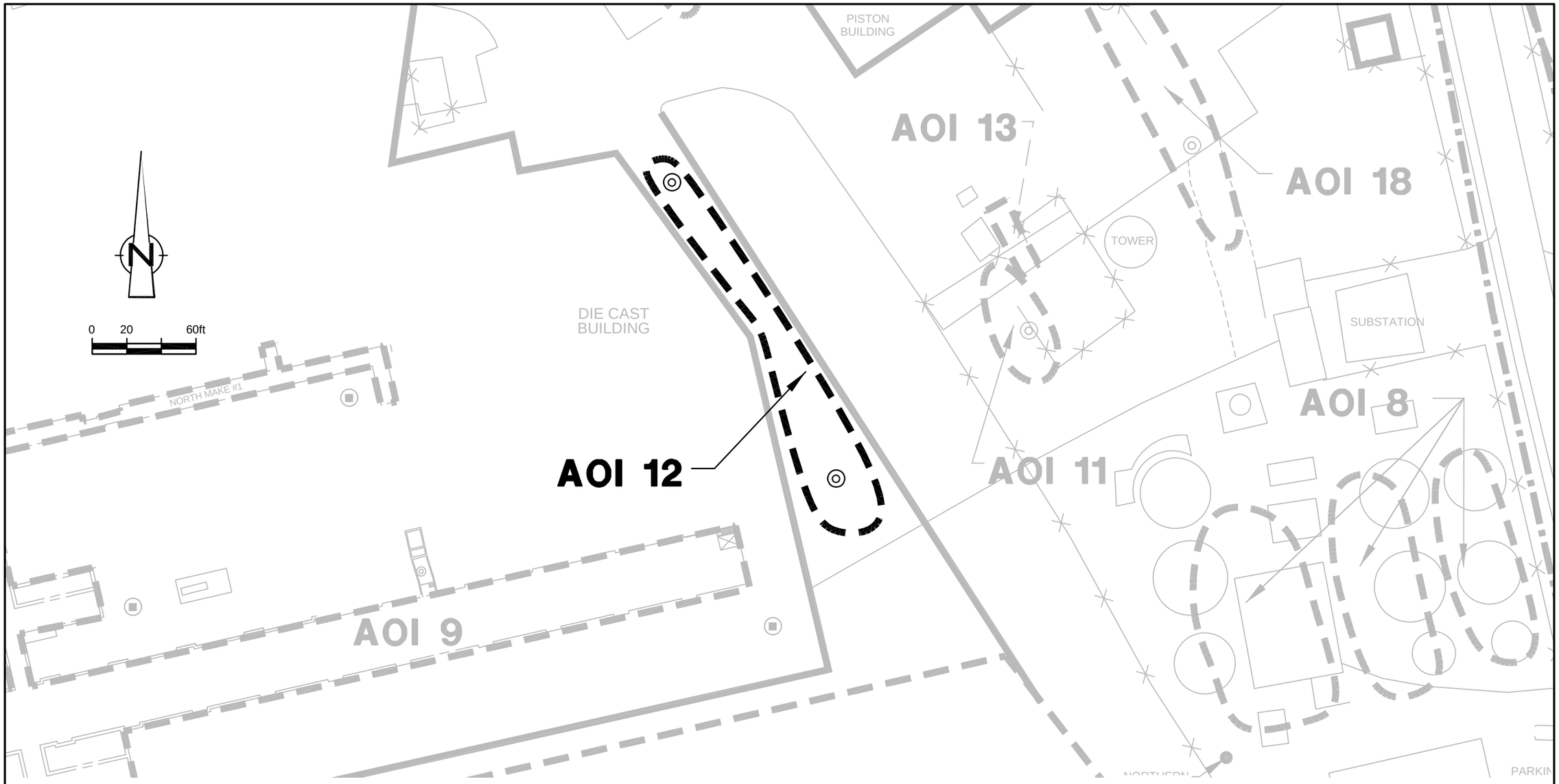
LEGEND

- APPROXIMATE AOI BOUNDARY
- PROPERTY BOUNDARY
- RAILROAD TRACKS
- FENCE LINE
- DIRT ROADS
- ROADS / PAVED AREAS
- PROPOSED BORING LOCATION
- PROPOSED SURFACE SOIL SAMPLE LOCATION
- PROPOSED SUMP WATER SAMPLE LOCATION
- PROPOSED CONCRETE WIPE SAMPLE LOCATION

figure 5.11

AOI 11 - ABOVEGROUND STORAGE TANKS
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO011 OCT 25/2001



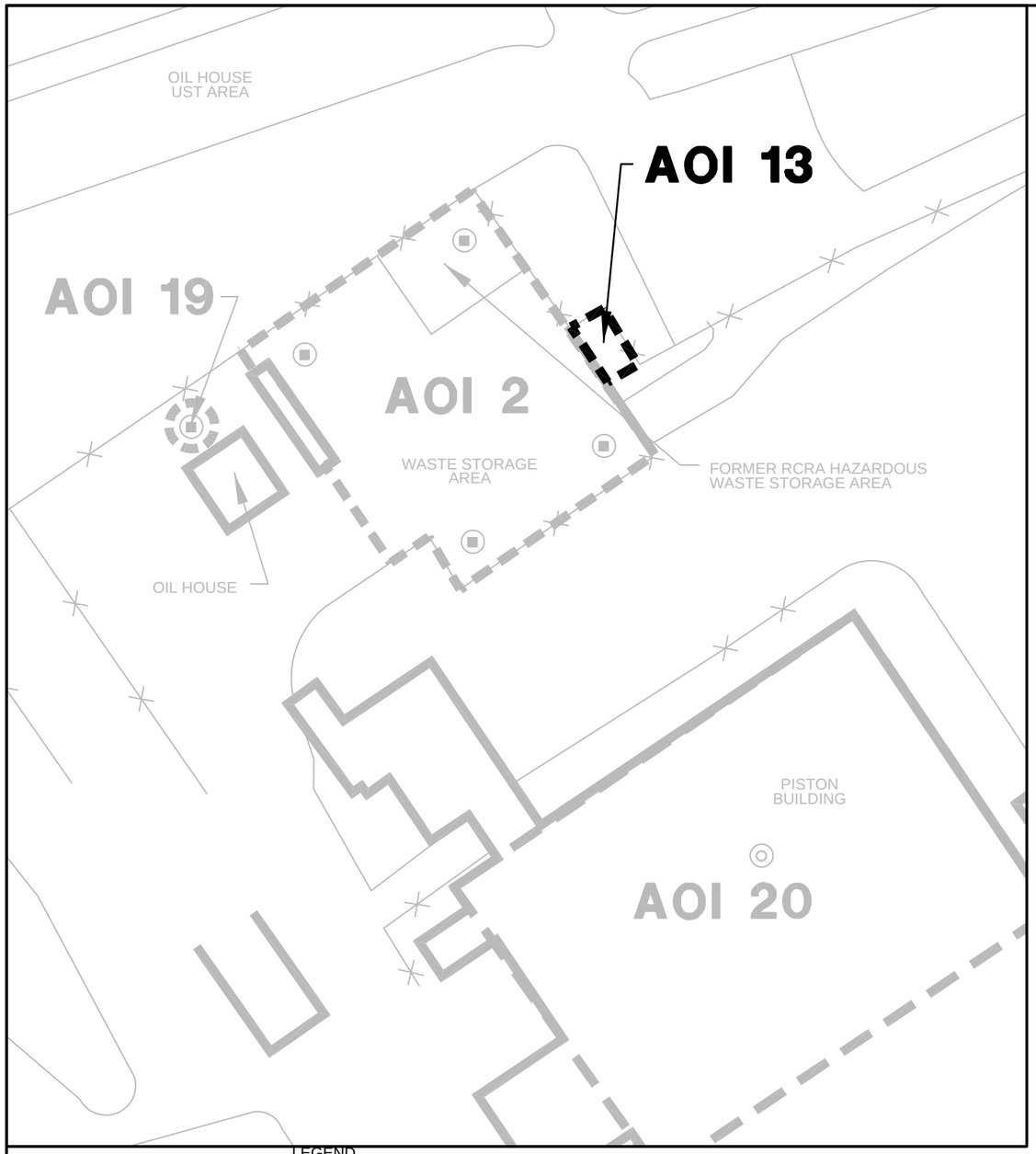
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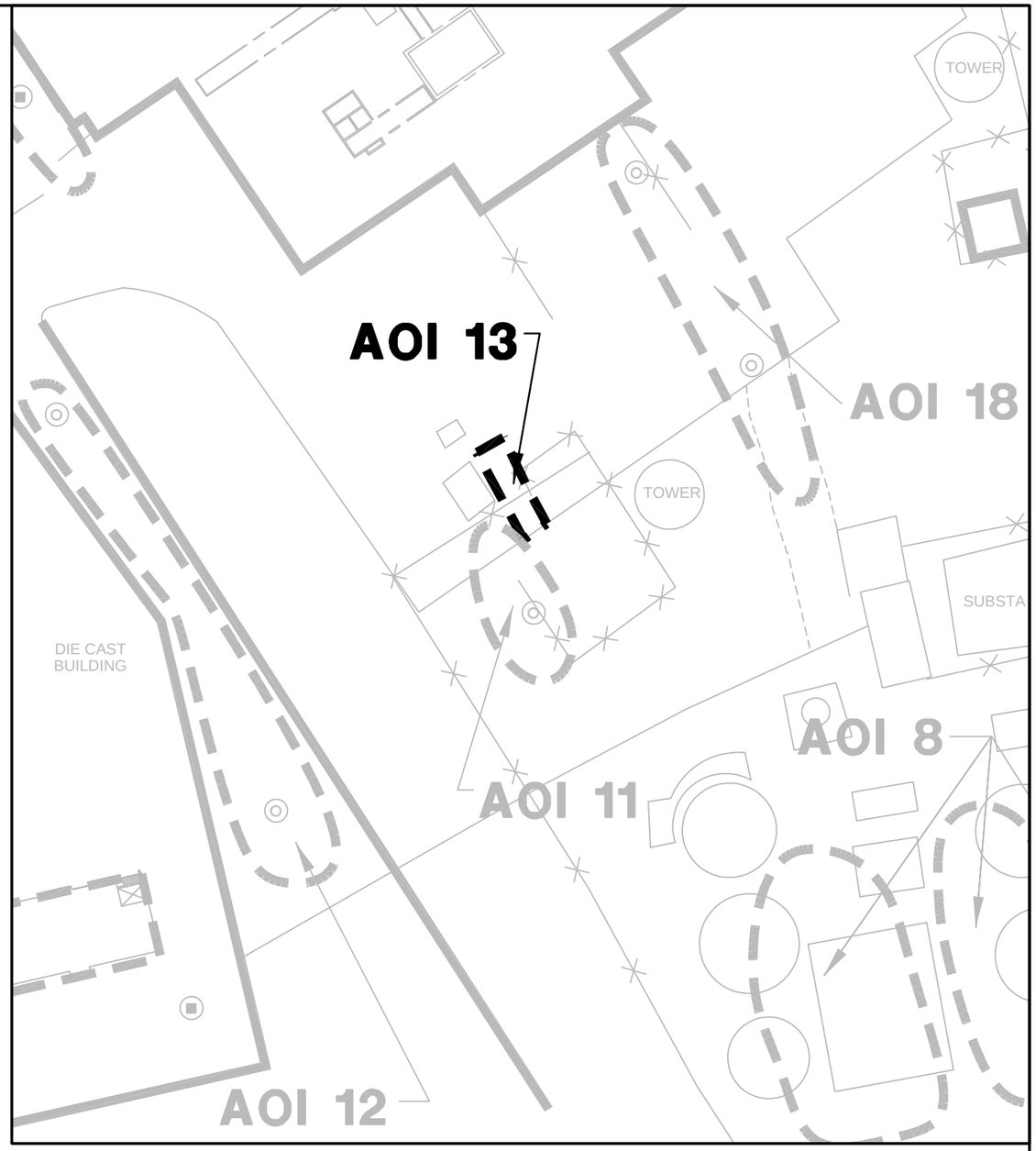
- APPROXIMATE AOI BOUNDARY
- PROPERTY BOUNDARY
- FENCE LINE
- ROADS / PAVED AREAS
- PROPOSED BORING LOCATION
- PROPOSED SURFACE WATER SAMPLE LOCATION
- PROPOSED SURFACE SOIL SAMPLE LOCATION

figure 5.12

AOI 12 - AREA AFFECTED BY THE RECLAIMED HYDRAULIC FLUID RELEASE
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO012 OCT 25/2001





LEGEND

- APPROXIMATE AOI BOUNDARY
- FENCE LINE
- ROADS / PAVED AREAS
- PROPOSED BORING LOCATION
- PROPOSED SURFACE SOIL SAMPLE LOCATION

N

0 20 60ft

figure 5.13

AOI 13 - UNDERGROUND STORAGE TANKS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO013 OCT 25/2001

N

0 50 100ft

LEGEND

- APPROXIMATE AOI BOUNDARY
- PROPERTY BOUNDARY
- STREAMS
- FENCE LINE
- ROADS / PAVED AREAS
- PROPOSED MONITORING WELL LOCATION
- PROPOSED BACKGROUND SOIL SAMPLE LOCATION
- PROPOSED BORING LOCATION
- PROPOSED SURFACE WATER SAMPLE LOCATION
- PROPOSED SEDIMENT SAMPLE LOCATION

AOI 14

EXACT LOCATION
UNDETERMINED

BAILEY SCALES ROAD

AOI 7

FORMER NPDES
OUTFALL 001

SPRING

figure 5.14

AOI 14 - Mc BRIDE COWS DISPOSAL AREA
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO014 OCT 25/2001

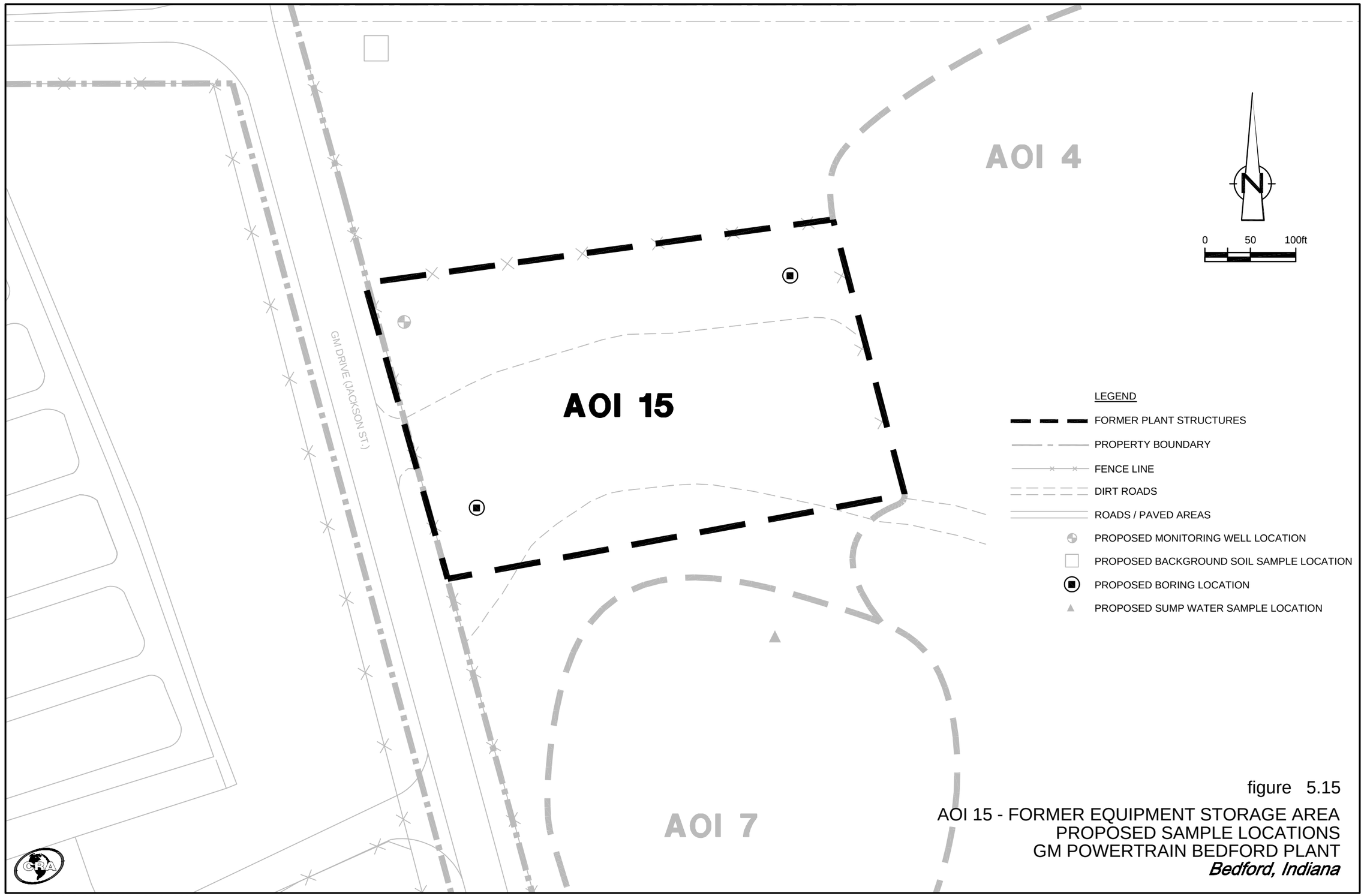
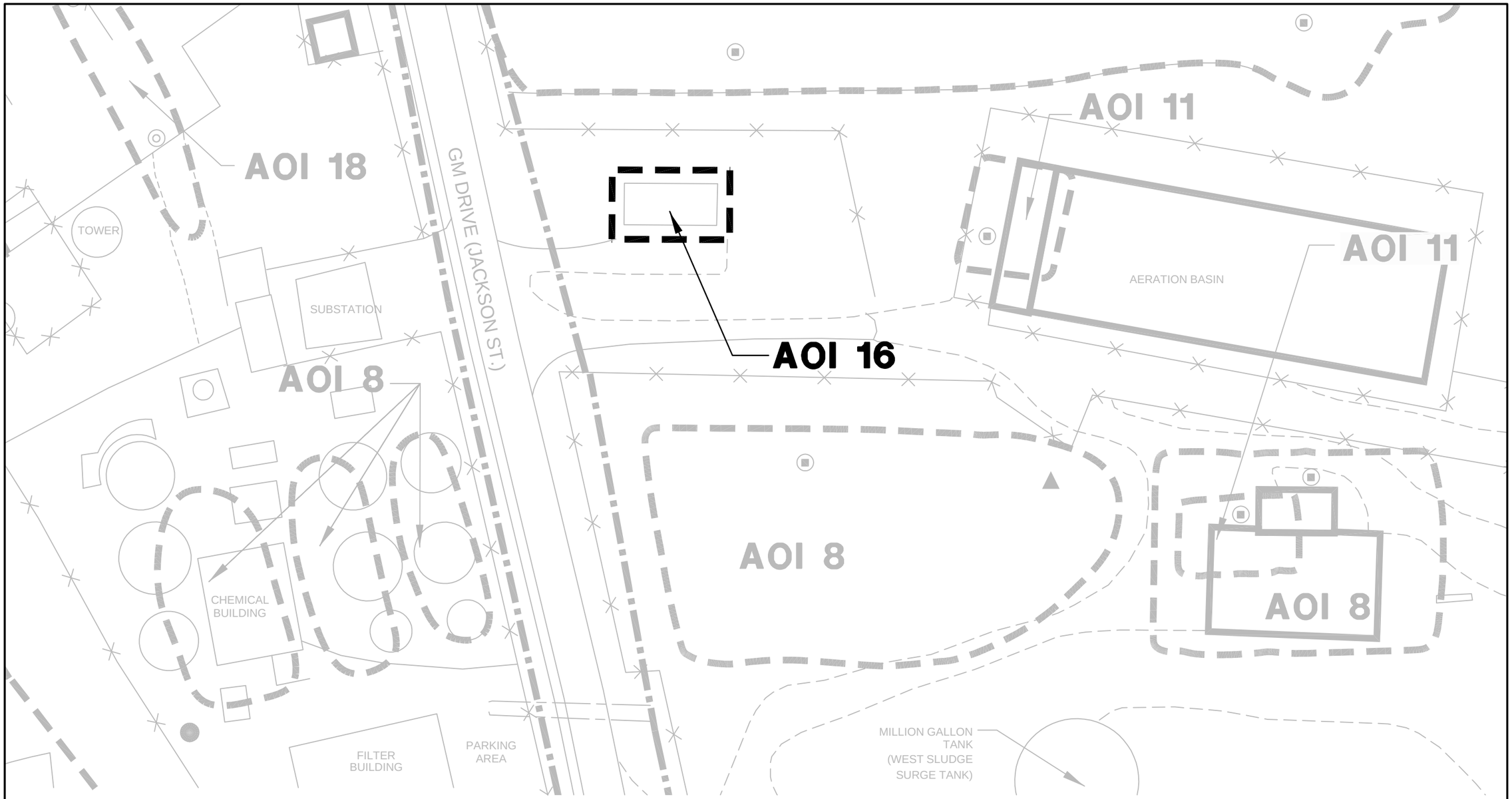


figure 5.15

AOI 15 - FORMER EQUIPMENT STORAGE AREA
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO015 OCT 25/2001



LEGEND

- APPROXIMATE FORMER AOI BOUNDARY
- PROPERTY BOUNDARY
- FENCE LINE
- DIRT ROADS
- ROADS / PAVED AREAS
- PROPOSED BORING LOCATION
- PROPOSED SURFACE WATER SAMPLE LOCATION
- PROPOSED SURFACE WATER SAMPLE LOCATION
- PROPOSED SURFACE SOIL SAMPLE LOCATION

N

0 20 60ft

figure 5.16

AOI 16 - FORMER ELECTRICAL SUBSTATION
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO016 OCT 25/2001

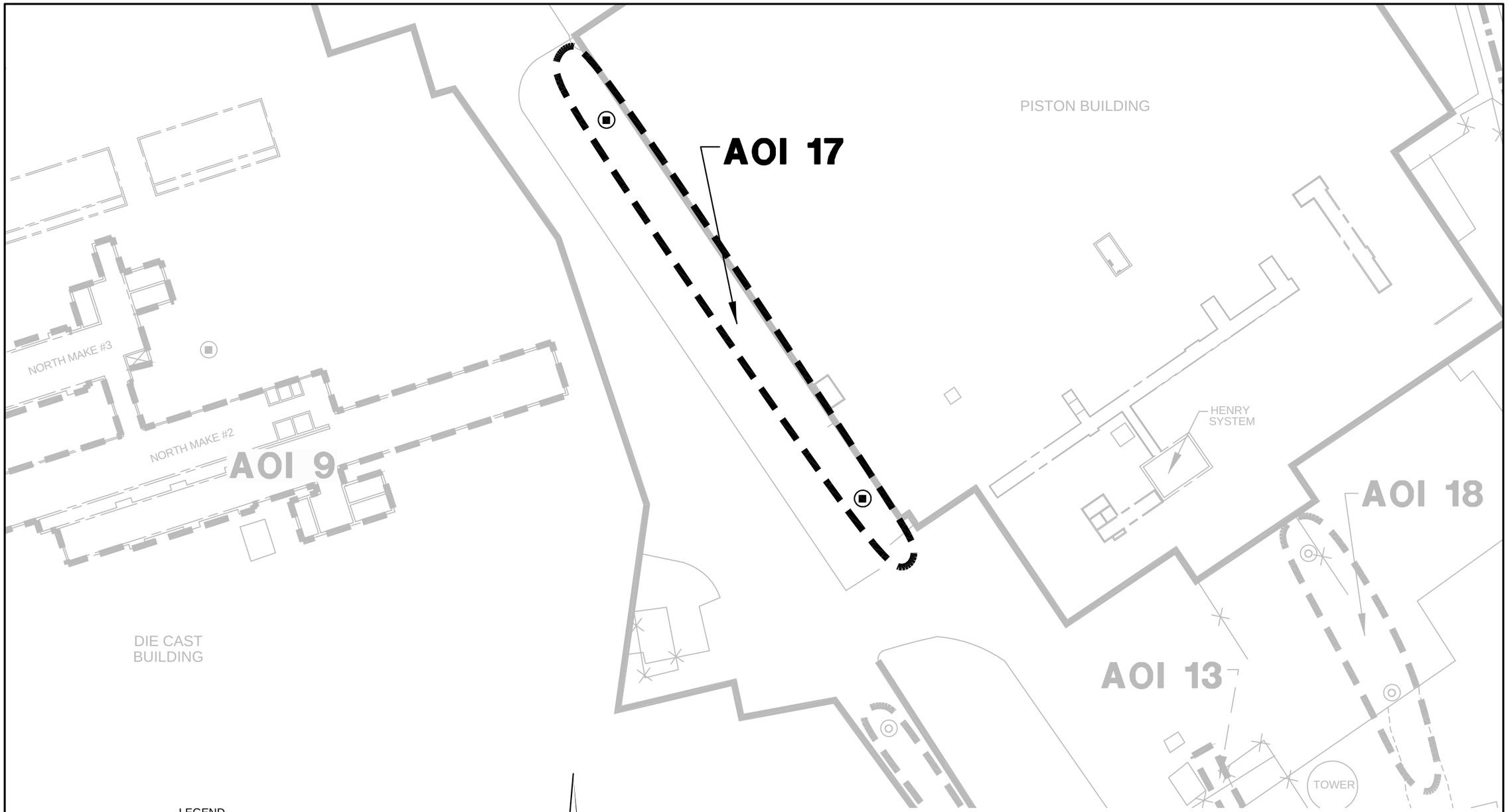


figure 5.17

AOI 17 - PISTON BUILDING OIL ACCUMULATIONS
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO017 OCT 25/2001

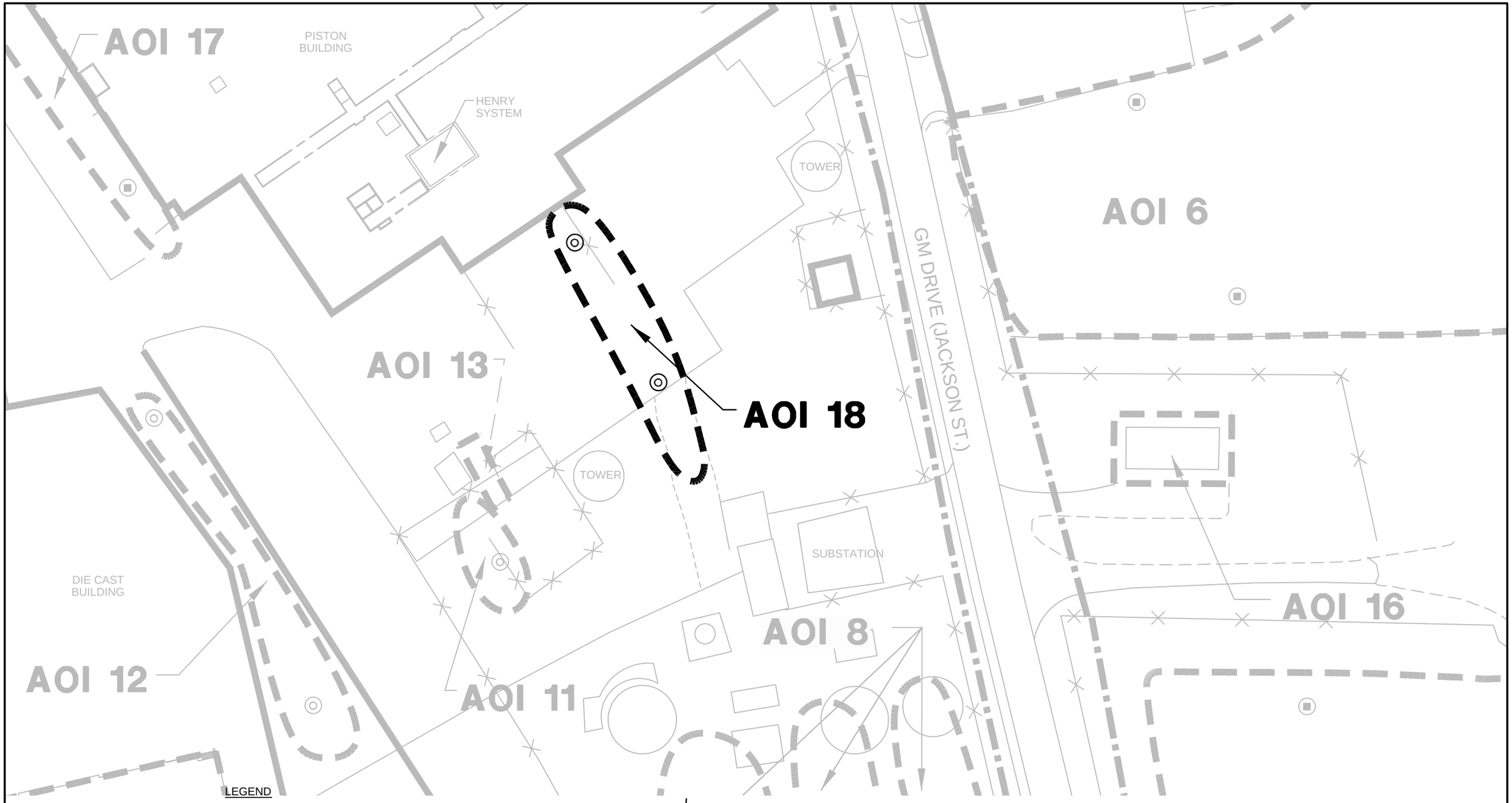


figure 5.18

AOI 18 - AREA AFFECTED BY THE HENRY SYSTEM DISCHARGE
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO018 OCT 25/2001

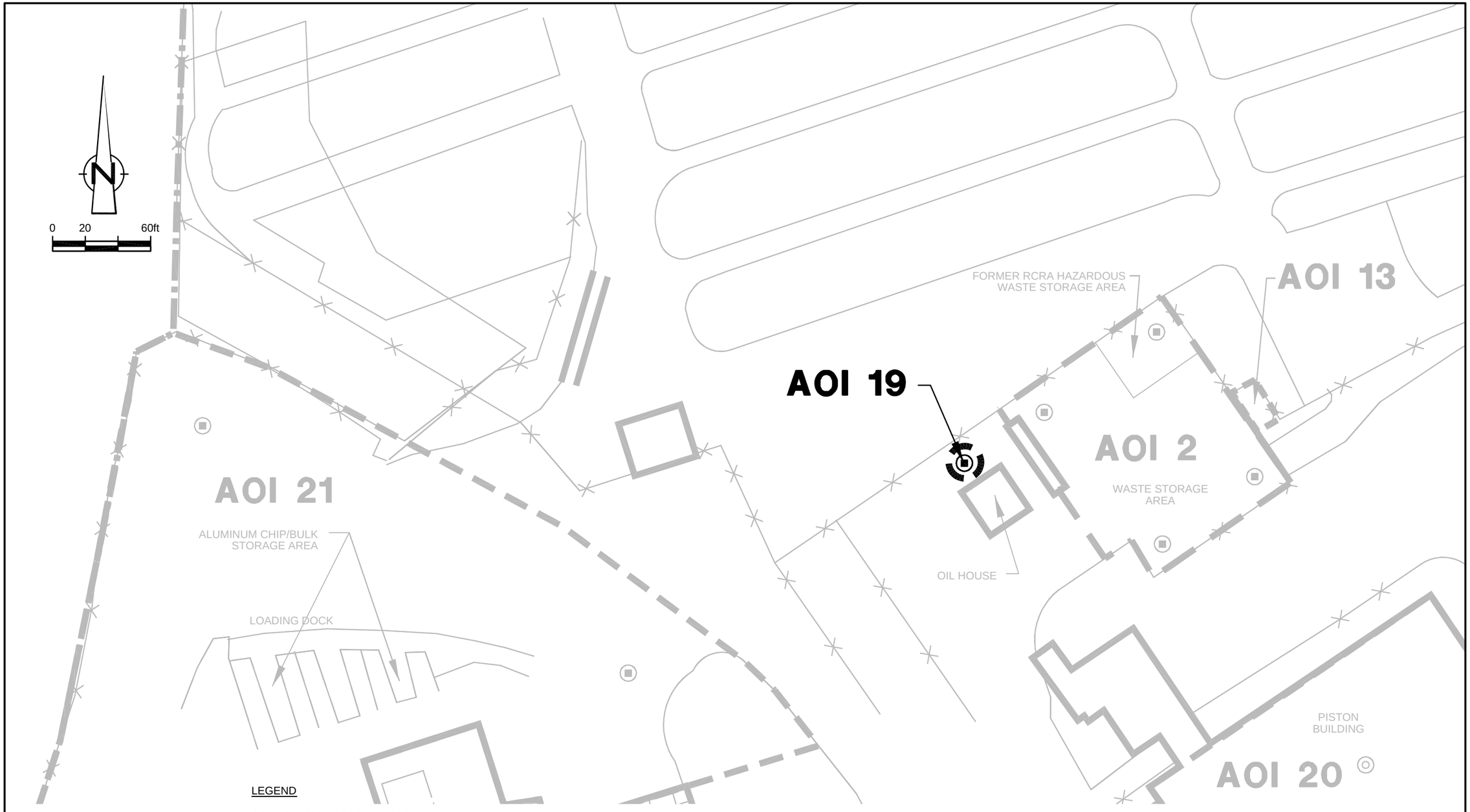


figure 5.19

AOI 19 - AREA AFFECTED BY THE PAINT AND THINNER SPILL
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO019 OCT 25/2001

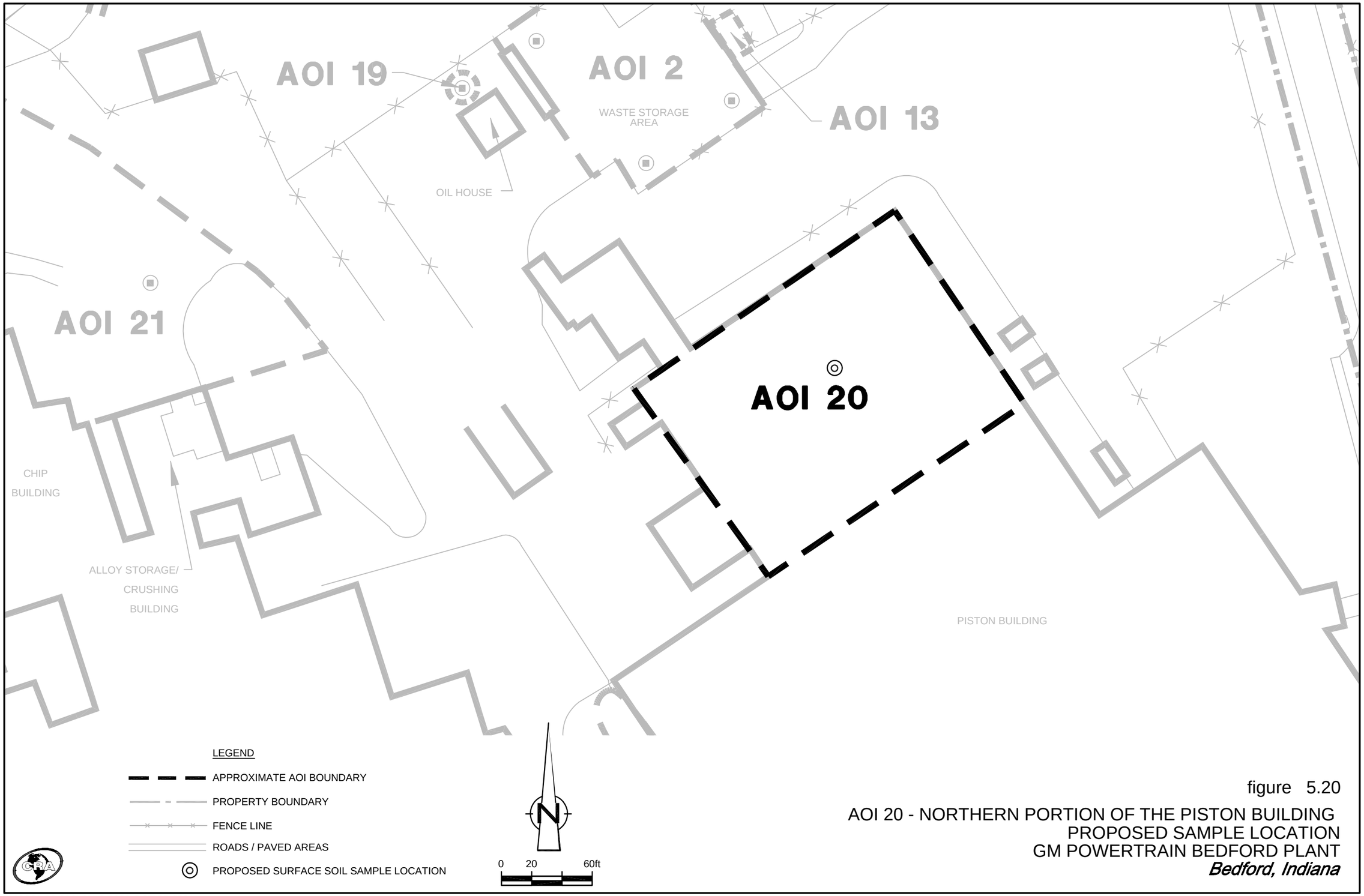


figure 5.20

AOI 20 - NORTHERN PORTION OF THE PISTON BUILDING
PROPOSED SAMPLE LOCATION
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO020 OCT 25/2001

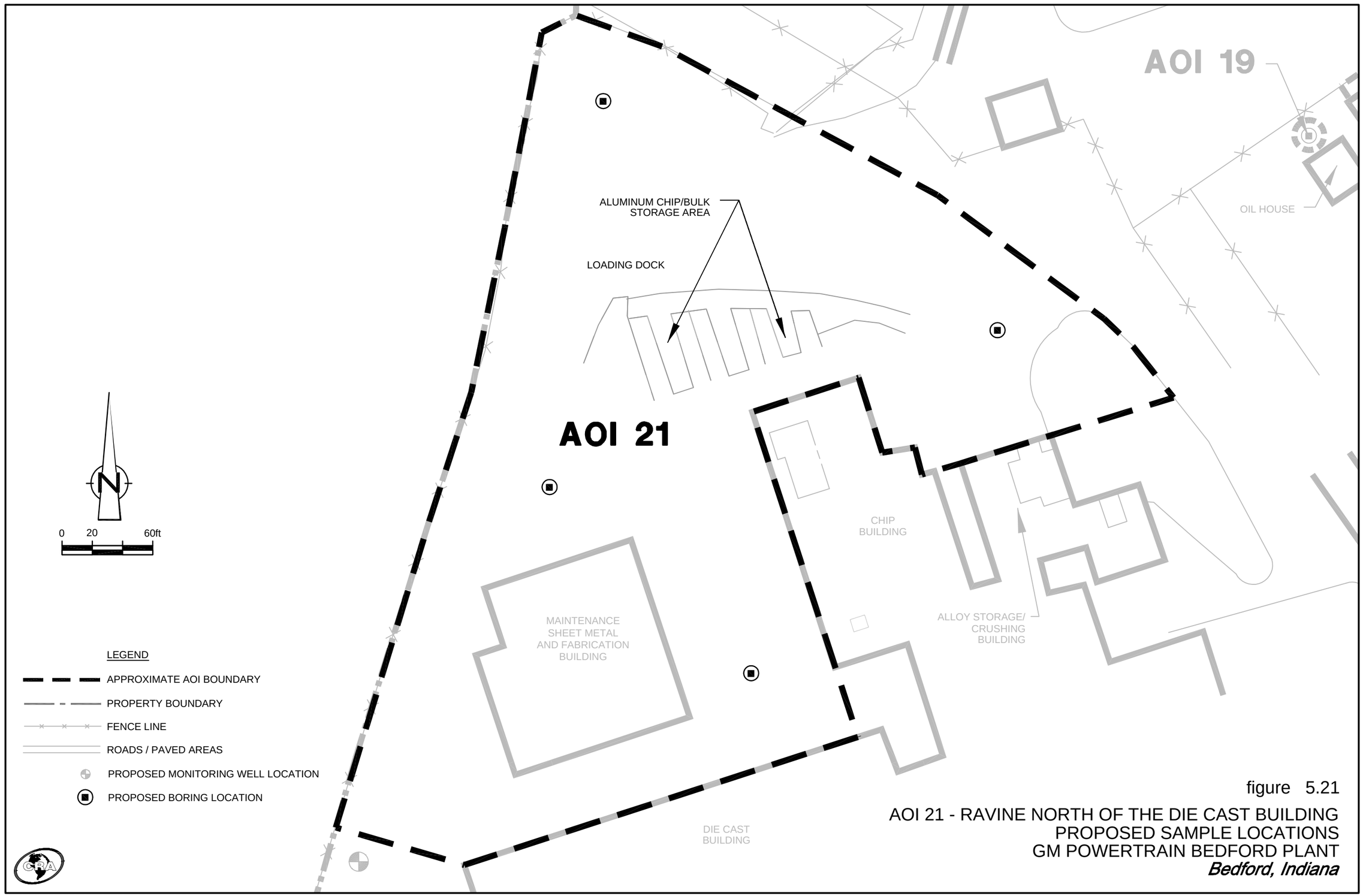


figure 5.21

AOI 21 - RAVINE NORTH OF THE DIE CAST BUILDING
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO021 OCT 15/2001

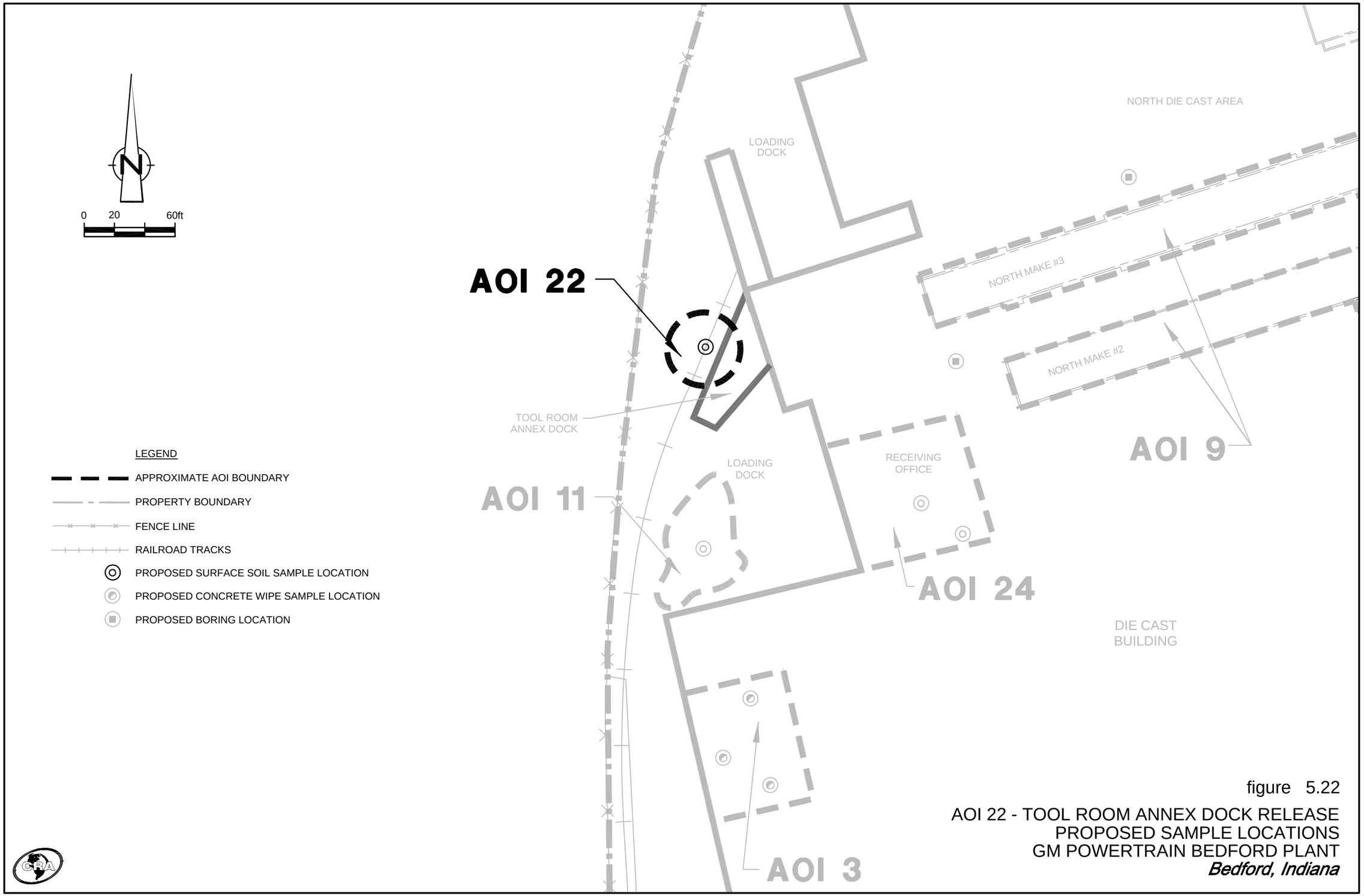


figure 5.22

AOI 22 - TOOL ROOM ANNEX DOCK RELEASE
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO022 OCT 25/2001

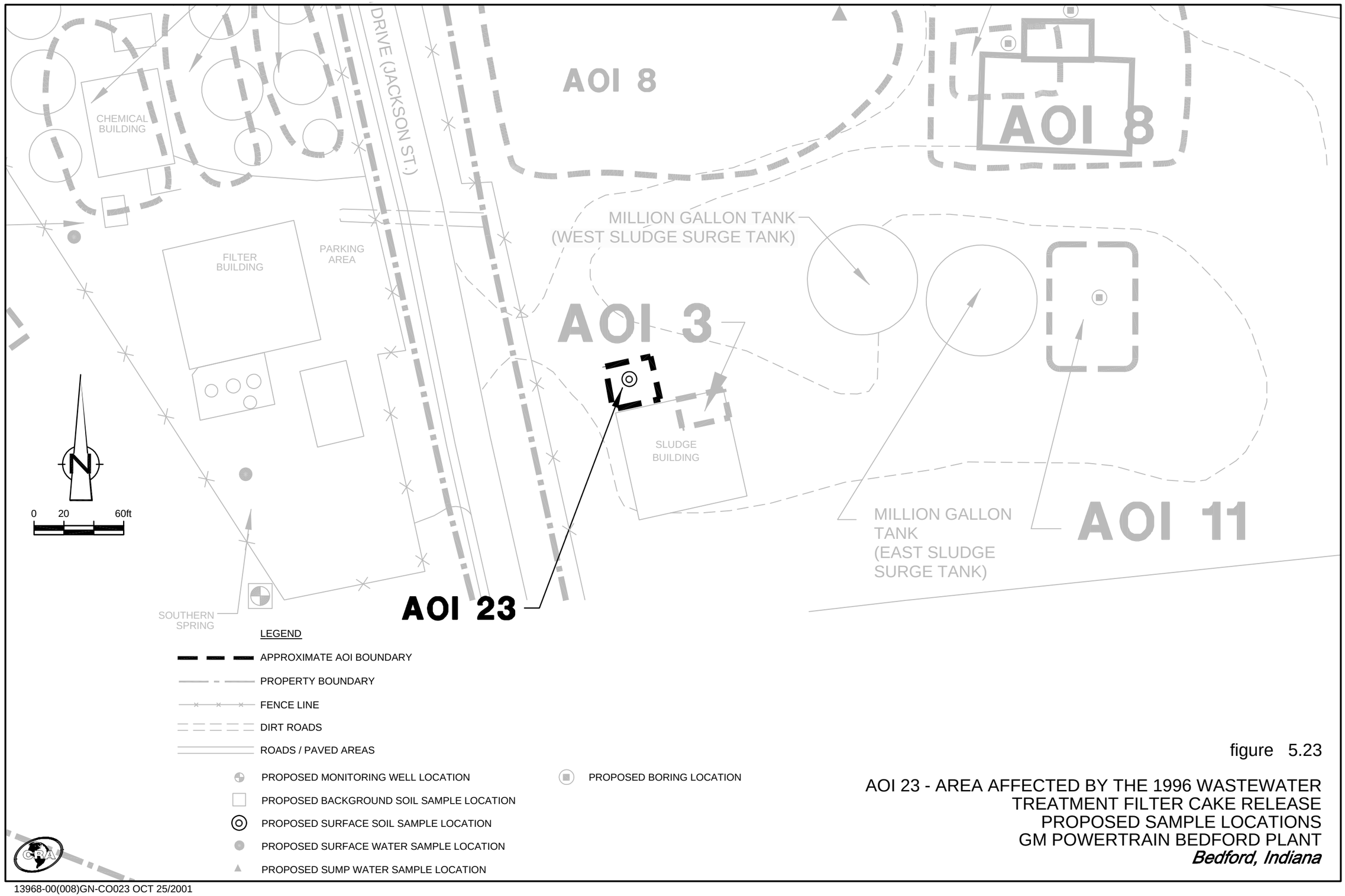


figure 5.23

AOI 23 - AREA AFFECTED BY THE 1996 WASTEWATER TREATMENT FILTER CAKE RELEASE
PROPOSED SAMPLE LOCATIONS
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO023 OCT 25/2001

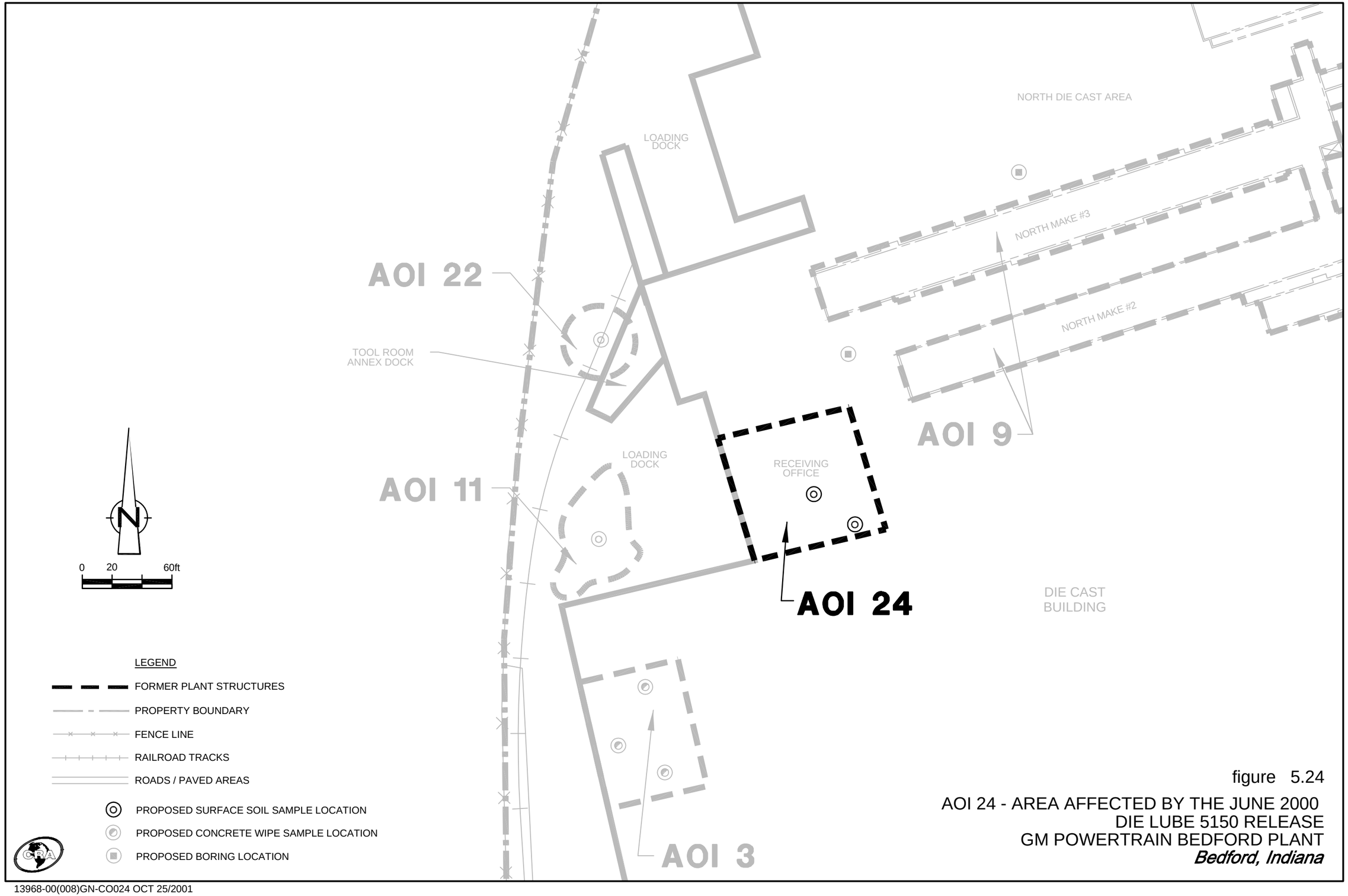


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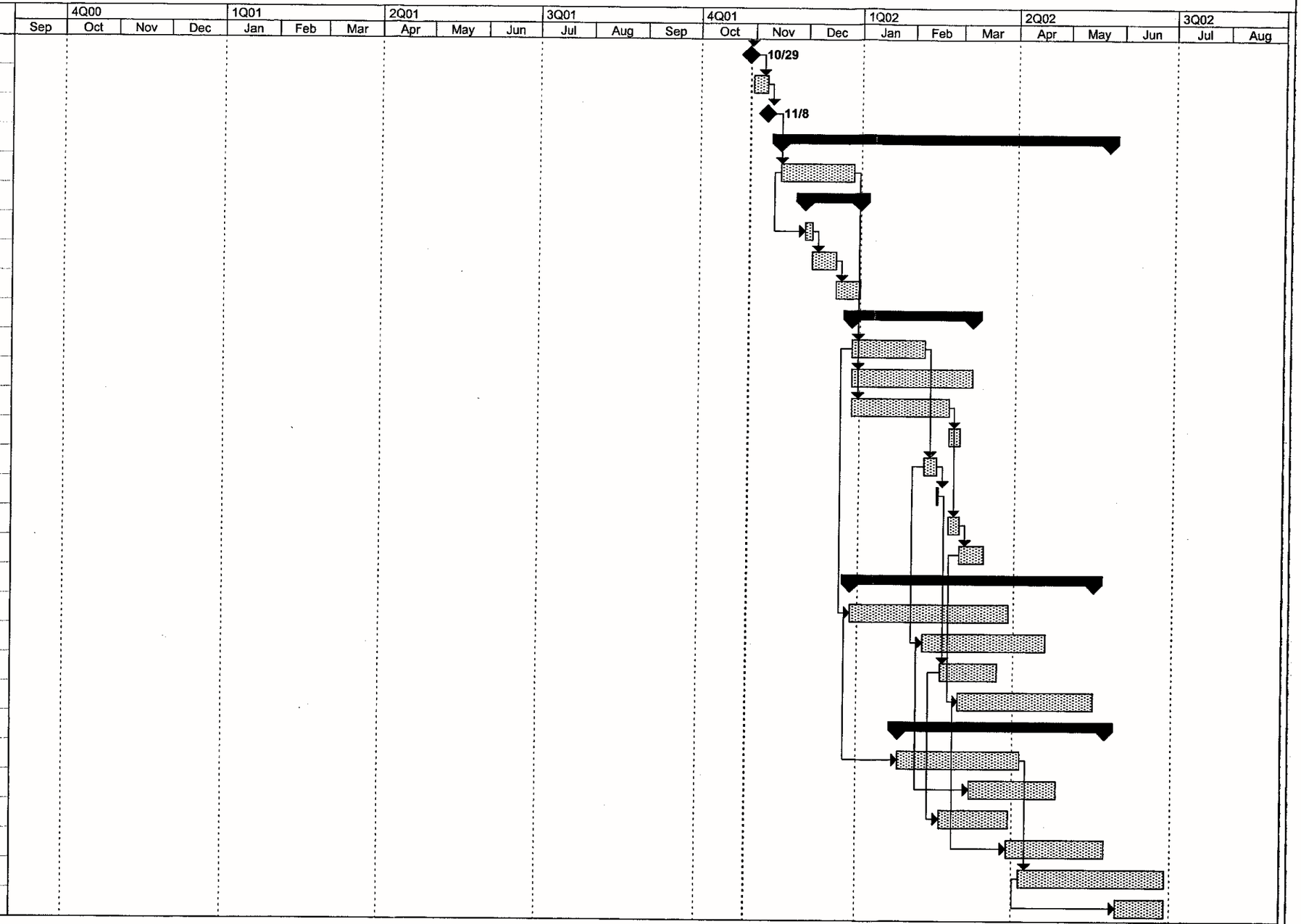
AOI 24 - AREA AFFECTED BY THE JUNE 2000
DIE LUBE 5150 RELEASE
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

13968-00(008)GN-CO024 OCT 25/2001

FIGURE 8.1

General Motors Corporation - Powertrain
Bedford Plant
RFI Work Plan Schedule

ID	Task Name
73	**Submit Final RFI Work Plan**
74	**U.S. EPA Review of RFI Work Plan**
75	**U.S. EPA Meeting/Call to Discuss RFI Work Plan**
76	**Field Investigation**
77	Contracts/Field Prep/Mobilization
78	**Geophysics Survey**
79	Field Array Survey/Setup
80	Implement Field Survey
81	Data Reduction/Boring Selection
82	**Subsurface Drilling**
83	Soil Borings/Soil Sampling
84	Bedrock Coring/Sampling/Logging
85	Monitoring Well/Packer Installation
86	Horizontal/Vertical Survey
87	**Sediment/Surface Water/Sump Sampling**
88	**Concrete Wipe Sampling**
89	**Well Development**
90	**Groundwater Sampling**
91	**Laboratory Analyses**
92	Soil Analyses
93	Sediment/Surface Water/Sump Analyses
94	Concrete Wipe Analyses
95	Groundwater Analyses
96	**Data Validation**
97	Soil Data
98	Sediment/Surface Water/Sump Water Data
99	Concrete Wipe Data
100	Groundwater Data
101	**Data Reduction and Analysis**
102	**Preparation of Data Tables/Data Screen Figures**



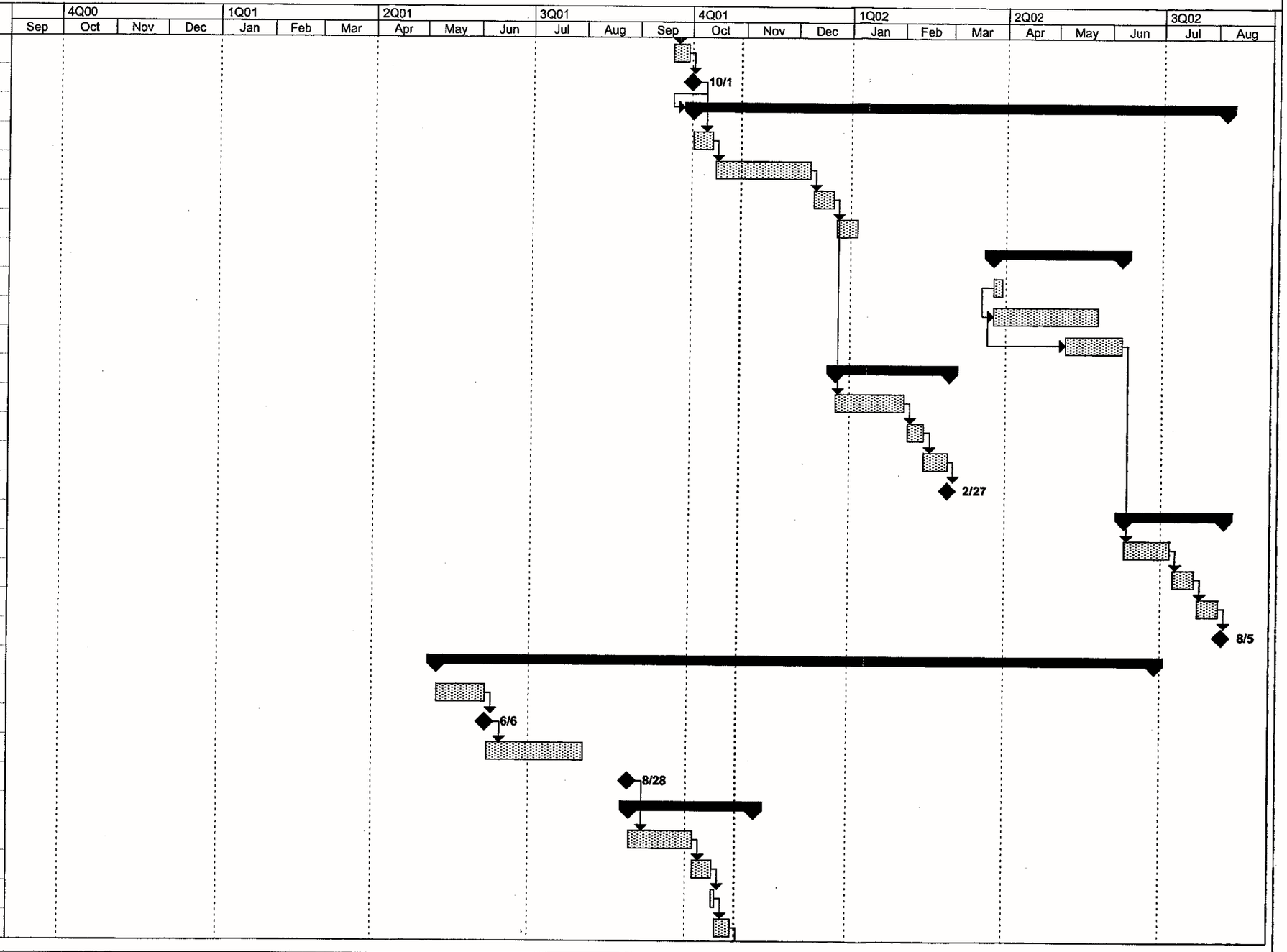
Project: 013968 Project Schedule
Date: Mon 10/29/01

Task | Split | Progress | Milestone | Summary | Rolled Up Task | Rolled Up Split | Rolled Up Milestone | Rolled Up Progress | External Tasks | Project Summary

FIGURE 8.1

General Motors Corporation - Powertrain
Bedford Plant
RFI Work Plan Schedule

ID	Task Name
42	Final FSP Preparation
43	Submit Final FSP to U.S. EPA
44	**Implement Field Sampling Plan**
45	Stream, Sediment, Soil Sampling
46	Laboratory Analyses of Collected Samples
47	Data Validation of Laboratory Analyses
48	Develop and Transmit Letters to Owners
49	**High-Flow Surface Water Sampling**
50	Field Sampling
51	Laboratory Analyses
52	Data Validation of Laboratory Analytical
53	**Draft Stream Sampling TM Preparation**
54	Draft Sampling Stream Sampling TM
55	Review of Draft TM
56	Final Draft TM Preparation
57	Submit Final TM to U.S. EPA
58	**Addendum to Stream Sampling TM (add. surface water)**
59	Preparation of Draft TM Addenda
60	Review of Draft TM Addenda
61	Final Draft TM Preparation
62	Submit Final TM to U.S. EPA
63	**RCRA Facility Investigation**
64	Development of Sampling Matrix
65	Meeting with U.S. EPA to Discuss Proposed RFI Work Plan
66	Revisions/Discussions with U.S. EPA
67	Site Meeting/Tour of AOIs
68	**RFI Work Plan**
69	**Draft RFI Work Plan Preparation**
70	**Review of Draft RFI Work Plan**
71	**Final Draft RFI Work Plan Preparation**
72	**Review Final Draft RFI Work Plan**



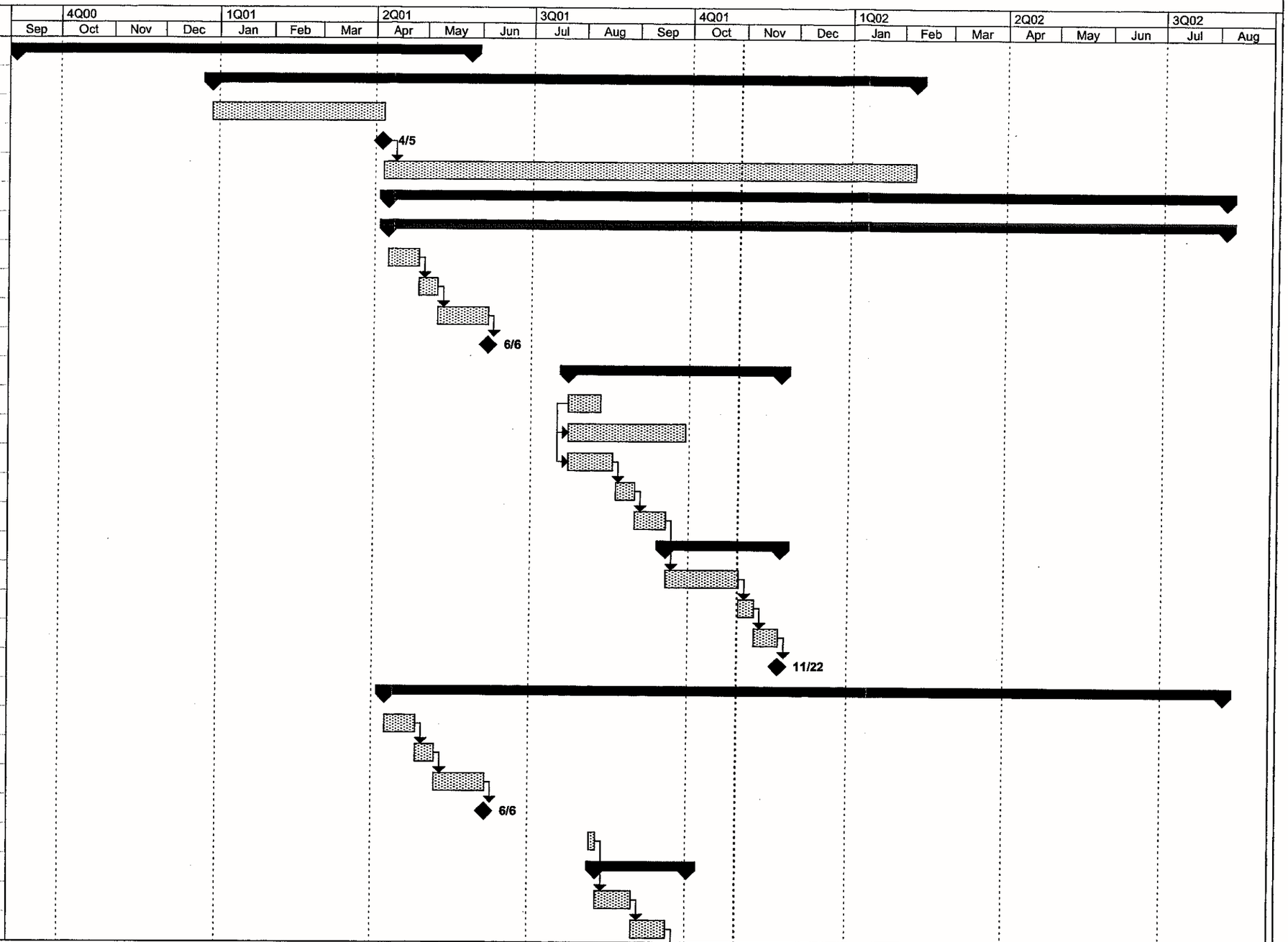
roject: 013968 Project Schedule
Date: Mon 10/29/01

Task | Progress | Summary | Rolled Up Split | Rolled Up Progress | Project Summary
Split | Milestone | Rolled Up Task | Rolled Up Milestone | External Tasks

FIGURE 8.1

General Motors Corporation - Powertrain
Bedford Plant
RFI Work Plan Schedule

ID	Task Name
1	**CCR Preparation**
12	**Develop and Implement Community Relations Plan**
13	Develop Community Relations Plan
14	Meeting to Discuss Community Relations
15	Implement Community Relations
16	**Preliminary RFI Activities Work Plan Development**
17	**Off-Site Well Survey and Sampling**
18	Develop Draft Work Plan
19	Review of Draft Work Plan
20	Preparation of Final Work Plan
21	Off-Site Well Survey and Sampling Work Plan Submittal to U.S. EPA
22	**Implement Off-Site Well Survey/Sampling**
23	Street Canvassing
24	City Records Search
25	Laboratory Analyses of Collected Samples
26	Data Validation of Laboratory Analyses
27	Develop Letters and Transmit Results to Owners
28	**Off-Site Well Survey and Sampling Technical Memorandum**
29	Draft Off-Site Well Survey and Sampling TM Preparation
30	Review of Draft TM
31	Final Draft TM Preparation
32	Submit Final TM to U.S. EPA
33	**Stream Sampling Work Plan**
34	Develop Draft Work Plan
35	Review of Draft Work Plan
36	Preparation of Final Work Plan
37	Stream Sampling Work Plan
38	Implement Field Reconaissance
39	**Develop Field Sampling Plan**
40	Preparation of Draft Field Sampling Plan
41	Review of Field Sampling Plan

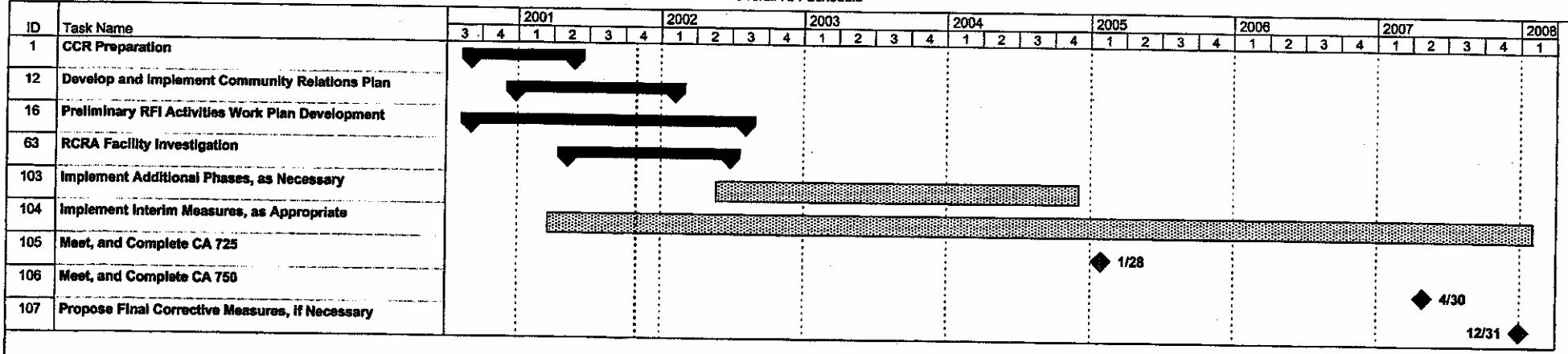


Project: 013968 Project Schedule
Date: Mon 10/29/01

Task | Progress | Summary | Rolled Up Split | Rolled Up Progress | Project Summary
Split | Milestone | Rolled Up Task | Rolled Up Milestone | External Tasks

FIGURE 8.2

General Motors Corporation - Powertrain
Bedford Plant
Overall RFI Schedule



Project: 013968 Project Schedule
Date: Mon 10/29/01

Task | Split | Progress | Milestone | Summary | Rolled Up Task | Rolled Up Split | Rolled Up Milestone | Rolled Up Progress | External Tasks | Project Summary

TABLE 1.1

SUMMARY OF PROPOSED INVESTIGATION

GENERAL MOTORS POWERTRAIN BEDFORD FACILITY

Area of Interest (AOI)		Investigation to be performed
Site-Wide Hydrogeological Investigation		• Soil sampling. • Groundwater sampling.
Background Soil Sampling		• Soil sampling.
AOI 1	Former Railroad Operations and Minerals Processing Facility	• Soil Sampling. • Surface water sampling at two springs.
AOI 2	Waste Storage Area	• Soil Sampling.
AOI 3	PCB Storage Areas	• Concrete Sampling.
AOI 4	Former North Disposal Area	• Soil Sampling. • Sediment sampling at seeps. • Surface water sampling at seeps.
AOI 5	Former East Sand Disposal Area	• Soil sampling.
AOI 6	Former Sludge Disposal and Fire Training Area	• Initial geophysical delineation. • Soil sampling.
AOI 7	Former North Lagoon	• Soil sampling. • Bedrock sampling. • Groundwater sampling from collection sump.
AOI 8	Former South Lagoons	• Soil sampling. • Bedrock Sampling. • Groundwater sampling from collection sump.
AOI 9	Service Tunnels	• Soil sampling.

TABLE 1.1 cont.

SUMMARY OF PROPOSED INVESTIGATION

GENERAL MOTORS POWERTRAIN BEDFORD FACILITY

	Area of Interest (AOI)	Investigation to be performed
AOI 10	Existing Stormwater Lagoon and Outfall 003	• Sediment sampling in Existing Stormwater Lagoon and at spring area. • Surface water sampling in Existing Stormwater Lagoon and at spring area.
AOI 11	Above Ground Storage Tanks	
	- 29 Used Oil ASTs	• Soil sampling.
	- Mineral Oil Release	• Soil sampling.
	- Diesel Oil Release	• Soil sampling.
AOI 12	Area Affected by the Reclaimed Hydraulic Fluid Release	• Soil sampling.
AOI 13	Underground Storage Tanks	
	- Oil House USTs	• Requesting Closure Verification from IDEM.
	- Piston Yard USTs	• Requesting Closure Verification from IDEM.
AOI 14	McBride Cows Disposal Area	• Initial geophysical delineation. • Soil sampling.
AOI 15	Former Equipment Storage Area	• Soil sampling.
AOI 16	Former East Electrical Substation	• No longer considered an AOI. Property owned by local electric utility.
AOI 17	Piston Building Oil Accumulations	• Soil sampling.
AOI 18	Area Affected by the Henry System Discharge	• Soil sampling.

TABLE 1.1 cont.

SUMMARY OF PROPOSED INVESTIGATION

GENERAL MOTORS POWERTRAIN BEDFORD FACILITY

	Area of Interest (AOI)	Investigation to be performed
AOI 19	Area Affected by the Paint and Thinner Spill	• Soil sampling.
AOI 20	Northern Portion of the Piston Building	• Soil sampling.
AOI 21	Filled Ravine North of the Die Cast Building	• Soil sampling.
AOI 22	Tool Room Annex Dock Release	• Soil sampling.
AOI 23	Area Affected by the 1996 Wastewater Treatment Filter Cake Release	• Soil sampling.
AOI 24	Area Affected by the 2000 Die Lube 5150 Release	• Concrete inspection. • Soil sampling.

TABLE 5.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
GENERAL MOTORS POWERTRAIN BEDFORD PLANT

	AOI	*Sample Matrix*	*Field Parameters*	*Laboratory Parameters*	*Investigative Samples* [3]	*Quality Control Samples*		
						Field Blanks [4]	*Field Duplicates* [5]	*MS/MSD* [4]
1	Former Railroad Operations and Minerals Processing Facility	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics, Chloride	21 / 7	1/20	1/10	1/20
		surface water	pH, dissolved oxygen, specific conductance, turbidity, temperature	VOCs, SVOCs, PCBs, Inorganics, Chloride	2 / 2	1/20	1/10	1/20
2	Waste Storage Area	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	12 / 4	1/20	1/10	1/20
3	PCB Storage Areas	concrete [1]	Not Applicable	PCBs	3 / 3	1/20	1/10	1/20
4	Former North Disposal Area	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	15 / 5	1/20	1/10	1/20
		sediment	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	3 / 3	1/20	1/10	1/20
		surface water	pH, dissolved oxygen, specific conductance, turbidity, temperature	VOCs, SVOCs, PCBs, Inorganics	3 / 3	1/20	1/10	1/20
5	Former East Sand Disposal Area	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	12 / 4	1/20	1/10	1/20
6	Former Sludge Disposal and Fire Training Area	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	21 / 7	1/20	1/10	1/20
7	Former North Lagoon and Outfall 001	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	8 [6] / 2	1/20	1/10	1/20
		groundwater	pH, dissolved oxygen, specific conductance, turbidity, temperature	VOCs, SVOCs, PCBs, Inorganics	1 / 1	1/20	1/10	1/20

TABLE 5.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
GENERAL MOTORS POWERTRAIN BEDFORD PLANT

	AOI	*Sample Matrix*	*Field Parameters*	*Laboratory Parameters*	*Investigative Samples* [3]	*Quality Control Samples*		
						Field Blanks [4]	*Field Duplicates* [5]	*MS/MSD* [4]
8	Former South Lagoons and Outfall 002	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	7 [7] / 2	1/20	1/10	1/20
		groundwater	pH, dissolved oxygen, specific conductance, turbidity, temperature	VOCs, SVOCs, PCBs, Inorganics	1 / 1	1/20	1/10	1/20
9	Service Tunnels *	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	18 / 9	1/20	1/10	1/20
10	Existing Stormwater Lagoon and Outfall 003	sediment	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	3 / 3	1/20	1/10	1/20
		surface water	pH, dissolved oxygen, specific conductance, turbidity, temperature	VOCs, SVOCs, PCBs, Inorganics	2 / 2	1/20	1/10	1/20
11	Above Ground Storage Tanks							
	29 Used Oil ASTs	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	9 / 3	1/20	1/10	1/20
	Mineral Oil Release	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	1 / 1	1/20	1/10	1/20
	Diesel Oil Release	soil	PID VOC screen	VOCs, SVOCs, PCBs	1 / 1	1/20	1/10	1/20
12	Area Affected by the Reclaimed Hydraulic Fluid Release	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	2 / 2	1/20	1/10	1/20
13	Underground Storage Tanks Oil House USTs Piston Yard USTs	Contact IDEM for Closure Approval						

TABLE 5.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
GENERAL MOTORS POWERTRAIN BEDFORD PLANT

	AOI	*Sample Matrix*	*Field Parameters*	*Laboratory Parameters*	*Investigative Samples* [3]	*Quality Control Samples*		
						Field Blanks [4]	*Field Duplicates* [5]	*MS/MSD* [4]
14	McBride Cows Disposal Area	soil	PID VOC screen	PCBs	2 / 2	1/20	1/10	1/20
15	Former Equipment Storage Area	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	6 / 2	1/20	1/10	1/20
16	Former East Electrical Substation	none [2]	none [2]	none [2]	0 / 0	0 / 0	0 / 0	0 / 0
17	Piston Building Oil Accumulations	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	6 / 2	1/20	1/10	1/20
18	Area Affected by the Henry System Discharge	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	2 / 2	1/20	1/10	1/20
19	Area Affected by the Paint and Thinner	soil	PID VOC screen	VOCs, PCBs	3 / 1	1/20	1/10	1/20
20	Northern Portion of the Piston Building	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	1 / 1	1/20	1/10	1/20
21	Filled Ravine North of the Die Cast Building	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	12 / 4	1/20	1/10	1/20
22	Tool Room Annex Dock Release	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	1 / 1	1/20	1/10	1/20
23	Area Affected by the 1996 Wastewater Treatment Filter Cake Release	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	1 / 1	1/20	1/10	1/20

TABLE 5.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
GENERAL MOTORS POWERTRAIN BEDFORD PLANT

	AOI	*Sample Matrix*	*Field Parameters*	*Laboratory Parameters*	*Investigative Samples* [3]	*Quality Control Samples*		
						Field Blanks [4]	*Field Duplicates* [5]	*MS/MSD* [4]
24	Area Affected by the June 2000 Die Lube 5150 Release	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	2 / 2	1/20	1/10	1/20
Site-Wide Hydrogeological Investigation		soil	PID VOC screen	Inorganics, TOC, grain size analysis, hydraulic conductivity	16 / 9	1/20	1/10	1/20
		groundwater	pH, dissolved oxygen, specific conductance, turbidity, temperature	VOCs, SVOCs, PCBs, Inorganics	18 / 9	1/20	1/10	1/20
Background Soil Sampling		soil	PID VOC screen	Inorganics, TOC, grain size	5 / 5	1/20	1/10	1/20

Notes:

General: Specific analytes are listed in the Quality Assurance Project Plan (QAPP) (Appendix C).
Based on the results of the soil sampling and the site-wide hydrogeological investigation, AOI-specific monitoring wells may be installed.

* = Access to the tunnel areas within the plant is severely limited due to operating machinery and molten metal transfer activities. Completion of this sampling will be coordinated with plant production schedules.

(1) Concrete wipe samples at the Die Cast Building Former PCB Storage Area consistent with 40 CFR 761.123.

(2) It has been determined that this property is not owned by GM. Cinergy Corporation (the local electric utility) owns and operated the property where the former electrical substation was located, and therefore this area is no longer considered an AOI.

(3) Number of investigative samples / number of sample locations. Additional samples may be collected if evidence of contamination is observed.

(4) Based on 1 / 20 number of investigative samples. Field equipment rinseate blanks will be collected at a minimum frequency of one per day of sampling.

(5) Based on 1 / 10 number of investigative samples per matrix, or at a minimum of one per event per matrix.

(6) Number of soil samples include two samples from the bedrock surface.

(7) Number of soil samples include one sample from the bedrock surface.

APPENDIX A
PROJECT MANAGEMENT PLAN

GM POWERTRAIN BEDFORD PLANT
105 GM DRIVE
BEDFORD, INDIANA

EPA ID# IND006036099

Prepared For:
GENERAL MOTORS CORPORATION

Conestoga-Rovers & Associates

8615 West Bryn Mawr
Chicago, Illinois 60631

Office: (773) 380-9933
Fax: (773) 380-6421

OCTOBER 26, 2001
REF. NO. 013968 (8)

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LIST OF FIGURES
(Following Text)

FIGURE A.4.1 PROJECT MANAGEMENT ORGANIZATION

1.0 INTRODUCTION

This Project Management Plan (PMP) is submitted as an appendix to and forms part of the Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) Work Plan prepared by General Motors Corporation (GM) for the GM Powertrain – Bedford Plant (Bedford Facility).

This part of the RFI Work Plan presents the project management approach for the RFI to be undertaken at the Bedford Facility. Project management of the RFI includes:

- Selecting, coordinating and scheduling staff, contractors, subcontractors and laboratories;
- Overseeing project implementation to ensure the technical requirements of the RFI Work Plan are met; and
- Ensuring that work proceeds in accordance with the schedules, practices and standards set forth in each of the parts within, and any subsequent parts to, this RFI Work Plan.

The RFI will be conducted under the management of the General Motors Corporation Worldwide Facilities Group - Remediation Team. Conestoga-Rovers & Associates (CRA), 8615 West Bryn Mawr Avenue, Chicago, Illinois, 60631, will be GM's environmental engineer for the RFI. CRA will be responsible for overseeing day-to-day activities, including:

- Management of subcontractors (drilling firms, analytical laboratories, etc.) and field activities, as detailed in the RFI Work Plan;
- Consideration of health and safety;
- Data validation and data management;
- Quality Assurance/Quality Control (QA/QC) corrective action as provided for in the Quality Assurance Project Plan (QAPP), and
- Data evaluation and interpretation.

The ecological risk assessment will be completed by Exponent, located at 15375 SE 30th Place, Suite 250, Bellevue, Washington, 98007. The human health risk assessment will be completed by ENVIRON International (ENVIRON), located at 214 Carnegie Center,

Princeton, New Jersey, 08540. DecisionQuest, 1101 14th Street NW, Suite 200, Washington, DC, 20005, will be GM's community relations consultant for the RFI.

The remainder of this Plan describes the project organization, responsibilities, and the approach to the investigation analysis/report preparation.

2.0 TECHNICAL APPROACH

The overall approach that will be used for the RFI will be to collect the data that are necessary to complete the RFI Report and Environmental Indicators and to allow for the evaluation of corrective measures (if required). The goal of completing the RFI Report will be achieved by implementing the approved RFI Work Plan, and any Addenda, thereto.

3.0 SCHEDULE

The schedule for implementation of the RFI is presented in Section 8.0 of the RFI Work Plan. It will be the role of the Project Management Team identified in Section 4.0 to ensure that the schedules are met, or to identify the reasons why scheduled activities were modified, if necessary.

4.0 PROJECT MANAGEMENT ORGANIZATION

Figure A.4.1 presents GM's project management organization for the RFI.

The Project Manager for the U.S. Environmental Protection Agency (U.S. EPA) is:

Mr. Peter Ramanauskas
U.S. EPA, Region 5
77 West Jackson Blvd (DW-8J)
Chicago, Illinois 60604-3590
Phone: (312) 886-7890
Fax: (312) 353-4788

The Project Manager for GM is:

Ms. Cheryl Hiatt
General Motors Corporation
Worldwide Facilities Group
Remediation Team
Troy Tech Park South - Building A
1996 Technology Drive
Mail Code: 483-619-356
Troy, Michigan 48083
Phone: (248) 680-5219
Fax: (248) 680-5129

All communications between U.S. EPA (and other regulatory agencies),and GM, and all documents, plans, reports, approvals and other correspondence concerning activities performed during the RFI, will be directed through Ms. Cheryl Hiatt and Mr. Peter Ramanauskas.

CRA's Project Director is Mr. Glenn Turchan; CRA's Senior Project Engineer/Advisor is Mr. James McGuigan; and CRA's Project Manager is Mr. William K. Steinmann. CRA's Quality Assurance (QA) Officer is Mr. Paul Wiseman. CRA's Health and Safety Officer is Mr. Matt Lazaric. CRA will perform and supervise all field activities of the RFI in accordance with the RFI Work Plan.

ENVIRON will be GM's human health risk assessment consultant for the RFI. Mr. Steve Song is ENVIRON's Human Health Risk Assessment Project Director. Mr. CY Jeng is the Project Manager for the human health risk evaluation. ENVIRON will perform the human health risk evaluation and prepare the Environmental Indicators Report.

Exponent will be GM's ecological risk assessment consultant for the RFI. Mr. Pieter Booth is Exponent's Ecological Risk Assessment Project Director. Mr. Rick Bodishbaugh is the Project Manager for the ecological risk evaluation and will perform the ecological risk evaluation.

DecisionQuest will be GM's community relations consultant for the RFI. Mr. Richard Goss will be the DecisionQuest Project Manager for community relations issues.

Due to the karstic geologic setting, GM may retain additional outside, third party experts as needed. GM may utilize these consultants at key milestones throughout the RFI as a technical advisor. Dr. Bernhard Kueper, DNAPL contamination/remediation expert and professor, Queen's University, Kingston, Ontario has been retained as a Technical Advisor for this project.

The following laboratories have been identified as the analytical subcontractors, depending upon their specialty.

Media	*Laboratory*
Soil, Groundwater, Surface Water, Sediment, Biota, NAPL	Severn Trent Laboratory, North Canton, OH
Off-Site Drinking Well Water	Aqua Tech Environmental Laboratories, Melmore, OH
DNAPL Fluid Properties	Queen's University (DNAPL's Groundwater Research Group) Kingston, Ontario, Canada
Geotechnical	Schleede-Hampton Associates, Inc. Birmingham, Michigan Core Laboratories Houston, Texas

The drilling subcontractors will be determined following competitive bidding protocols. The firms selected by GM for the drilling activities will be identified to U.S.EPA prior to commencement of field activities.

All firms will provide project management as appropriate to their responsibilities and will administer oversight where subcontractors are utilized under their contracts.

5.0 PROJECT MANAGEMENT QUALIFICATIONS

As identified in Section 4.0, GM has assembled a project management team and technical resource personnel with the necessary experience and capabilities required for this project. Brief descriptions of all personnel roles and qualifications are listed below.

5.1 GM PROJECT MANAGER

Ms. Cheryl Hiatt: Ms. Hiatt is the Project Manager for GM and will be responsible for implementing the project. Ms. Hiatt has the authority to commit the resources necessary to meet the project objectives and requirements. Ms. Hiatt will ensure that technical, financial, and scheduling objectives are successfully achieved. Ms. Hiatt will be the primary contact for matters concerning the project.

5.2 CRA QUALIFICATIONS

CRA is fully qualified to conduct all of the required professional services associated with the RFI at the Facility. CRA has extensive experience in conducting hydrogeological investigations at RCRA and Superfund sites in the United States. In addition, CRA has prepared corrective action plans and implemented various phases of corrective actions at a variety of sites throughout the United States.

Mr. Glenn Turchan: Mr. Turchan will act as CRA's Project Director. Mr. Turchan is Executive Vice President of CRA and will ensure that the necessary CRA resources are available for the project. Mr. Turchan will also serve as a technical advisor and peer reviewer for completing the necessary scope of work and for all documents prepared for this RFI.

Mr. James McGuigan: Mr. McGuigan will act as CRA's Senior Project Engineer/Advisor. Mr. McGuigan has extensive experience in projects of this nature. He will ensure that technical quality and scheduling are maintained throughout all activities. The CRA Project Manager will brief him regularly regarding all activities.

Mr. William Steinmann: Mr. Steinmann will act as CRA's Project Manager and site hydrogeologist. He will ensure that all day-to-day tasks are conducted in a professional and timely manner. Mr. Steinmann will keep the CRA Senior Project Engineer/Advisor

and the GM Project Manager fully informed on the status of ongoing tasks. He will also attend technical meetings, as required, and will prepare and coordinate the issuance of the RCRA Facility Investigation Report.

5.3 ADDITIONAL CRA TECHNICAL RESOURCE PERSONNEL

The following is a list of technical resource personnel who will assist with specialized aspects of the program.

Quality Assurance (QA) Officer: Mr. Paul Wiseman, a chemist with CRA, will act as CRA's QA officer for the project and will be responsible for overviewing laboratory activities, analytical data assessment and validation, and deciding laboratory data corrective actions, if required.

Health and Safety Officer: Mr. Matt Lazaric will be CRA's Site Health and Safety Officer. Mr. Lazaric will supervise the CRA Site Representative, will provide managerial guidance with respect to CRA Health and Safety, and will participate in and/or review field decisions regarding CRA Health and Safety.

Field QA Officer: Mr. Jeff Nichols will be CRA's Field QA Officer. Mr. Nichols will be responsible for the field staff and reports directly to the CRA Senior Project Engineer/Advisor and CRA Project Manager.

5.4 ENVIRON QUALIFICATIONS

ENVIRON is fully qualified to perform human health risk assessments in accordance with U.S.EPA's Risk Assessment Guidance for Superfund (RAGS). ENVIRONs Life Sciences group specializes in a range of areas including toxicology, epidemiology, biostatistics, and public health and risk assessment. The Life Sciences group, working closely with the experts in ENVIRON's Environmental Sciences and Engineering group, has addressed hundreds of environmental problems, including RCRA corrective action and identification of risk-based remediation goals. ENVIRON will prepare the Environmental Indicator Reports. ENVIRON will provide a qualifications package, if requested.

5.5 EXPONENT QUALIFICATIONS

Exponent is fully qualified to perform ecological risk assessments in accordance with U.S.EPA guidance. Exponent has a full-time staff of 675 located in 17 offices throughout the nation. Exponent scientists provide research and analysis services in a wide-range of technical disciplines, including ecological risk assessment. Exponent has conducted ecological risk assessments in aquatic and terrestrial environments from subtropical to artic latitudes. Exponent's ecology staff have expertise in a variety of disciplines, including biology, chemistry, ecology, toxicology, fisheries, oceanography, limnology, and bio-statistics. In addition, Exponent's ecology staff can draw on the knowledge and expertise of scientists, engineers, physicians, and business consultants specializing in more than seventy technical disciplines. Exponent will provide a qualifications package, if requested.

5.6 DECISIONQUEST QUALIFICATIONS

DecisionQuest is fully qualified to perform community relations during the RFI. For more than ten years, the Washington, D.C. office of DecisionQuest (formerly Environmental Issues Management, Inc.) has provided communication services covering a wide array of environmental issues and programs, including voluntary cleanup programs and RCRA Corrective Actions. DecisionQuest-Washington, D.C. (DQ-DC) has provided communications counsel on more that 100 environmental and risk-based projects in 32 states across the country. DQ-DC's involvement in these project has provided substantive experience in risk communication and the scientific issues associated with each particular situation. DQ-DC staff also possess extensive experience in hazardous waste and health issues, as well as in community, government and media relations.

6.0 LABORATORY QUALIFICATIONS

Severn Trent Laboratories (STL) is fully qualified to conduct analyses in accordance with U.S.EPA analytical methods specified in the Quality Assurance Project Plan (QAPP) for environmental samples (the QAPP is presented as Appendix C of the RFI Work Plan).

Aqua Tech Environmental Laboratories (ATEL) is fully qualified to conduct analyses in accordance with U.S.EPA analytical methods specified in the QAPP for residential well water. ATEL is a certified public drinking water laboratory for the State of Indiana.

The DNAPL's in Groundwater Research Group (Groundwater Group) within the Department of Civil Engineering at Queen's University is a leading research institution in the field of contaminant migration and remediation. The Groundwater Group at Queen's University is fully qualified to perform physical analysis of the DNAPL, as specified in the RFI Work Plan, QAPP, and addenda thereto.

Schleede-Hampton Associates, Inc. (SHA) is fully qualified to conduct analyses in accordance with U.S.EPA analytical methods specified in the QAPP for geotechnical samples.

Core Laboratories (Core Labs) is fully qualified to conduct geotechnical analysis of rock cores, in accordance ASTM and U.S.EPA methods specified in the RFI Work Plan, QAPP, and addenda thereto. Core Labs is an international company specializing in petroleum reservoir analyses and has extensive experience with rock core analyses.

All laboratories will provide a qualifications package, if requested.

7.0 DRILLING SUBCONTRACTOR QUALIFICATIONS

The drilling subcontractor will be required to meet the following qualifications:

1. Satisfy applicable Occupational Safety and Health Administration (OSHA) health and safety training requirements;
2. Provide suitable equipment and manpower to perform drilling activities within specified timeframes; and
3. Previous experience relating to similar soil boring installation programs.

The drilling subcontractor will provide a qualifications package, if requested.

8.0 OTHER CONTRACTORS/SUBCONTRACTORS

The qualifications of all contractors, subcontractors, and their personnel used in implementation of the RFI Work Plan will be documented and provided to U.S. EPA, if requested.

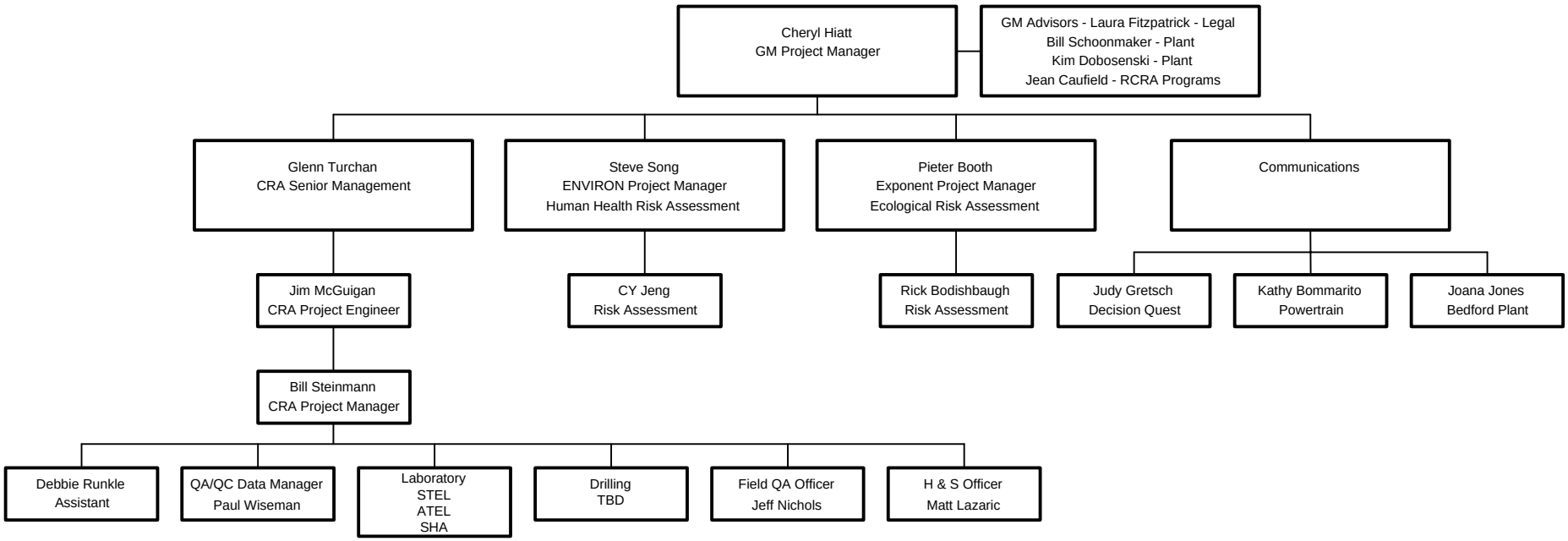


figure A.4.1

PROJECT MANAGEMENT ORGANIZATION
GM POWERTRAIN BEDFORD PLANT
Bedford, Indiana

APPENDIX B
FIELD SAMPLING PLAN

GM POWERTRAIN BEDFORD PLANT
105 GM DRIVE
BEDFORD, INDIANA

EPA ID# IND006036099

Prepared For:
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ATTACHMENT 2: FIELD FORMS

1.0 INTRODUCTION

This Field Sampling Plan (FSP) is submitted as an appendix to and forms part of the Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) Work Plan prepared by General Motors Corporation (GM) for the GM Powertrain Bedford Plant (Bedford Facility) in Bedford, Indiana (EPA ID# IND006036099).

The FSP describes procedures for the collection of surface and subsurface soil samples, groundwater and surface water samples, sediment samples, and concrete samples. Standard Operating Procedures (SOPs) are included in Attachment 1. The FSP provides additional detail on procedures where an SOP is unavailable. If no SOP is available for the particular field method, a complete description of the method will be provided in this text.

2.0 GENERAL SAMPLING PROTOCOLS

2.1 SAMPLING

Samples will be collected at the locations and frequencies specified in the RFI Work Plan. As discussed in Section 4.3 of the Quality Assurance Project Plan (QAPP), samples will be collected in order of decreasing analyte volatility (i.e., volatile organic compounds (VOCs) first, semi-volatile organic compounds (SVOCs) second, which are followed by containers for the remaining analysis). Table B.1 summarizes sample collection and analysis including quality assurance/quality control (QA/QC) samples.

The following protocols will be employed during all sampling conducted during the RFI:

1. All sampling instruments and equipment will be cleaned in accordance with the protocol presented herein prior to collecting samples for chemical analyses at each location (refer to SOP 9.0).
2. A double pair of disposable nitrile gloves will be used at each sample location for chemical analyses. A new overpair will be used for each sample. Additional glove changes at each sample location will be made for conditions such as: if the gloves are observed to be torn, or the gloves are suspected of being soiled from a source other than the sample media itself.

3. Quality assurance samples will be collected as outlined in the QAPP, which is presented in Appendix C of the RFI Work Plan.

4. All sampling generated wastes such as gloves, tyvek, etc. will be collected and containerized for proper disposal (refer to SOP 12.0).

5. Samples will be identified using labels and a tag affixed to the neck with a wire. Samples will be labeled and tagged noting the project name, Facility, unique sample number, sample location, sample interval (if appropriate), analysis required, preservative added, date, time and sampler's initials, in accordance with the QAPP, Appendix C of the RFI Work Plan. All sample preservation protocols will be followed in adherence with the QAPP. A hard cover bound field book and/or field forms will be maintained to record all samples and sampling events (refer to SOP 1.5).

6. Containers for sample collection and preservation requirements will be determined as required by the analytical parameters (refer to the QAPP, Appendix C of the RFI Work Plan). All sample bottles will be provided by the laboratory and will be prepared using a standard laboratory validated washing procedure. The sample bottles will be delivered to the Site in sealed containers with trip blanks for VOCs.

7. All collected sample shipments for chemical analysis will be iced in supplied coolers after collection and labeling. Any remaining space will be filled with packing to cushion the containers within the shipment coolers. Each cooler will be sealed with two seals comprised of Chain-of-Custody tape (or custody seal) and the sampler's name. The cooler will then be sealed with packing tape (refer to SOP 6.6).

   All samples will be delivered to the laboratory by a commercial courier. Currently, the courier will be Delivery Inc. (**1-800-782-7079**). Unless special arrangements are made, the courier will be at the field trailer every night at 7:00 p.m.

8. Samples will be shipped under chain-of-custody procedures as outlined in the QAPP and SOP 6.6.

2.2 EQUIPMENT CLEANING

Upon mobilization of the drill rig to the Facility, and prior to commencing work, the drill rig and all associated equipment will be thoroughly cleaned in accordance with

SOP 9.0. Cleaning of non-dedicated downhole equipment will be completed prior to use and between each borehole.

2.3 WASTE HANDLING

All soil/rock cuttings, development/purge water, excess sampling water, and decontamination water/fluids will be placed in containers and stored in GM's disposal area (as required) until they can be properly characterized, in accordance with SOP 12.0. All soil cuttings (unless the hole is less than two feet deep) and/or water collected during well purging and development will be tested using the Toxicity Characteristics Leaching Procedure (TCLP) to determine appropriate disposal options. If a soil boring/sample is located less than two feet deep, then the soil will be placed back into the borehole. If test pits are conducted, all contents will be replaced in the hole in the order it came out, unless sludges or containerized waste is removed. This waste will be properly stored, characterized, and disposed according to appropriate regulations.

2.4 UTILITY CLEARANCE

Prior to commencing work at the Facility, all utilities will be cleared in accordance with SOP 1.4.

For this project, the following municipal utility clearance service is:

- **Indiana's ONE-CALL system: 1-800-382-5544**

For subsurface work being conducted outside the City of Bedford and within Lawrence County, the following utility service does not subscribe to the ONE-CALL system:

- **North Lawrence Water Corporation 1-812-279-2774**

All locations on-Site must be cleared through the Plant Engineering Department:

- **Kim Dobosenski 1-812-279-7404**

2.5 FIELD INSTRUMENT USE AND CALIBRATION

All field instruments utilized during the field implementation will be used and calibrated in accordance with SOP 8.0.

3.0 SOIL SAMPLING PROTOCOLS

3.1 SUBSURFACE SOIL SAMPLING

Soil samples will be collected continuously during drilling to identify and classify soil materials (refer to SOPs 2.3, 2.6, and 6.1). Drilling techniques for subsurface soil sampling will include hollow-stem auger and direct-push methods. Soil samples will be collected using the standard penetration test method and/or Shelby tubes, or alternately, continuous sampling techniques such as direct push methodologies (e.g., Geoprobe). All collected soil samples will be described and classified according to the Unified Soil Classification System (USCS), as described in SOP 2.6).

The soil samples selected for chemical analyses will be collected in accordance with SOPs 6.1 and 6.6, and Section 4.0 of the QAPP. Specific protocols (not included in the SOPs) for collecting VOC samples are provided below:

- VOC analysis will utilize the EnCore Sampler™ as follows:
  - Depress the locking lever on the EnCore T-handle. Place the coring body, rod end first into the open end of the T-handle, aligning the two slots on the coring body with the two locking pins in the T-handle. Twist coring body clockwise to lock pins in slots. Check to ensure sampler is locked in place. The sampler is now ready for use.
  - Turn the T-handle with the T in the upward position and the coring body in the downward position. Using T-handle, push sampler into soil until coring body is completely full. When full, the small o-ring will be centered in T-handle viewing hole. Remove sampler from soil and wipe any excess soil from coring body exterior.
  - Cap coring body while it is still on the T-handle. Push and twist cap over bottom until grooves on locking arms seat over ridge on coring body. The cap must be seated to seal the sampler.

- Remove the capped sampler by depressing locking lever on the T-handle while twisting and pulling sampler from T-handle.
- Attach the completed perforated label (from the EnCore Sampler™ bag) to cap on coring body.
- Return full EnCore Sampler™ to zipper bag. Seal bag and fill out label on the outside of the bag.

The QA/QC procedures for the analyses are outlined in the QAPP, which is presented in Appendix C of the RFI Work Plan.

3.2 SURFACE SOIL SAMPLING

Surface soil samples will be collected in accordance with the following protocols:

1. Surficial soil samples will be collected using a precleaned stainless steel trowel or other appropriate tool (e.g., hand auger, SPT, Shelby tube, etc). Each sample will consist of soil from the 0-2-foot interval (excluding pavement). Any surficial debris (i.e., grass cover) should be removed from the area where the sample is to be collected using a separate pre-cleaned device.
2. A double pair of disposable nitrile gloves will be used at each sample location (the outer gloves will be changed for each sample);
3. Prior to use, for each sample, all sampling equipment will be precleaned using the prescribed decontamination procedure (refer to SOP 9.0).
4. Samples for VOC analysis will be collected using EnCore Sampler™ as follows:
   - Depress the locking lever on the EnCore T-handle. Place the coring body, rod end first into the open end of the T-handle, aligning the two slots on the coring body with the two locking pins in the T-handle. Twist coring body clockwise to lock pins in slots. Check to ensure sampler is locked in place. The sampler is now ready for use.
   - Turn the T-handle with the T in the upward position and the coring body in the downward position. Using T-handle, push sampler into soil until coring body is completely full. When full, the small o-ring will be centered in T-handle viewing hole. Remove sampler from soil and wipe any excess soil from coring body exterior.

- Cap coring body while it is still on the T-handle. Push and twist cap over bottom until grooves on locking arms seat over ridge on coring body. The cap must be seated to seal the sampler.
- Remove the capped sampler by depressing locking lever on the T-handle while twisting and pulling sampler from T-handle.
- Attach the completed perforated label (from the EnCore Sampler™ bag) to cap on coring body.
- Return full EnCore Sampler™ to zipper bag. Seal bag and fill out label on the outside of the bag.

4. Samples for other chemical analysis will be in accordance with SOP 6.1.
5. The sample locations will be surveyed upon completion.

3.3 BOREHOLE ABANDONMENT

Upon completion of soil boring/sampling, the boreholes will be abandoned/sealed in accordance with SOP 2.5.

4.0 HYDROGEOLOGIC INVESTIGATION

4.1 MONITORING WELL INSTALLATION PROCEDURES

Monitoring well installations will be completed in accordance with U.S. EPA technical guidance and referenced SOPs, where available. Soil samples for stratigraphic definition will be collected continuously in accordance with SOPs 2.3 and 6.1. All cuttings will be containerized and disposed of in accordance with SOP 12.0.

Overburden Monitoring Wells

The overburden monitoring wells will be completed in accordance with SOP 3.2. The following provides specific additional details, as appropriate:

- Due to the anticipated cohesive overburden deposits, a ten foot long, two-inch diameter PVC well screen and PVC riser will be placed into and centered in the augers. The bottom of the well screen will be set at the top of the first confining unit

encountered below saturation. Screen lengths may be modified by the CRA field representative dependent upon the geologic conditions encountered.

- Clean washed, #2 silica sand will be used as the filter pack material, if applicable. Bentonite materials used for borehole sealing and grouting will consist of pH-neutral bentonite.

Bedrock Monitoring Wells

The bedrock monitoring wells will be completed as follows:

1. A borehole will be advanced to depth in accordance with SOPs 2.3, 2.6, 3.2 and 6.1, utilizing water cooled coring and reaming methods.
2. After reaching competent bedrock, or other confining unit, a minimum six-inch diameter low carbon steel outer casing will be lowered into the borehole and grouted in place using a cement-bentonite grout. The grout will be allowed to cure for a 24-hour period prior to recommencement of drilling. Prior to continuation of drilling at the borehole, the surface casing will be flushed with potable water to reduce the chances of contaminant carry-down.
3. Once the casing has set, the borehole will be cored using an appropriate size core barrel in accordance with SOPs 2.4 and 4.7. The competent bedrock will be cored and logged in 20-foot vertical increments. This logging will consist of visual inspection of the cores, borehole diameter (caliper), and optical acoustic televiewer for the first three bedrock wells. Boreholes will not be advanced vertically past any detected separate-phase liquids (refer to SOP 6.4).
4. After the termination depth is reached, the core-hole may be reamed using a tricone roller bit, if a well screen and riser pipe are to be installed, or may be left open for the installation of a multi-level packer system. Cutting generated during reaming will be flushed from the borehole with potable water.

For boreholes where well screen and riser pipe are to be installed:

1. Once the borehole has been reamed, a ten-foot length of two-inch diameter PVC, well screen and PVC, riser pipe will be installed in the borehole.
2. Well construction will be completed in a similar fashion as described in SOP 3.2.

For an open-borehole completion (below the overburden casing);

1. Multi-level packers will be installed if more than one water-bearing zone is encountered to reduce the potential for cross-contamination.
2. The multi-level packer system will be installed to separate encountered water-bearing fracture zones, or upper zones that may potentially transmit water during storm events.
3. Final well completion will consist of the installation of the protective casing, as described in SOP 3.2.

4.2 WATER LEVEL MEASUREMENTS

Following installation of the monitoring wells, a ground elevation and a top of casing (TOC) measuring point elevation survey will be conducted for all recently completed wells. The groundwater elevation at each well will be measured in accordance with SOP 5.1 and will be completed within a 24-hour period in order to avoid readings over potential changes in weather. All data will be reduced to water elevations and contoured, if applicable.

4.3 WELL DEVELOPMENT

All monitoring wells will be developed prior to sampling in accordance with SOP 3.6, upon agreement with U.S. EPA.

4.4 WELL DECOMMISSIONING

All well abandonment/decommissioning will be completed in accordance with SOP 3.7, as necessary.

5.0 GROUNDWATER QUALITY SAMPLING PROTOCOLS

All wells used for groundwater quality monitoring will be sampled utilizing low-flow sampling techniques according to SOP 6.3. Monitoring and sample collection for non-aqueous phase liquid will be completed in accordance with SOP 6.4, if applicable. Wells will not be sampled for groundwater analyses if NAPL is present in the well.

6.0 SEDIMENT SAMPLING PROTOCOLS

Sediment samples will be collected in accordance with the following protocols:

1. A double pair of disposable nitrile gloves will be used at each sample location for chemical analyses. A new overpair will be used for each sample. Additional glove changes will be made as conditions warrant.
2. Prior to use, for each sample, all sampling equipment will be precleaned using the prescribed decontamination procedure (refer to SOP 9.0).
3. Sediment samples will be collected from downstream locations working upstream (as applicable).
4. Sediment samples will be collected using precleaned stainless steel trowels to a maximum depth of 10 centimeters (or 4-inches), if possible
5. Alternatively, an Eckman, or other similar grab sampling equipment, must be used according to the following protocols:
    a. Use the metal handles for water depths less than nine feet, or attach rope to the sampler for water depths greater than nine feet.
    b. Open the sampler on a clean, dry surface.
    c. Place the two release wires around the release pins.
    d. Gently lower the sampler through the water column until the sampler reaches the sediment surface, being careful to maintain proper orientation.
    e. After the sampler is allowed to "settle" into the sediments under its own weight, release the trigger weight to dislodge the release wires and allow the sampler to close.
    f. Gently pull the sampler out of the water and immediately lower into a clean container, taking care to maintain the sampler upright.

g. Open the doors of the sampler at the top and inspect the sample. Samples may be collected if the following conditions are met:

- The sampler is not overfilled to the point that the sediment is touching the top of the sampler, or has extruded beyond the top of the sampler,
- Water is present over the sediment within the sampler,
- The water is not overly turbid,
- The sediment surface is relatively undisturbed, and
- The desired penetration depth is achieved.

5. Sediment samples will be collected from the fine-grained sediment deposition, if possible.

6. All samples will be homogenized (except for VOC analysis) in a precleaned stainless steel bowl and then transferred into the appropriate sample bottles. Samples collected for VOC analysis will not be homogenized but may be collected as previously described in Section .3.0 Subsurface Soil Sampling Protocols (EnCore™ Sampler). Alternatively, sediment samples may be collected in a 4-oz, wide-mouth glass jar (zero head space) for samples that cannot be collected in EnCore Samplers™ (excessively wet, or non-cohesive sediments).

3. The sample locations will be surveyed upon completion.

7.0 SURFACE WATER INVESTIGATION SAMPLING PROTOCOLS

Surface water samples will be collected in accordance with the following protocols:

1. A double pair of disposable nitrile gloves will be used when collecting each surface water sample. Outer gloves will be changed between each sample. Additional glove changes will be made as conditions warrant;

2. Surface water samples will be collected from downstream locations working upstream;

3. Surface water samples will be collected prior to any sediment sampling; and

4. Samples will be collected in accordance with SOP 6.2.

8.0 CONCRETE INSPECTION AND SAMPLING PROTOCOLS

8.1 CONCRETE INSPECTION

Concrete inspection has been performed to assess whether materials from Facility activities could have entered the environment from AOI 3 and AOI 24. The concrete floor and containment was visually inspected for signs of potential conduits to the underlying soil.

8.2 CONCRETE SAMPLING

Wipe samples for PCB analyses will be collected at AOI 3 (former PCB Storage Area with in the Die Cast Building) in accordance with 40 CFR*/*761.123, as follows:

1. Sample locations will be biased toward obvious staining, if present. Otherwise, samples will be collected at the locations shown on Figure 5.3 of the RFI Work Plan.
2. Foundry dust will be removed from the area to be sampled, if necessary.
3. A 10 by 10 cm template will be used to delineate a 100 square centimeter (cm^2) area.
4. A gauze pad or glass wool saturated with hexane will be used to wipe the entire 100 cm^2 area, horizontally and vertically.
5. The gauze pad or glass wool will be sealed in a glass vial and submitted for analysis in accordance with 40 CFR*/*761.272.
6. The sample locations will be measured from permanent features upon completion.

9.0 FIELD LOG AND FIELD FORMS

The written field data will be recorded on standardized, pre-printed field forms or in a field log book, in accordance with SOP 1.5. Examples of field forms are included as Attachment 2.

10.0 SAMPLE SHIPMENT AND CONTAINERS

All samples collected and submitted to the laboratory for analysis will be handled and shipped in accordance with SOP 6.6 and Section 2.0 of this FSP.

TABLE B.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
GENERAL MOTORS POWERTRAIN BEDFORD FACILITY

	AOI	*Sample Matrix*	*Field Parameters*	*Laboratory Parameters*	*Investigative Samples* [3]	*Quality Control Samples*		
						Equipment Blanks [4]	*Field Duplicates* [4]	*MS/MSD* [5]
1	Former Railroad Operations and Minerals Processing Facility	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics, Chloride	21 / 7	1/10	1/10	1/20
		surface water	pH, specific conductance, turbidity, temperature	VOCs, SVOCs, PCBs, Inorganics, Chloride	2 / 2	1/10	1/10	1/20
2	Waste Storage Area	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	12 / 4	1/10	1/10	1/20
3	PCB Storage Areas	concrete [1]	PID VOC screen	PCBs	3 / 3	1/10	1/10	1/20
4	Former North Disposal Area	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	15 / 5	1/10	1/10	1/20
		sediment	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	3 / 3	1/10	1/10	1/20
		surface water	pH, specific conductance, turbidity, temperature	VOCs, SVOCs, PCBs, Inorganics	3 / 3	1/10	1/10	1/20
5	Former East Sand Disposal Area	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	12 / 4	1/10	1/10	1/20
6	Former Sludge Disposal and Fire Training Area	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	21 / 7	1/10	1/10	1/20
7	Former North Lagoon and Outfall 001	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	6 / 2	1/10	1/10	1/20
		groundwater	pH, specific conductance, turbidity, temperature	VOCs, SVOCs, PCBs, Inorganics	1 / 1	1/10	1/10	1/20

TABLE B.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
GENERAL MOTORS POWERTRAIN BEDFORD FACILITY

	AOI	*Sample Matrix*	*Field Parameters*	*Laboratory Parameters*	*Investigative Samples* [3]	*Quality Control Samples*		
						Equipment Blanks [4]	*Field Duplicates* [4]	*MS/MSD* [5]
8	Former South Lagoons and Outfall 002	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	6 / 2	1/10	1/10	1/20
		groundwater	pH, specific conductance, turbidity, temperature	VOCs, SVOCs, PCBs, Inorganics	1 / 1	1/10	1/10	1/20
9	Service Tunnels *	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	18 / 9	1.8	1.8	0.9
10	Existing Stormwater Lagoon and Outfall 003	sediment	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	3 / 3	1/10	1/10	1/20
		surface water	pH, specific conductance, turbidity, temperature	VOCs, SVOCs, PCBs, Inorganics	2 / 2	1/10	1/10	1/20
11	Above Ground Storage Tanks							
	29 Used Oil ASTs	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	9 / 3	1/10	1/10	1/20
	Mineral Oil Release	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	1 / 1	1/10	1/10	1/20
	Diesel Oil Release	soil	PID VOC screen	VOCs, SVOCs, PCBs	1 / 1	1/10	1/10	1/20
12	Area Affected by the Reclaimed Hydraulic Fluid Release	soil	PID VOC screen	VOCs, SVOCs, PCBs	2 / 2	1/10	1/10	1/20
13	Underground Storage Tanks Oil House USTs Piston Yard USTs	Contact IDEM for Closure Approval						

TABLE B.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
GENERAL MOTORS POWERTRAIN BEDFORD FACILITY

	AOI	*Sample Matrix*	*Field Parameters*	*Laboratory Parameters*	*Investigative Samples* [3]	*Quality Control Samples*		
						Equipment Blanks [4]	*Field Duplicates* [4]	*MS/MSD* [5]
14	McBride Cows Disposal Area	soil	PID VOC screen	PCBs	2 / 1	1/10	1/10	1/20
15	Former Equipment Storage Area	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	6 / 2	1/10	1/10	1/20
16	Former East Electrical Substation	none [2]	none [2]	none [2]	0 / 0	1/10	1/10	1/20
17	Piston Building Oil Accumulations	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	6 / 2	1/10	1/10	1/20
18	Area Affected by the Henry System Discharge	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	2 / 2	1/10	1/10	1/20
19	Area Affected by the Paint and Thinner	soil	PID VOC screen	VOCs, PCBs	3 / 1	1/10	1/10	1/20
20	Northern Portion of the Piston Building	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	1 / 1	1/10	1/10	1/20
21	Filled Ravine North of the Die Cast Building	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	12 / 4	1/10	1/10	1/20
22	Tool Room Annex Dock Release	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	1 / 1	1/10	1/10	1/20
23	Area Affected by the 1996 Wastewater Treatment Filter Cake Release	soil	PID VOC screen	VOCs, SVOCs, PCBs, Inorganics	1 / 1	1/10	1/10	1/20

TABLE B.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
GENERAL MOTORS POWERTRAIN BEDFORD FACILITY

	AOI	*Sample Matrix*	*Field Parameters*	*Laboratory Parameters*	*Investigative Samples* [3]	*Quality Control Samples*		
						Equipment Blanks [4]	*Field Duplicates* [4]	*MS/MSD* [5]
24	Area Affected by the June 2000 Die Lube 5150 Release	soil	PID VOC screen	VOCs, SVOCs, PCBs	1 / 1	1/10	1/10	1/20
Site-Wide Hydrogeological Investigation		soil	PID VOC screen	Inorganics, TOC, grain size analysis, hydraulic conductivity	16 / 9	1/10	1/10	1/20
		groundwater	pH, specific conductance, turbidity, temperature	VOCs, SVOCs, PCBs, Inorganics	18 / 9	1/10	1/10	1/20
Background Soil Sampling		soil	PID VOC screen	Inorganics, TOC, grain size	5 / 5	1/10	1/10	1/20
Total [6]					216 104	22	22	11

Notes:

General: Specific analytes are listed in the Quality Assurance Project Plan (QAPP) (Appendix C).
Based on the results of the soil sampling and the site-wide hydrogeological investigation, AOI-specific monitoring wells may be installed.

* = Access to the tunnel areas within the plant is severely limited due to operating machinery and molten metal transfer activities. Completion of this sampling will be coordinated with plant production schedules.

(1) Concrete core samples at the Die Cast Building Former PCB Storage Area consistent with 40 CFR 761.268

(2) It has been determined that this property is not owned by GM. Cinergy Corporation (the local electric utility) owns and operated the property where the former electrical substation was located, and therefore this area is no longer considered an AOI.

(3) Number of investigative samples / number of sample locations. Additional samples may be collected if evidence of contamination is observed.

(4) Based on 1/10 number of investigative samples. Total QA/QC samples are provided at the bottom of the table.

(5) Based on 1/20 number of investigative samples. Total QA/QC samples are provided at the bottom of the table.

(6) Does not include samples that may be collected based on other investigation results or inspection.

ATTACHMENT 1

STANDARD OPERATING PROCEDURES

REMEDIATION SECTION	PROCEDURE # **SOP 1.4**
WORLDWIDE FACILITIES GROUP	EFFECTIVE DATE:
GENERAL MOTORS CORPORATION	
REVISION #: **0**	REVISION DATE:

Utility Clearance

INTRODUCTION

Invasive field investigation activities such as drilling, soil gas surveys, test excavation or remedial construction activities require location of underground utilities prior to initiating work. Such clearance is sound practice in that it minimizes the potential for damage to underground facilities and more importantly, is protective of the health and safety of personnel. Under no circumstances will invasive activities be allowed to proceed without obtaining proper utility clearance by the appropriate public agencies and/or private entities. **This clearance requirement applies to all work on both public and private property, whether located in a dense urban area or a seemingly out-of-the-way rural location.**

The responsibility of obtaining this clearance lies with the Consultant or Contractor performing the work.

In most states such utility clearance is required by law, and obtaining clearance includes contacting a public or private central clearance agency via a "one-call" telephone service and providing them with proposed exploration location information. This is discussed in more detail herein. It is important to note that public utility agencies may not, and usually don't have information regarding utility locations on private property. As such, utility clearance on GM plant property must be cleared using available site drawings, and written approval must be obtained from plant personnel with appropriate knowledge of existing utilities.

PROCEDURES REFERENCED

- 1.1 – Interior Inspections
- 1.2 – Exterior Inspections
- 1.3 – Soil Gas Surveys
- 2.1 – 2.3 – Subsurface Investigations
- 4.1 – EM Surveys

PROCEDURE

- Before marking any proposed exploration or underground construction locations, it is critical that all readily-available information on underground utilities and structures be obtained. This includes publicly-available information as well as information in the possession of private landowners. Any drawings obtained must be reviewed in detail for information pertaining to underground utilities.

- Using the information obtained, the site should be viewed in detail for physical evidence of buried lines or structures, including pavement cuts and patches, variation in or lack of vegetation, variations in grading, etc. (Care must also be taken to avoid overhead utilities as well). Presence of surface elements of buried utilities should be documented, such as manholes, gas or water service valves, catch basins, monuments or other evidence.

- Overhead utility lines must be taken into account when choosing exploration and excavation locations. Most states require a minimum of 10 ft. of clearance between equipment and energized wires. Such separation requirements may also be voltage-based and may vary depending on state or municipality regulations.

  In evaluating clearance from overhead lines, the same restrictions may apply to "drops", or wires on a utility pole connecting overhead and underground lines.

- Using the information obtained and observations made, *proposed* exploration or construction locations should be marked in the field. Marking locations can be accomplished using spray paint on the ground, stakes, or other means. **All markings of proposed locations should be made in white**, in accordance with the generally-accepted universal color code for facilities identification (AWMA 4/99):

  - White: Proposed Excavation or Drilling location
  - Pink: Temporary Survey Markings
  - Red: Electrical Power Lines, Cables, Conduit and Lighting Cables
  - Yellow: Gas, Oil, Steam, Petroleum or Gaseous Materials
  - Orange: Communication, Alarm or Signal Lines, Cables or Conduits
  - Blue: Potable Water
  - Purple: Reclaimed Water, Irrigation and Slurry Lines
  - Green: Sewers and Drain Lines

- In order to effectively evaluate the proposed locations with these entities, detailed, accurate measurements between the proposed locations and existing surface

features should be obtained. Such features can be buildings, street intersections, utility poles, guardrails, etc.

- Obtaining the utility clearance generally involves two entities:
  - The designated "One-Call" underground facilities protection organization for the area; and
  - The landowner.

  Both entities must be contacted and the proposed locations evaluated in light of information available for existing underground facilities. The detailed measurement information described above will be required by the "one call" agency. The owners of the applicable, participating underground utilities are obligated mark their respective facilities at the site in the colors described above. Utility stake-out activities will typically not commence for approximately 72 hours after the initial request is made.

- The public and private utility entities generally only mark the locations of their respective underground facilities within public rights-of-way. Determination of the locations of these facilities on private property will be the responsibility of the project Consultant or Contractor. If available information does not contain sufficient detail to locate underground facilities with a reasonable amount of confidence, alternate measures may be appropriate, as described below. In some cases, the memory of a long-time employee of a facility on private property may be the best or only source of information. It is incumbent on the Consultant or Contractor to exercise caution and use good judgement when faced with uncertainty.

- **Notes: It is important to note that not all utilities are participants in the "one-call" agency or process. As such, inquiries must be made with the "one-call" agency to determine which entities do not participate, so they can be contacted independently.**

  Most utility Stake-outs have a limited time period for which they remain valid, typically two to three weeks. It is critical that this time period be taken into account to prevent expiration of clearance prior to completion of the invasive activities, and the need to repeat the stake-out process.

- Care must be exercised to document receipt of notice from the involved agencies of the presence or absence of utilities in the vicinity of the proposed locations. The attached form SOP 1.3-01 can be used to record contacts made and responses received from each entity.

Most agencies will generally provide a telephone or fax communication indicating the lack of facilities in the project area. **If contact is not made by all of the agencies identified by the "one-call" process, do not assume that such utilities are not present. Re-contact the "one-call" agency to determine the status.**

- For complicated sites with multiple proposed locations and multiple utilities, it is advisable to arrange an on-site meeting with utility representatives. This will minimize the potential for miscommunication amongst the involved parties.

- Completion of the utility stake out process is not a guarantee that underground facilities will not be encountered in excavations or boreholes; in fact, most "one-call" agencies and individual utilities do not offer guarantees, nor do they accept liability for damage that might occur. Accordingly, it is advisable that any invasive activities proceed with extreme caution in the upper four to five feet in the event the clearance has failed to identified an existing facility. This may necessitate hand-excavation or probing to confirm potential presence of shallow utilities. If uncertainty exists for any given utility, extra activities can be initiated to solve utility clearance concerns. These options include:

  Hand digging, augering or probing to expose or reveal shallow utilities and confirm presence and location. In northern climates this may require advancing to below frost line, typically at least four feet.
  Screening the proposed work areas with utility locating devices, and/or hiring a utility locating service to perform this task. The private utility locating service is a growing industry that has formed a national organization. The National Utility Locate Contractors Association (NULCA) can be reached at 715-635-6004.

EQUIPMENT

- White Spray paint
- Wooden stakes, painted white or containing white flagging
- Color-code key
- Available drawings

REFERENCE

- American Public Works Association, April 1999, Uniform Color Code (http://www.apwa.net/)

REMEDIATION SECTION	PROCEDURE # **1.5**
WORLDWIDE FACILITIES GROUP	EFFECTIVE DATE:
GENERAL MOTORS CORPORATION	
REVISION #: **0**	REVISION DATE:

Field Data Recording – Field Books, Log Forms and Electronic Data

INTRODUCTION

This procedure describes protocol for documenting standard investigation activities in the field. Field data serves as the cornerstone for an environmental project, not only for site characterization but for additional phases of investigation or remedial design. Inaccurate or incomplete field data may create significant problems and additional project costs. In addition, recorded field data becomes a legal record of project work, and should be approached with that in mind. Producing legally-defensible data includes proper and appropriate recording of field data as it is obtained in a manner that will preserve it for future use.

This procedure provides guidelines for accurate, thorough collection and preservation of written and electronic field data.

PROCEDURES REFERENCED

- 8.0: Field Instruments: Usage and Calibration

PROCEDURE

Typical field data to be recorded generally includes, but is not limited to, the following:

- general field observations;
- numeric field measurements and instrument readings;
- quantity estimates;
- sample locations and corresponding sample numbers;
- relevant comments and details pertaining to the samples collected;
- documentation of activities, procedures and progress achieved;
- contractor pay item quantities;

- weather conditions;
- a listing of personnel involved in site-related activities;
- a log of conversations, site meetings and other communications; and
- field decisions made and pertinent information associated with the decisions.

Written Field Data

Written field data is generally recorded on one of two media: A standardized, pre-printed field log form, or a bound field log book. In general, use of a field log form is preferable as it prompts field personnel to make appropriate observations and record data in a standardized format. This promotes completeness and consistency from one person to the next. Most of the GM SOPs include standardized field log forms.

In the absence of an appropriate pre-printed form, the data should be recorded in an organized and structured manner in a dedicated project field log book. Log books must be hard-cover, bound so that pages cannot be added or removed, and should be made from high-grade 50% rag paper with a water-resistant surface.

The following are guidelines for use of field log forms and log books:

1. Information must be factual and complete. Do not abbreviate.

2. All entries will be made in black indelible ink with a ballpoint pen and will be written legibly. Do not use "rollerball" or felt tip-style pens, since the water-soluble ink can run or smear in the presence of moisture.

3. All pages in a log book must be consecutively numbered. Field log forms should also be consecutively numbered.

4. Each day's work must start a new log book page.

5. At the end of each day, the current log book page must be *signed and dated* by the field personnel making the entries.

6. When using field log forms, they must also be *signed and dated.*

7. Make data entries immediately upon obtaining the data. Do not make temporary notes in other locations for later transfer to log forms or log books; this only increases the potential for error or loss of data.

8. Entry errors are to be crossed out with a single line, dated and initialed by the person making the correction.

9. Do not leave blanks on log forms, if no entry is applicable for a given data field, indicate so with "NA" or a dash ("--").

10. At the earliest practical time, photocopies of log forms and log book pages should be made and placed in the project file as a backup in the event the book or forms are lost are damaged.

11. Log books should be dedicated to one project only, i.e., do not record data from multiple projects in one log book.

Electronic Data

Electronic data recording is widely used in environmental investigation and remediation projects. In general, it involves electronic measurement of field information through the use of monitoring instruments, sensors, gauges, and equipment controls. The following is a list of guidelines for proper recording and management of electronic field data:

1. Field data management should follow requirements of a project-specific data management plan (DMP), if one exists.

2. Use only instruments that have been calibrated in accordance with manufacturer's recommendations.

3. Usage of instruments, controls and computers for the purpose of obtaining field data should only be performed by personnel properly trained and experienced in the use of the equipment and software.

4. Use only fully-licensed software on PCs and laptops. Software piracy, even if unintentional, *is a felony* and exposes GM, its contractors, and their employees to severe criminal and financial penalties.

5. Loss of electronic files may mean loss of irreplaceable data. Every effort should be made to back up electronic files obtained in the field as soon as practical. A backup file placed on a disk and kept in a separate location from the original will minimize the potential for loss.

6. Electronic files, once transferred from field instruments or laptops to office computers, should be protected if possible to prevent unwanted or inadvertent manipulation or modification of data. Several levels of protection are usually available for spreadsheets, including making a file "read-only" or assigning a password to access the file.

7. Protect floppy disks from exposure to moisture, excessive heat or cold, magnetic fields, or other potentially damaging conditions.

8. Remote monitoring is often used to obtain stored electronic data from site environmental systems. A thorough discussion of this type of electronic field data recording is beyond the scope of this SOP. Such on-site systems are generally capable of storing a limited amount of data as a comma-delimited or spreadsheet file. Users must remotely access the monitoring equipment files via modem or other access, and download the data. In order to minimize the potential for loss of data, access and downloading of data should be performed frequently enough to insure the data storage capacity of the remote equipment is not exceeded.

EQUIPMENT

- 5" by 7" National 407 Field Book, with high-grade 50% rag paper with water-resistant surface, hard-cover, or equivalent;
- Appropriate field log forms;
- Indelible ball point pen (do not use "rollerball" or felt-tip style pens);
- Straight edge;
- Pocket calculator;
- Laptop computer (if required).

REFERENCES

REMEDIATION SECTION	PROCEDURE # SOP 2.3
WORLDWIDE FACILITIES GROUP	EFFECTIVE DATE: May 14, 2001
GENERAL MOTORS CORPORATION	
REVISION #:	REVISION DATE:

SOIL BORINGS

INTRODUCTION

The following presents a description of the methods generally employed for the installation of boreholes and the collection of subsurface soil samples. Boreholes are typically installed to define geologic conditions for hydrogeologic and geotechnical evaluation; to allow the installation of monitoring wells and piezometers; and to allow the collection of subsurface soil samples (generally above the water table) for chemical analysis.

Several manual methods are available for the collection of shallow subsurface soil samples (e.g., hand augers, post-hole augers, vibratory hammers). However, the most common method used by GM to advance boreholes is rotosonic drilling techniques, hollow-stem augers (HSA), or the use of a direct-push equipment. Rotosonic drilling and direct-push techniques are preferred boring approaches at GM Facilities if groundwater samples are being collected and analysis could be affected by tubidity because there is less disturbance of surrounding soil. SOP 2.2 Drilling Techniques/Background, provides insight into the advantages/disadvantages of these drilling methods.

PROCEDURES REFERENCED

- Utility Clearance (SOP 1.4)
- Drilling Techniques (SOP 2.2)
- Soil Classification (SOP 2.6)
- Rock Classification (SOP 2.7)
- Soil Sample Collection (SOP 6.1)

BOREHOLE REQUIREMENTS

The following activities must be undertaken prior to undertaking a borehole installation and subsurface soil sampling program.

i) Assemble all equipment and supplies required per the Work Plan.

ii) Obtain a site plan and any previous stratigraphic logs. Determine the exact number and location of boreholes to be installed and the depths of samples for chemical analysis.

iii) Contact the analytical group to arrange/determine:
- laboratory;
- glassware/sample jars;
- cooler;
- shipping details;
- start date; and
- expected duration.

iv) Establish borehole locations in field using available landmark or by surveying methods if necessary.

v) Arrange for utility clearance of franchised utilities and site utilities.

vi) Determine notification needs with the Project Manager. Have the regulatory groups, landowner, GM Facility personnel, and laboratory been informed of the sampling event?

vii) Determine the methods for handling and disposal of drill cuttings, wash waters, and spent decontamination fluids.

Once the prior planning and preparation activities are completed, the borehole installation and subsurface soil sampling program can proceed. The typical series of events which takes place is:

- locating and marking of borehole locations (if not already completed);
- equipment decontamination;
- final visual examination of proposed drilling area for utility conflicts/final hand auger or post-hole check to verify utility absence;
- advancement of borehole and collection of the soil sample;
- field screening of soil sample;
- description of soil sample;
- sample preparation and packaging;
- abandonment of boreholes;
- surveying of borehole locations and elevations; and
- field note completion and review.

i) Location and Marking of Boreholes/Final Visual Check

The proposed borehole locations marked on the site plan are located in the field and staked. On most sites, this will likely be done several days in advance of the drill rig arriving on site. Unless boreholes are to be installed on a fixed grid, the proposed locations are usually strategically placed to assess site conditions.

Once the final location for the proposed boring has been selected and utility clearances are complete, one last visual check of the immediate area should be performed before drilling proceeds. This last visual check should confirm the locations of any adjacent utilities (subsurface or overhead) and verification of adequate clearance. If gravity sewers or conduits exist in the area, any access manholes or chambers should be opened and the conduit/sewer alignments confirmed. Do not enter manholes unless confined space procedures are followed.

ii) Borehole Advancement

If possible, it is prudent to use a hand auger or post-hole digging equipment to a sufficient depth to verify the absence of buried utilities and pipelines. This procedure should clear the area to the full diameter of the drilling equipment which will follow.

If it is necessary to relocate any proposed borehole due to terrain, utilities, access, etc., the Project Manager must be notified and an alternate location will be selected (utilities must be re-cleared if a boring is moved even by a few feet).

Prior to use and between each borehole location at an environmental site, the drilling and sampling equipment must be decontaminated. All decontamination must be conducted in accordance with the project-specific plans or the methods presented in SOP 9.0

The clean augers/tooling are covered with clean plastic sheeting to prevent contact with foreign materials.

- Collection of soil samples is one of the most important considerations in selecting drilling methods. Therefore, the need for reviewing drilling techniques (SOP 2.2) and the Site objectives must first be considered. Soil Classification will be completed in accordance with SOP 2.6. Sections iii) and iv) describe borehole soil sampling procedures using direct-push tooling and hollow stem augering/split spoon sampling (Standard Penetration Testing - SPT), respectively.

iii) Direct-Push/Macro-Core™ Soil Sampler

The operation of the direct-push/Macro-Core™ Soil Sampler (or equivalent) consists of "pushing" the sampler into the subsurface and then retrieved using a direct-push soil probing machine. The collected soil core is contained within an internal soil liner (acetate, polyethylene or teflon) and removed from the sampler once returned to the ground surface.

Sampler length is variable depending on equipment available (2 ft., 4 ft., 5 ft.). Once the soil liner has been removed and the outer sampler deconed, a new liner is inserted and the sampler reassembled. The clean sampler is then driven back down the same hole to collect the next soil sample.

The Macro-Core™ sampler can be used in either the open-tube or closed-point sampling mode. The open-core sample mode is most commonly used in stable soil conditions. In unstable soils, the piston rod point system prevents collapsed soil from entering the sampler as it is advanced back down the hole. Once at the sample depth, the piston rod is unthreaded and released. The sampler is then driven into the subsurface to fill the sampler with soil, the piston point rides on top of the soil, as it enters the sampler.

Once recovered the soil liner with collected soils is opened (cut lengthwise) and examined to collected soil screening information, soil logging information, and soils for chemical analysis.

iv) Standard Penetration Testing (SPT) Sampling and Testing Procedure

This method is used to obtain representative samples of subsurface soil materials and to determine a measure of the in situ relative density of the subsurface soils. The test methods described below must be followed to obtain accurate SPT values. The split spoon is typically driven in advance of a hollow stem auger string which allows collection of the disturbed but representative sample.

SPT sampling is performed by using a split barrel sampler in accordance with ASTM D1586. The split barrel sampler, or split spoon, consists of an 18- or 24-inch long, 2-inch outside diameter tube, which comes apart length wise into two halves. The split spoon is typically driven in advance of a hollow stem auger string which allows collection of the disturbed but representative soil sample.

Once the borehole is advanced to the target depth and the borehole cleaned of cuttings, representative soil samples are collected in the following manner:

- the split-spoon sampler should be inspected to ensure it is properly cleaned and decontaminated. The driving shoe (tip) should be relatively sharp and free of severe dents and distortions;
- the cleaned split-spoon sampler is attached to the drill rods and lowered into the borehole. Do not allow the sampler to drop onto the soil;
- after the sampler has been lowered to the bottom of the hole, it is given a single blow to seat it and make sure that it is in undisturbed soil. If there still appear to be excessive cuttings in the bottom of the borehole, remove the sampler from the borehole and remove the cuttings; and
- mark the drill rods in three or four successive 6-inch (0.15 m) increments, depending on sampler length, so that the advance of the sampler under the impact of the hammer can be easily observed for each 6-inch (0.15 m) increment.

The sampler is then driven continuously for either 18 or 24 inches (0.45 or 0.60 m) by use of a 140-pound (63.5 kg) hammer. The hammer may be lifted and dropped by either the cathead and rope method, or by using a trip, automatic, or semi-automatic drop system. The hammer should free-fall a distance of 30 inches (±1 inches) (760 mm, ±25 mm) per blow. Measure the drop at least daily to ensure that the drop is correct. To ensure a free-falling hammer, no more than 2 1/4 turns of the rope may be wound around the cathead (see ASTM D1586). The number of blows applied in each 6-inch (0.15 m) increment is counted until one of the following occurs:

- a total of 50 blows have been applied during any one of the 6-inch (0.15 m) increments described above;
- a total of 100 blows have been applied;
- there is no advancement of the sampler during the application of ten successive blows of the hammer (i.e., the spoon is "bouncing" on a stone or bedrock); or
- the sampler has advanced the complete 18 or 24 inches (0.45 or 0.60 m) without the limiting blow counts occurring as described above.

In some cases where the limiting number of blow counts has been exceeded, the Consultant may direct the driller to attempt to drive the sampler more if collection of a greater sample length is essential.

On the field form, record the number of blows required to drive each 6-inch (0.15 m) increment of penetration. The first 6 inches is considered to be a seating drive. The sum of the number of blows required for the second and third 6 inches (0.15 m) of penetration is termed the "standard penetration resistance" or the "N-value".

Note: If the borehole has sloughed and there is caved material in the bottom, the split spoon may push through this under its own weight, but now the spoon is partially "pre-filled". When the spoon is driven the 18 or 24 inches representing its supposedly empty length, the spoon fills completely before the end of the drive interval. Two problems arise:

1. the top part of the sample is not representative of the in-place soil at that depth; and

2. the SPT value will be artificially higher toward the bottom of the drive interval since the spoon was packed full. These conditions should be noted on the field log.

The sampler is then removed from the borehole and unthreaded from the drill rods. The open shoe (cutting end) and head of the sampler are partially unthreaded by the drill crew and the sampler is transferred to the geologist/engineer work surface.

Note: A table made out of two sawhorses and a piece of plywood is appropriate, or a drum, both covered with plastic sheeting.

The open shoe and head are removed by hand, and the sampler is tapped so that the tube separates.

> *Note: Handle each split spoon with clean disposable gloves if environmental issues are being investigated.*

Measure and record the length of sample recovered making sure to discount any sloughed material that is present on top of the sample core.

Caution must be used when conducting SPT sampling below the groundwater table, particularly in sand or silt soils. These soils tend to heave or "blow back" up the borehole due to the difference in hydraulic pressures between the inside of the HSA and the undisturbed soil. To equalize the hydraulic pressure, the inside of the HSA must be filled with water or drilling mud. The drilling fluid level within the boring or hollow-stem augers needs to be maintained at or above the in situ groundwater level at all times during drilling, removal of drill rods, and sampling. Since heave or blow back is not always obvious to the driller, it is essential that the water level in the borehole always be maintained at or above the groundwater level.

Heaving conditions and the use of water or mud should be noted on the field logs.

SPT sampling below the water table in sands and silt occasionally results in low SPT values being obtained due to the heaving effect disturbing the soil especially if the water level in the hole has not been maintained at the in situ water level. Suspect low N values should be noted on the field logs. If it is critical to have accurate N values below the water table, other methods can be employed, such as conducting a dynamic cone penetration test. This quick and easy test involves attaching a cone shaped tip to the end of the drill rods, and driving the tip into the ground similar to the SPT method, except that the borehole is not pre-augered. Cones may be driven 20 to 40 feet through a formation without augering. Blow counts are recorded for each foot (0.3 m) of advancement.

A variation of split barrel sampling involves the use of a longer barrel in conjunction with hollow stem augers. The sampling barrel is installed inside the auger with a swivel attachment to limit rotation of the barrel. After completion of a 5-foot auger penetration, the auger is left in place and the barrel retrieved from the borehole. The sampler should be handled and the sample retrieved in the same way as described above for SPT sampling.

Thin-Walled Samplers (Shelby Tubes)

Thin-walled samplers are used to collect relatively undisturbed samples (as compared to split-spoon samples) of soft to stiff clayey soils. Shelby tubes are commonly used. The Shelby Tube has an outside diameter of 2 or 3 inches and is 3 feet long. These undisturbed samples are used for certain laboratory tests of structural properties (consolidation, hydraulic conductivity, shear strength) or other tests that might be influenced by sample

disturbance. Procedures for conducting thin-walled tube sampling are provided in ASTM D1587, and are briefly described below.

- the soil deposit being sampled must be cohesive in nature, and relatively free of sand, gravel, and cobble materials, as contact with these materials will damage the sampler;
- clean out the borehole to the sampling elevation using whatever method is preferred that will ensure the material to be sampled is not disturbed. If groundwater is encountered, maintain the liquid level in the borehole at or above groundwater level during the sampling operation;
- bottom discharge bits are not permitted. Side discharge bits may be used, with caution. Jetting through an open-tube sampler to clean out the borehole to sampling elevation is not permitted. Remove loose material from the center of a casing or hollow-stem auger as carefully as possible to avoid disturbance of the material to be sampled;
- place the sample tube so that its bottom rests on the bottom of the hole. Advance the sampler into the formation without rotation by a continuous and relatively rapid motion; usually hydraulic pressure is applied to the top of the drill rods;
- determine the length of advance by the resistance and condition of the formation, but the length shall never exceed 5 to 10 diameters of the tube in sands and 10 to 15 diameters of the tube in clays;
- in no case should the length of advance be greater than the sample-tube length minus an allowance for the sampler head and a minimum of 3 inches for cuttings.
- the tube may be rotated to shear the bottom of the sample 2 to 3 minutes after pressing in, and prior to retrieval to ensure the sample does not slide out of the tube. Lift the weight of the rods off of the tube prior to rotating.
- withdraw the sampler from the formation as carefully as possible in order to minimize disturbance of the sample;
- package and transport the sample in accordance with Paragraph ix).

On occasion it maybe required to extraction of the sample from the tube be conducted in the field.

- a sample extruder, which consists of a clamp arrangement to hold the tube and a hydraulic ram to push the sample through the tube, is usually mounted on the side of the rig. To prevent cross-contamination, be certain that the extruder is field cleaned between each sample;
- the sample is then extruded into a carrying tray; these are often made from a piece of 4-inch or 6-inch diameter PVC pipe cut lengthwise. Be certain that the carrying tray is field cleaned between each sample. The sample is carried to the work station to describe the sample, trim the potentially cross contaminated exterior, and place it in the appropriate container; and
- the Shelby tube may then be thoroughly field cleaned and decontaminated for reuse. Since they are thin-walled, the tubes are easily damaged, crimped, or otherwise distorted during handling or pushing. The Shelby Tube should be inspected before use and any which are significantly damaged should be rejected.

v) Borehole Completion

At the completion of the soil boring, once the soil/groundwater samples have been collected, the borehole annulus is then abandoned. Borehole abandonment options are identified in SOP 2.5 - Borehole Abandonment/Sealing. Each boring will be surveyed to establish vertical/horizontal information; field ties (i.e., swing ties) will also be collected to document the boring location. Once completed, a stratigraphic log will be prepared for reporting purposes.

EQUIPMENT

- Drilling Equipment
- Stratigraphy Log (Overburden)
- Tape Measure

REFERENCE

1. ASTM D420-93 Guide to Site Characterization for Engineering, Design, and Construction Purposes
2. ASTM D1452-80 Practice for Soil Investigation and Sampling by Auger Borings
3. ASTM D1586-84 Test Method for Penetration Test and Split-Barrel Sampling of Soils
4. ASTM D1587-94 Practice for Thin-Walled Tube Geotechnical Sampling of Soils
5. ASTM D2488-93 Practice for Description and Identification of Soils (Visual-Manual Procedure)
6. National Water Well Association, Handbook of Suggested Practices for the Design and Installation of Ground-Water Monitoring Wells.1989
7. EPA OSWER-9950.1,1986. RCRA Ground-Water Monitoring Technical Enforcement Guidance Document

REMEDIATION SECTION	PROCEDURE #SOP - 2.4
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SUBSURFACE INVESTIGATION ACTIVITIES: BEDROCK CORING

INTRODUCTION

The procedure procedures describes the methodology for bedrock coring.

PROCEDURES REFERENCED

- Bedrock Logging and Classification (SOP - 2.7)

CORING PROCEDURE

- Prior to initiating coring activities, ensure that the overburden portion of the hole is isolated from the bedrock portion of the hole using an overburden casing, routed in-place.
- Coring must be performed utilizing an approved coring method and size.
- Potable water or air can be utilized as circulating medium.
- If required, all rock cuttings produced will be properly contained and disposed of in accordance with the Work Plan requirements.
- All coring activities shall be performed following procedures outlined in ASTM Standard D2113.
- All bedrock core runs should be completed without interruption so penetration rates can be determined.
- Upon completion of bedrock coring activities, the corehole shall be flushed to remove all residual bedrock cuttings and measured to confirm final depth

EQUIPMENT/MATERIALS

- Drilling equipment
- Appropriate coring equipment
- Bedrock stratigraphic log form
- Tape measure
- Hand lense
- Camera
- Work Plan
- Health and Safety Plan

REFERENCE

1. American Society for Testing and Materials (1991) Standard D2113-8307 *"Standard Practice for Diamond Core Drilling for Site Investigations"* Annual Book of ASTM Standards, Section 4, volume 04.08
2. American Society for Testing and Material (1991) Standard D5434-93 *"Standard Guide for Field Logging of Subsurface Exploration of Soil and Rock"* Annual Book of ASTM Standards, Section 4, Volume 04.09

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SUBSURFACE INVESTIGATION ACTIVITIES: BOREHOLE ABANDONMENT/SEALING

INTRODUCTION

The following procedure describes common techniques for the abandonment/sealing of overburden boreholes. Borehole completion may have been performed by a rotosonic drilling technique, direct push sampling device, hollow stem augering/split spoon sampling, solid stem augering or other soil sample collection techniques. The method of borehole abandonment selected for a program will be dependent on a number of factors such as: depth to groundwater, presence of contamination (and degree of contamination i.e., light or dense non-aqueous phase liquids - NAPL), confining layer presence and/or physical setting (i.e., open field/vacant land, vs. facility setting). The Work Plan guiding these activities (soil boring/boring closure) will dictate which method of borehole abandonment/seating is required. Rotosonic drilling methods are often preferred for soil boring activities on GM sites where appropriate. The borehole abandonment/sealing techniques reviewed in the following consist of:

- soil cutting backfill;
- bentonite chip backfill; or
- cement/bentonite grout backfill using tremie techniques.

Boreholes need to be abandoned and sealed properly to prevent surface water entry to the groundwater regime, to eliminate any physical hazard, and to prevent/protect groundwater movement from one aquifer to another.

PROCEDURES REFERENCED

- Soil Boring (SOP-2.3)

BOREHOLE ABANDONMENT/SEALING PROCEDURE

A. Soil Cutting Backfill

Typically employed when working above groundwater table and at shallow depths (maximum depth 2 feet).

- The final depth of borehole will be measured and recorded.
- Cuttings are dropped into borehole after sample equipment is removed.
- Drill rod and/or probe rodding is used to compact/compress cuttings to allow return of all cuttings back into borehole.
- Mound final surface of cuttings above ground surface to allow settlement and promote surface water runoff away from boring. Final restoration will be completed in accordance with needs of the GM Facilities representative.
- Borehole abandonment will be documented in field records/notes.

B. Bentonite Chip Backfill

Typically employed when working above of just into the groundwater table

- Excess cuttings have been drummed for disposal or excess cuttings have been spread at ground surface.
- The depth of the borehole will be measured and recorded.
- Bentonite chips will be dropped into borehole as hollow stem augers are removed, or after the boring equipment has been removed from the borehole (solid stem auger, probing tools, split spoon samplers).
- Sufficient water will be needed to hydrate bentonite chips as they are place.
- The bentonite chip backfill will be extended to within 1 foot of ground surface, the final borehole space will be backfilled with native soil and mounded slightly to allow settlement and promote surface water runoff away from the boring. Alternatively, the borehole cuttings may be mixed with bentonite to complete the abandonment/sealing task. Final restoration will be completed in accordance with needs of the GM Facilities representative.
- Borehole abandonment will be documented in field records/notes.

C. Cement/Bentonite Grout Backfill

Typically employed when working below the groundwater table, or in an area where a confining layer exists and the potential for groundwater/NAPL movement along a preferential pathway (i.e., former borehole) must be eliminated.

- The final depth of borehole will be measured and recorded.
- The volume of grout required will be calculated from the above measurements.
- A grout mix of one bag (94 lbs.) of Portland Cement and three pounds of bentonite with approximately 7.5 gallons of clean water will be prepared.
- Using a tremie tube placed at the base of the borehole the grout will be pumped until observed at the required elevation. The tremie tube will be raised as the grout level rises (positive displacement technique).
- The bentonite/grout backfill will be extended to within 1 foot of ground surface, the final borehole space will be backfilled with native soil and mounded slightly to allow

settlement and promote surface water runoff away from boring. Final restoration will be completed in accordance with the GM Facilities representative.

- Borehole abandonment will be documented, noting depth of borehole, volume of grout used and mix ratio.
- Groundwater displaced from the borehole may or may not required containment depending on borehole setting and/or water quality.

NOTE: AT THE COMPLETION OF BOREHOLE ABANDONMENT/SEALING ACTIVITIES (REGARDLESS OF METHODOLOGY EMPLOYED) IT IS NECESSARY TO CHECK FOR SURFACE SETTLEMENT A FEW DAYS AFTER WORK COMPLETION TO DETERMINE IF THE BOREHOLE AREA REQUIRES "TOPPING OFF".

Final Restoration

The area around the borehole and the borehole surface shall be restored as directed by the GM Facility representative (e.g., asphalt, concrete, vegetation). Time for borehole settlement may be permitted, then final restoration performed; or alternatively final restoration may be required immediately in active interior work areas.

Cleanup

The area around the borehole shall be completed cleaned up of any investigation related materials (litter, etc.).

EQUIPMENT/MATERIALS

- Grout pump/mixing equipment

REFERENCE

1. United States Environmental Protection Agency (1992) "Guide to Management of Investigation-Derived Wastes", Quick Reference Fact Sheet
2. ASTM D5299 "Guide for Decommissioning of Ground Water Wells, Vadose Zone Monitoring Devices, Boreholes and Other Devices for Environmental Activities

REMEDIATION SECTION	PROCEDURE # SOP-2.6
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SOIL CLASSIFICATION

INTRODUCTION

The stratigraphic log is a factual description of the soil at the borehole location and is relied upon to interpret the soil characteristics, and their influence and significance in the subsurface environment. The accuracy of the stratigraphic log is to be verified by the person responsible for interpreting subsurface conditions. An accurate description of the soil stratigraphy is essential for a reasonable understanding of the subsurface conditions. Confirmation of the field description by examination of representative soil samples by the project geologist, hydrogeologist, or geotechnical engineer (whenever practicable) is recommended.

The ability to describe and classify soil correctly is a skill that is learned from a person with experience and by systematic training and comparison of laboratory results to field descriptions.

It is GM's Policy to log soils according to the Unified Soil Classification System (USCS) described in the following.

PROCEDURES REFERENCED

- Test Pits (SOP-2.1)
- Soil Borings (SOP-2.3)

DESCRIPTIONS

Several methods for classifying and describing soils or unconsolidated sediments are in relatively widespread use. The Unified Soil Classification System (USCS) is the most common. With the USCS, a soil is first classified according to whether it is predominantly coarse-grained or fine-grained.

The description of fill soil is similar to that of natural undisturbed soil except that it is identified as fill and not classified by USCS group, relative density, or consistency. Those logging soils must attempt to distinguish between soils that have been placed (i.e., fill) and not naturally present; or soils that have been naturally present but disturbed (i.e., disturbed native).

It is necessary to identify and group soil samples consistently to determine the subsurface pattern or changes and non-conformities in soil stratigraphy in the field at the time of drilling. The stratigraphy in each borehole during drilling is to be compared to the stratigraphy found at the previously completed boreholes to ensure that pattern or changes in soil stratigraphy are noted and that consistent terminology is used.

Visual examination, physical observations and manual tests (adapted from ASTM D2488, visual-manual procedures) are used to classify and group soil samples in the field and are summarized in this subsection. ASTM D2488 should be reviewed for detailed explanations of the procedures. Visual-manual procedures used for soil identification and classification include:

- visual determination of grain size, soil gradation, and percentage fines;
- dry strength, dilatancy, toughness, and plasticity (thread or ribbon test) tests for identification of inorganic fine grained soil (e.g., CL, CH, ML, or MH); and
- soil compressive strength and consistency estimates based on thumb indent and pocket penetrometer (preferred) methods.

The three main soil divisions are: coarse grained soil (e.g., sand and gravel), fine grained soil (e.g., silt and clay), and soil with high natural organic matter content (e.g., peat and marl).

Coarse Grained Soil

The USCS group symbols for coarse grained soil are primarily based on grain or particle size, grain size distribution (gradation), and percent fines (silt and clay content).

Coarse-grained soils are then further subdivided according to the predominance of sand and gravel. Course grained soil is made up of more than 50 percent, by weight, sand size, or larger (75 μm diameter, No. 200 sieve size or larger). It is noted that there are other definitions for coarse grained or coarse textured soil and for sand size such as soil having greater than 70 percent particles equal to or greater than 50 μm diameter.

Descriptions for grain size distribution of soil include; poorly graded (i.e., soil having a uniform grain size, SP and GP) and well graded (i.e., poorly sorted; having wide range of particle sizes with substantial intermediate sizes, SW and GW).

Coarse grained soils are further classified based on the percentage of silt and clay it contains (fines content). Coarse grained soils containing greater than 12 percent fines is commonly described as dirty. This description arises from the soil particles that adhere when the soil is rubbed between the hands or adhere to the sides of the jar after shaking or rolling the soil in the jar. The jar shake test which results in segregation of the sand and gravel particles is also used as a visual aid in determining gravel and sand percentages.

Examples of the group symbol, name, and adjectives used to describe the primary, secondary, and minor components of soil are; GW - Sandy Gravel (e.g., 70 percent gravel and 30 percent sand) or Sandy Gravel trace silt (less than 10 percent silt), and SP - Sand, uniform.

Relative density is an important parameter in establishing the engineering properties and behavior of coarse grained soil. Relative density of non-cohesive (granular) soil is determined from standard penetration test (SPT) blow counts (N values) (after ASTM Method D1586).

The SPT gives a reliable indication of relative density in sand and fine gravel. N values in coarse grained soil are influenced by a number of factors that can result in overestimates of relative density (e.g., in coarse gravel and dilatent silty fine sand) and can be conservative and underestimate the relative density (e.g., sand below the groundwater table and uniform coarse sand). These effects will be assessed by the project geotechnical engineer, if required, and need not be taken into account by field personnel.

Other dynamic methods, such as modified SPT and cone penetration tests, are used on occasion to supplement or replace the SPT method for certain site-specific conditions. The details of all modifications to the SPT or substitute methods should be recorded as they are required to interpret test results and correlate to relative density.

Fine Grained Soil

A soil is fine grained if it is made up of half or more of clay and silt (i.e., fines greater than 50 percent by weight passing the 75 μm (No. 200) sieve size). A description of visual-manual field methods and criteria (after ASTM D2488) that are used to further characterize and group fine grained soil (e.g., CL, CH, ML, or MH) including dry strength, dilatancy, toughness, and plasticity (thread or ribbon test) follows. Fine grained soils are subdivided on a basis of the liquid limit and the degree of plasticity.

The accurate identification of silts and clays can be aided by the use of some single field tests. Clay is sticky, will smear readily, and can be rolled into a thin thread even when the moisture content is low. When it is dry clay forms hard lumps. Silt on the other hand, has a low dry strength, can be rolled into threads only at high moisture content, and a wet silt sample will puddle when it is tapped.

CRITERIA FOR DESCRIBING DRY STRENGTH

Description	*Criteria*
None	The dry specimen crumbles into powder with mere pressure of handling.
Low	The dry specimen crumbles into powder with some finger pressure.
Medium	The dry specimen breaks into pieces or crumbles with considerable finger pressure.

High	The dry specimen crumbles into powder with finger pressure. Specimen will break into pieces between thumb and a hard surface.
Very High	The dry specimen cannot be broken between the thumb and a hard surface.

CRITERIA FOR DESCRIBING DILATANCY

Description	*Criteria*
None	No visible change in small wetted specimen when rapidly shaken in palm of hand.
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing.
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing or stretching.

CRITERIA FOR DESCRIBING TOUGHNESS

Description	*Criteria*
Low	Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft.
Medium	Medium pressure is required to roll the thread to near the plastic limit. The thread and the lump have medium stiffness.
High	Considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have very high stiffness.

CRITERIA FOR DESCRIBING PLASTICITY

Description	*Criteria*
Nonplastic	A 1/8-inch (3 mm) thread cannot be rolled at any water content.
Low	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit.
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be re-rolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit.
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be re-rolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit.

Examples of group symbol identification based on visual-manual procedures and criteria for describing fine grained soil are:

Group Symbol	*Dry Strength Plasticity*	*Dilatancy*	*Toughness*
ML	None to low	Slow to rapid	Low or thread cannot be formed
CL	Slight Medium to high	None to slow	Medium
MH	Low Low to medium	None to slow	Low to medium Low
CH	High to very high	None	High High

A requirement for positive classification by USCS group symbols (as described in Test Method ASTM D2487) is laboratory determination of particle size characteristics, liquid limit and plasticity index. The need for this type of testing will be determined by the project geologist, hydrogeologist, or geotechnical engineer.

Examples of name terminology that accompanies the group symbols are ML - Sandy Silt (e.g., 30 percent sand) and CL - Lean Clay with sand (e.g., 15 to 29 percent sand).

The correlation between N value and consistency for clays is rather unreliable. It is preferable to determine consistency using more appropriate static test methods, particularly for very soft to stiff clay soil. N value estimates of consistency are more reasonable for hard clay.

Unconfined compressive strength (Su) may be estimated in the field from the pocket penetrometer test method. To obtain a pocket penetrometer estimate of consistency and compressive strength, the soil core is cut perpendicular to the core length, the length of core (minimum 4 inches) is held in the hand and a moderate confining pressure is applied to the core (not sufficient to deform the core); the penetrometer piston tip is slowly inserted into the perpendicular face of the core until the penetrometer indents into the soil core to the mark indicated on the tip of the penetrometer piston; the penetrometer estimate of soil compressive strength (Su) is the direct reading of the value mark on the graduated shaft (in tons per square foot or other unit of pressure as indicated) indicated by the shaft ring marker, or in some models, by the graduated piston reading at the shaft body. To obtain an average estimate, this procedure is completed several times on both ends and mid cross-section of the core. For Shelby Tube (or thin wall sampler) samples the pocket penetrometer tip is applied to the exposed bottom of the sample at several locations.

Estimates of compressive strength for clay soil of very soft to stiff consistency are better established by in situ shear vane tests or other static test methods.

The description of consistency (or strength) is an important element in determining the engineering properties and strength characteristics of fine grained cohesive soil. Consistency terms (e.g., soft, hard) are based on the unconfined compressive strength (Su) and shear strength or cohesion (cu) of the soil.

The ease and pattern of soil vapor and groundwater movement in the subsurface is influenced by the natural structure of the soil. Soil structure, for the most part, depends on the deposition method and, to a lesser extent, climate.

Visual Appearance/Other Features

Those logging soils should also note the presence, depth and components of fill soils (if evident), and note the distinction between disturbed native soils (i.e., excavation likely performed) vs. undisturbed native soils.

Other features such as root presence/structure, and soil fractures should also be recorded. Soil fractures should be described noting fracture orientation (i.e., horizontal/vertical), length/aperture and appearance of soil infilling, oxidation and/or weathering (if present).

FIELD SCREENING

Field Sample Screening

Upon the collection of soil samples, the soil is screened with a photoionization detector (PID) for the presence of organic vapor. This is accomplished by running the PID across the soil sample. Record the highest reading and sustained readings.

Note: The PID measurement must be done upwind of the excavating equipment or any running engines so that exhaust fumes will not affect the measurements.

Another method of field screening is head space measurements. This consists of placing a portion of the soil sample in a sealable glass jar, placing aluminum foil over the jar top, and tightening the lid. Alternatively, plastic sealable bags maybe utilized for field screen in lieu of glass containers. The jar should only be partially filled. Shake the jar and set aside for at least 30 minutes. After the sample has equilibrated, the lid of the jar can be opened; the foil is punctured with the PID probe and the air (headspace) above the soil sample is monitored. Record this headspace reading on the field form or in the field book.

Note: Perform all headspace readings in an area that is not subject to wind. Also, in the winter, it is necessary to allow the samples to equilibrate in a warm area (e.g., site trailer, van, etc.). This requirement is dictated by the Work Plan.

All head space measurements must be completed under similar conditions to allow comparability of results.

NAPL DETECTION

During soil examination and logging, the sampler shall carefully check for the presence of light or dense NAPL. NAPL may be present in gross amounts or present in small/minute quantities. The adjectives and corresponding quantities used when describing NAPL within a soil matrix are as follows:

Visual Description	*Fraction of Soil Pore Volume Containing NAPL*
Saturated	>0.5
Some	0.5 - 0.25
Trace	<0.25

A complete description of NAPL, must describe the following:

- color;
- quantity;
- density (compared to water i.e., light/floats or heavy/sinks);
- odor (if observed); and
- viscosity (i.e., mobile/flowable, non-mobile/highly viscous-tar like).

The presence of an "iridescent sheen" by itself does not constitute 'NAPL presence', but may be an indicator that NAPL is close to the area.

NAPL presence within a soil matrix may be confirmed by placing a small soil sample within water, shaking, and observing for NAPL separation (i.e., light or dense), from the soil matrix.

Trace amounts of NAPL are identified/confirmed by a close visual examination of the soil matrix, [i.e., separate soil by hand (wearing disposable gloves)] and perform a careful inspection of the soil separation planes/soil grains for NAPL presence.

Often during the sample examination with a knife, an iridescent sheen will be noted on the soil surface (i.e., clay/silts) if the knife has passed through an area of NAPL.

There are a number of more sophisticated tests available to confirm/identify NAPL presence, these are:

- UV fluorescent analysis;
- hydrophobic dyes;
- centrifugation; and
- chemical analysis.

Typically consultants will utilize organic vapor detection results, visual examination, soil/water shake testing, and chemical analysis, to confirm NAPL presence. The more complex techniques described may be incorporated on sites where clear colorless NAPL is present and its field identification is critical to the program.

Note: When describing the presence of vegetative matter in the soil sample, do not use the term "organic" as this often leads to confusion with regards to the presence of organic chemicals (i.e., NAPL).

EQUIPMENT/MATERIALS

- Pocket knife or small spatula
- Small handheld lense
- Stratigraphic Log (Overburden) (Form 2.6-01)
- Tape Measure

REFERENCE

1. American Society for Testing and Materials (1991), Standard D1452-80, "Practice for Soil Investigation and Sampling by Auger Borings", Annual Book of ASTM Standard, Section 4, Volume 04.08.
2. ASTM Standards on Environmental Sampling (1995), Standard D 2488-93, "Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)"
3. ASTM Standards on Environmental Sampling (1995), Standard D 4700-91, " Guide for Soil Sampling from the Vadose Zone".
4. ASTM Standards on Environmental Sampling (1995), Standard D 1586-92, " Test Method for Penetration Test and Split-Barrel Sampling of Soils".
5. ASTM Standard D 2487, " Classification of Soils for Engineering Purposes (Unified Soil Classification System)".
6. Geotechnical Guage, Manufactured by W.F. McCollough, Beltsville, MD.
7. Sand Grading Chart, by Geological Specialty Company, Northport, Alabama.

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SUBSURFACE INVESTIGATION ACTIVITIES: BEDROCK LOGGING AND CLASSIFICATION

INTRODUCTION

This procedure is for the Logging and Classification of bedrock cores.

PROCEDURES REFERENCED

- Bedrock Coring (SOP - 2.4)

BEDROCK LOGGING AND CLASSIFICATION

- All Bedrock logging activities will be conducted according to procedures outlined in ASTM Standard D5434-93.
- All retrieved bedrock core shall be handled in a manner as to cause the least amount of mechanical fractures as possible.
- All retrieved bedrock cores will be placed in an appropriate sized core box with increasing depths aligned left to right.
- All bedrock core runs shall be separated and core depths marked utilizing wooden blocks.
- Upon the completion of each core run, the depth of corehole will be measured to properly document the termination depth of each core run.
- Each stratigraphic bedrock core run will be logged for all structural and lithographic features.
- All natural occurring fractures, structural and lithographic features and porosity will be logged for depth and documented on standard Bedrock Stratigraphic Log Forms.
- Rock Quality Designation (RQD) values and documentation on the bedrock log form will be calculated for each bedrock core run.
- RQD values will be calculated to indicate rock-mass properties according to Deere (1986) by summarizing all the bedrock core portions greater than 4-inches in length and dividing the sum of these pieces by the length of the bedrock core run. RQD is expressed as a percentage.

- The percentage of bedrock core recovery for each core run will be calculated and recorded on the bedrock log form.
- If potable water is utilized as a circulating medium, the volume of water lost during each bedrock core run will be recorded on the bedrock log form.
- Special attention will be paid to fracture surfaces to indicate if any fracture infilling or groundwater movement is indicated. All fractures will be measured for depth and recorded on the bedrock log form.
- A picture of each run of bedrock core will be taken to document each retrieved bedrock core run.
- Each completed core box fill be properly sealed to keep the bedrock core entact.
- Each core box will be labeled on the outside to include Site name, job number, boring number, date, bedrock core depth, interval, bedrock core run number, RQD and bedrock core recovery for each core run, fluid loss (if applicable) and bedrock core loggers name.
- Ensure that all bedrock coring equipment is properly decontaminated according to Site protocols prior to construction of the next well.

EQUIPMENT/MATERIALS

- Drilling equipment
- Appropriate coring equipment
- Bedrock stratigraphic log form
- Tape measure
- Hand lense
- Camera
- Work Plan
- Health and Safety Plan

REFERENCE

1. American Society for Testing and Materials (1991) Standard D2113-8307 *"Standard Practice for Diamond Core Drilling for Site Investigations"* Annual Book of ASTM Standards, Section 4, volume 04.08
2. American Society for Testing and Material (1991) Standard D5434-93 *"Standard Guide for Field Logging of Subsurface Exploration of Soil and Rock"* Annual Book of ASTM Standards, Section 4, Volume 04.09

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PROCEDURES FOR OVERBURDEN MONITORING WELL INSTALLATION

INTRODUCTION

This procedure presents the drilling, installation, and completion of overburden monitoring wells. The design and installation of monitoring wells involve the drilling of boreholes into various types of geologic formations. Designing and installing monitoring wells may require different drilling methods and installation procedure. This procedure provides the most common type of well installation in the overburden. However due to specific site and monitoring requirements the procedure may be revised as necessary to reflect these site specific needs. More detail as to well construction and installation will be located in the site-specific Field Sampling Plan.

Prior planning is required for the selection of the appropriate drilling method and selection of well materials.

PROCEDURES REFERENCED

- Subsurface Soil Sampling
- Soil Classification
- Well Development
- Decontamination and Waste Disposal

DRILLING PROCEDURES

- Drilling and sampling equipment arriving on site will be decontaminated prior to drilling in accordance with the Decontamination and Waste Disposal Procedure.

- Drilling generated waste materials will be disposed according to the Decontamination and Waste Disposal Procedure.

- Record construction and geologic information on the attached subsurface boring log.

- The borehole diameter shall be sufficient that well construction may proceed without any major difficulties and to ensure sufficient filtering capability of the sand. At a minimum the final well borehole shall be sufficient to provide a minimum of 3 inch clearance between the well material and sidewalls of the borehole. For most wells the well materials will be 2-inch diameter therefore a borehole of 8 inches will be required.

- The depth to the target interval may be determined from an existing adjacent monitoring well/boring or from information obtained from split spoon sampling of the borehole. The criteria for determining the target interval to be monitored will be presented in the Project Work Plan and Field Sampling Plan. A minimum 8-inch diameter borehole will be advanced to the target interval using in the order of preference rotosonic drilling, spun casing drilling or use of 4 1/4 -inch inside diameter hollow stem augers.

- In the instances where the borehole is advanced deeper than the target interval, a bentonite pellet seal will be tremied into the hole to bring the bottom of the boring to within 6 inches of the target interval. Six inches of filter sand will then be placed above the bentonite seal prior to further well installation.

- In some areas where the water table is known to be at or near the top of bedrock, the base of the overburden well will be installed into the top of bedrock.

- Split-spoon samples may be collected, if required by applicable project Work Plan. (see Subsurface Soil Sampling Procedure). If collected, soil samples will be classified in accordance with the Soil Classification Procedure.

WELL CONSTRUCTION

- The well construction procedures presented below are the recommended standards. However, due to variations in subsurface conditions, changes in these well construction standards may be necessary in order to facilitate the installation of the protective casing.

- Overburden wells will be constructed of either 2-inch Schedule 40 Flush-threaded Virgin PVC or type 316 Stainless Steel. Type of well material to use will be dependent upon known subsurface conditions. Wells constructed of PVC are preferable however in situations where sufficient levels of chlorinated solvents are present to affect the PVC well integrity, stainless steel will be the material of choice. Wells of mixed construction materials are not acceptable. The well screen will consist of machine slot or continuous wrap PVC or Stainless Steel with screen slot size appropriate for the type of subsurface material. Refer to SOP 3.1 for decision making analysis matrix.

- The bottom of the well screen will be placed to the bottom of the borehole. Ideally, the top of the well screen should be greater than 4 feet below grade. As the augers are slowly removed, clean washed silica sand filter pack will be placed in the annular space around the well screen and casing from the base of the screen to at least 2 feet above the screen.

- In wells that exhibit a water table elevation above the sand pack, a minimum of 2-foot thick layer of bentonite pellets will be placed above the sand pack. The seal will be hydrated and allowed to set for approximately 45 minutes.

- Cement/bentonite grout will be placed from the top of the bentonite seal to a point 5 feet below existing ground surface where conditions allow. The grout will be prepared in the ratio of one bag (94 pounds) of Type I or Type II Portland Cement to 3 to 5 pounds of bentonite powder mixed with approximately 7 gallons of potable water.

- Accurate measurements of the material depths will be made by sounding the annulus during installation. The volume of materials needed will be calculated and compared to the actual volume used. Material depths will be recorded on the subsurface boring logs.

- The well casing will be secured with a vented lockable cap. If the well is located in a high traffic area, the casing will be protected by 9-inch flush-mounted roadway box set in a concrete seal, or above ground casing and three bumper posts (concrete filled steel posts set in concrete approximately 3 to 4 feet from the protective casing).

Alternatively, in low traffic areas, the well casing may be cut above grade and completed with 4 or 6-inch diameter steel protective casing with approximately 3 feet of stick up, set in a concrete surface seal.

- For flush-mounted wells, a 9-inch diameter, water-tight protector will be installed complete with a sand drain. A lockable gripper plug will top the casing.

- After installation, the monitoring well will be labeled with the well identification and a reference point for water level and depth measurements will be notched into the well casing. The well will be allowed to sit for at least 24 hours prior to well development, and for two to three weeks between development and groundwater sampling.

EQUIPMENT

- Drilling Equipment
- Well Supplies
- Subsurface Boring Log
- Tape Measure

REFERENCE

1. American Society for Testing and Materials (1991), Standard D1452-80, "Practice for Soil Investigation and Sampling by Auger Borings", Annual Book of ASTM Standard, Section 4, Volume 04.08.

2. American Society for Testing and Materials (1991), Standard D2113-83 (87), "Diamond Core Drilling for Site Investigations", Annual Book of ASTM Standards, Section 4, Volume 04.08.

3. American Society for Testing and Materials (1991), Standard D5092, "Practices for Design and Installation of Ground Water Monitoring Wells in Aquifers", Annual Book of ASTM Standard, Section 4, Volume 04.08.

4. New York State Department of Environmental Conservation (1988), Draft Generic Environmental Impact Statement on the Oil, Gas, and Solution Mining Regulatory Program, Division of Mineral Resources.

5. Environmental Protection Agency (1986), RCRA Ground-Water Monitoring Technical Enforcement Guidance Document, OSWER-9950.1.

6. Environmental Protection Agency (1987), A Compendium of Superfund Field Operations Methods, EPA/540/P-87/001.

7. Environmental Protection Agency (1988), Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA, Interim Final, EPA/540/G-89/004.

REMEDIATION SECTION	PROCEDURE # 3.6
WORLDWIDE FACILITIES GROUP	EFFECTIVE DATE:
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WELL DEVELOPMENT PROCEDURES

INTRODUCTION

This procedure is for the development of groundwater monitoring wells that have been installed in overburden, top of bedrock, or deep bedrock formations. Before a newly constructed well can be used for water-quality sampling, measuring water levels, or aquifer testing, it must be developed. Well development refers to the procedure used to clear the well and formation around the screen of fine-grained materials (sands, silts, and clays) produced during drilling or naturally occurring in the formation.

Well development is completed to remove fine grained materials from the well but in such a manner as to not introduce fines from the formation into the sand pack. Well development continues until the well responds to water level changes in the formation (i.e., a good hydraulic connection is established between the well and formation) and the well produces clear, sediment-free water to the extent practical. Low-flow development is necessary if the contaminants of concern are sensitive to turbidity (e.g., metals, PCBs, SVOCs).

PROCEDURES REFERENCED

- Overburden Wells SOP 3.2
- Bedrock Wells SOP 3.3
- Waste Characterization SOP 12.0

WELL DEVELOPMENT PROCEDURES

The well development procedures presented below are the recommended standards. However, due to variations in conditions, changes in these well development standards may be necessary in order to facilitate the success completion of developing the monitoring well.

Well development can be accomplished by using in place pumps or using manual equipment; either peristaltic, bladder or other appropriate pumps depending on well depth. By doing so this helps in breaking down the filter cake that may have built up on the well screen.

- Don appropriate safety equipment.

- All non-dedicated equipment used for development purposes entering each monitoring well will be cleaned using a soapy wash (laboratory grade), tap water rinse, isopropyl alcohol rinse (with the exception of submersible pumps), and distilled/deionized water rinse.

- Uncap well and allow water level to stabilize. Attach appropriate pump and lower tubing into well.

- Turn on pump. If well runs dry, shut off pump and allow to recover.

- Collect the groundwater sample in a glass jar to determine relative turbidity, and measure and record the temperature, pH, turbidity and specific electrical conductance.

- The above steps will be repeated until groundwater is relatively silt-free; no further change is noted; the temperature, pH, turbidity and specific conductance readings have stabilized to within 10%. Final turbidity values should be less than 4 NTUs.

- After development, the well will not be sampled for at least two weeks.

WASTE DISPOSAL

- All waste generated will be disposed in accordance to the methods and procedures contained in the Waste Disposal SOP.

- All water generated during cleaning and development procedures will be collected and contained in accordance to the site specific disposal requirements.

- Personal protective equipment, such as gloves, disposable clothing, and other disposable equipment, resulting from personnel cleaning procedures and from soil sampling and handling activities, will be placed in plastic bags. These bags will be transferred into appropriately labeled 55-gallon drums or a covered roll-off box for appropriate disposal.

EQUIPMENT

- Appropriate health and safety equipment
- Knife
- Power source (generator)
- Field book or logs
- Well keys
- Graduated pails
- Pump and tubing
- Cleaning supplies (including non-phosphate soap, buckets, brushes, laboratory-supplied distilled/deionized water, tap water, cleaning solvent, aluminum foil, plastic sheeting, etc.)
- Water level meter
- pH/temperature/conductivity meter
- Clear glass jars (e.g., drillers' jars)

REFERENCE

1. Environmental Protection Agency (1986), RCRA Ground-Water Monitoring Technical Enforcement Guidance Document, OSWER-9950.1.

2. Environmental Protection Agency (1987), A Compendium of Superfund Field Operations Methods, EPA/540/P-87/001.

3. Environmental Protection Agency (1988), Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA, Interim Final, EPA/540/G-89/004.

REMEDIATION SECTION	PROCEDURE # 3.7
WORLDWIDE FACILITIES GROUP	EFFECTIVE DATE:
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WELL DECOMMISSIONING PROCEDURES

INTRODUCTION

This procedure is for the decommissioning/abandonment of groundwater monitoring wells that have been installed in overburden, top of bedrock, or deep bedrock formations. Well decommissioning refers to the procedure used to properly abandon or remove the monitoring well from the formation while taking the proper precautions to help eliminate cross-contamination.

The proper methods for properly abandoning monitoring wells are either by leaving the well materials in place and pressure grouting with a cement/bentonite slurry directly into the well or by over-drilling with augers, removing the well material and back filling with a cement bentonite slurry.

PROCEDURES REFERENCED

- Overburden Wells SOP 3.2
- Bedrock Wells SOP 3.3
- Decontamination SOP 9.0
- Waste Disposal SOP 12.0

WELL DECOMMISSIONING PROCEDURES

1. Pressure Grouting (if removal of the well casing is not possible)

- The borehole log from the monitoring well needs to be obtained to determine the well construction in order to prepare the proper materials and calculate the quantity of cement/bentonite slurry that will be required.

- The cement pad and the well protector around the monitoring pad needs to be removed and the immediate area around the monitoring well dug out. The riser pipe is to be cut off approximately one to two-feet below ground surface.

- A tremie pipe will be placed into the well completely to the bottom. A cement/bentonite slurry will then be pressure grouted in to the monitoring well backfilling completely to the surface. The grout will be prepared in the ratio of one bag (94 pounds) of Type I or Type II Portland Cement to 3 to 5 pounds of bentonite powder mixed with approximately 7 gallons of potable water. The grout will be allowed to sit for approximately one hour to allow any settlement of the cement/bentonite slurry and then augment if needed.

2. Overdrilling

- Based on the diameter of the monitoring well, this information can be obtained from the well completion diagram, the proper sized augers need to be specified.

- The cement pad and the well protector around the monitoring pad needs to be removed and the immediate area around the monitoring well dug out. The riser pipe is to be cut off approximately one to two-feet below ground surface.

- The augers are then placed over the riser pipe of the monitoring well and then drilling commences. The drilling continues until the final depth to which the monitoring well was installed is reached. The well materials are then removed (pulled) from the augers.

- A Cement/bentonite grout will be placed from the bottom of the borehole to the top of the augers. As each flight of augers is removed from the ground, the cement/bentonite grout will continue to be placed in the augers, to the top. This will continue until all the augers have been removed from the borehole. The grout will be prepared in the ratio of one bag (94 pounds) of Type I or Type II Portland Cement to 3 to 5 pounds of bentonite powder mixed with approximately 7 gallons of potable water.

- The area final restoration will be completed in accordance with the directions of the GM Facility representative (e.g. asphalt, concrete, vegetation). In active work areas final restoration maybe necessary immediately; or is permissible, time to allow settlement of the abandoned well area may be permitted prior to final restoration performed.

- Documentation/Notification requirements include modification of the well log to reflect closure and if necessary notification to the appropriate regulatory agency.

WASTE DISPOSAL

- All waste generated will be disposed in accordance to the methods and procedures contained in the Work Plan.

- All material generated during well decommissioning procedures will be collected and contained on site in roll-off boxes or 55-gallon drums for future analysis and appropriate disposal.

- Personal protective equipment, such as gloves, disposable clothing, and other disposable equipment, resulting from personnel cleaning procedures and from well closure activities, will be placed in plastic bags. These bags will be handled in accordance with the work plan.

EQUIPMENT

- Drilling Equipment

- Well Supplies

- Subsurface Boring Log

- Tape Measure

REFERENCE

1. Michigan Department of Public Health, Ground Water Quality Control Section – Division Of Water Supply (1988), Michigan Water Well Grouting Manual, MDPH GW-3-302
2. ASTM D5229 " Guide for Decommissioning of Ground Water Wells, Vadose Zone Monitoring Devices, Boreholes and other Devices For Environmental Activities.

REMEDIATION SECTION	PROCEDURE # **5.1**
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Manual Water Level Measurement Procedure

INTRODUCTION

This procedure describes measurement of water levels in groundwater monitoring and extraction wells, piezometers and boreholes. This procedure does not cover automated measurement of water levels with a transducers/datalogger, and does not cover measurement of phase-separated liquids.

Water levels in monitoring wells will be measured prior to each sampling event and at other times as indicated in the project Work Plan. Water levels will be acquired in a manner that provide accurate data that can be used to calculate vertical and horizontal hydraulic gradients and other hydrogeologic parameters. Accuracy in obtaining the measurements is critical to insure the useability of the data.

PROCEDURES REFERENCED

- 8.0 Field Instruments – Usage and Calibration
- 6.4 Non-Aqueous Phase Liquid (NAPL) Monitoring and Sample Collection For Laboratory Aanalysis
- 9.0 Equipment Decontamination

PROCEDURE

In order to provide reliable data, water levels must be collected over as short a period of time as practical. Barometric pressure can affect groundwater levels and, therefore, observation of significant weather changes during the period of water level measurements must be noted. Tidal fluctuations, navigation controls on rivers, rainfall events and groundwater pumping can also affect groundwater level measurements. Personnel collecting water level data must note if any of these controls are in effect during the groundwater level collection period. Due to possible changes during the groundwater level collection period, it is imperative that the time of data collection at each station be accurately recorded.

In conjunction with groundwater level measurements, surface water (e.g., ponds, lakes, rivers, and lagoons) often are monitored as well. This information is very helpful (and can be critical) in understanding the hydrogeologic setting of the site and most importantly how contaminants may move beneath the site.

The depth to groundwater will be measured with an electronic depth-indicating probe. Prior to obtaining a measurement, a fixed reference point on the well casing shall be established for each well to be measured. Unless otherwise established, the reference point is typically established and marked on the north side of the well casing. Avoid using protective casings or flush-mounted roadboxes for reference, due to the greater potential for damage or settlement.

If provided for in the project Work Plan, the elevation of the reference point shall be obtained by accepted surveying methods, to the nearest 0.01 ft.

The water level probe will be lowered into the well until the meter indicates (via indicator light or tone) the water is reached. The probe will be raised above water level and slowly lowered again until water is indicated. The cable will be held against the side of the inner protective casing at the point designated for water level measurements and a depth reading taken. This procedure will be followed three times or until a consistent value is obtained. The value will be recorded to the nearest 0.01 feet on the Groundwater Level Monitoring Report form (Form SOP 5.1-01) or other designated data recording location if specified in the project Work Plan.

Upon completion, the probe will be raised to the surface and together with the amount of cable that entered the well casing, will be decontaminated in accordance with methods described in **Procedure 9.0, Equipment Decontamination.**

EQUIPMENT

- Battery-operated, non-stretch electronic water level probe with permanent markings at 0.01 ft. increments (traceable to national measurement standards), such as the Solinst Model 101 or equivalent.

- The calibrated cable on the depth indicator will be checked against a surveyor's steel tape once per quarter year. A new cable will be installed if the cable has changed by more than 0.01% (0.01 feet for a 100 foot cable). See also Procedure **8.0, Field Instruments – Usage and Calibration.**

REFERENCE

- ASTM 4750 Test Method for Determining Subsurface Liquid Levels in a Borehole or Monitoring Well (Observation Well)

- ASTM D6000 Guide for Presentation of Water-Level Information from Ground-Water Sites

REMEDIATION SECTION	PROCEDURE # SOP 6.1
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SOIL SAMPLE COLLECTION

INTRODUCTION

Prior to sampling a Site, the purpose and objectives of the sampling program should be clearly stated and should include background information on site history. Numerous factors such as the size of the area, mode of contamination, the physical/chemical properties of the contaminant, and soil type need to be considered. For Site clean-ups, the purpose of the sampling is to determine the concentrations of contaminants at representative locations across the Site. Adequate planning must occur to assure that samples are representative to fulfill the purposes of the sampling.

PROCEDURES REFERENCED

- Soil Borings (SOP 2.3)
- Drilling Techniques (SOP 2.2)
- Soil Classification (SOP 2.6)
- Equipment Decontamination (Section 9.0)

SAMPLE COLLECTION

Sample Collection

The boring is advanced incrementally to permit intermittent or continuous sampling. Test intervals and locations are normally stipulated by the project engineer or geologist. Typically, samples are taken continuously.

For environmental sampling, always change gloves between collecting subsequent soil samples to prevent cross-contamination. All tools (e.g., samplers, spatulas, etc.) must be field cleaned prior to use on each sample.

Any drilling procedure that provides a suitably clean and stable hole before insertion of the sampler and assures that the penetration test or other sampling technique is performed on essentially undisturbed soil is acceptable. The drilling method is to be selected based on

the subsurface conditions. Each of the following procedures have proven to be acceptable for specific subsurface conditions:

- open-hole rotary drilling method;
- continuous flight hollow-stem auger method (with inside diameter between 2.2 and 6.5 inches);
- wash boring method; and
- continuous flight solid auger method (with auger diameter between 2.2 and 6.5 inches).

Several drilling methods are not acceptable. These include: jetting through an open tube sampler and then sampling when the desired depth is reached; use of continuous flight solid auger equipment below the groundwater table in non-cohesive soils; casing driven below the sampling depth prior to sampling; and advancing a borehole with bottom discharge bits. It is not permissible to advance the boring for subsequent insertion of the sampler solely by means of previous sampling when performing standard penetration testing.

The following subsections describe the specific protocol for Standard Penetration Test sampling and Shelby Tube sampling.

Standard Penetration Testing (SPT) Sampling and Testing Procedure

This method is used to obtain representative samples of subsurface soil materials and to determine a measure of the in situ relative density of the subsurface soils. The test methods described below must be followed to obtain accurate SPT values.

SPT sampling is performed by using a split barrel sampler in accordance with ASTM D1586. The split barrel sampler, or split spoon, consists of an 18- or 24-inch long, 2-inch outside diameter tube, which comes apart length wise into two halves.

Once the borehole is advanced to the target depth and the borehole cleaned of cuttings, representative soil samples are collected in the following manner:

- the split-spoon sampler should be inspected to ensure it is properly cleaned and decontaminated. The driving shoe (tip) should be relatively sharp and free of severe dents and distortions;
- the cleaned split-spoon sampler is attached to the drill rods and lowered into the borehole. Do not allow the sampler to drop onto the soil;
- after the sampler has been lowered to the bottom of the hole, it is given a single blow to seat it and make sure that it is in undisturbed soil. If there still appear to be excessive cuttings in the bottom of the borehole, remove the sampler from the borehole and remove the cuttings; and

- mark the drill rods in three or four successive 6-inch (0.15 m) increments, depending on sampler length, so that the advance of the sampler under the impact of the hammer can be easily observed for each 6-inch (0.15 m) increment.

The sampler is then driven continuously for either 18 or 24 inches (0.45 or 0.60 m) by use of a 140-pound (63.5 kg) hammer. The hammer may be lifted and dropped by either the cathead and rope method, or by using a trip, automatic, or semi-automatic drop system. The hammer should free-fall a distance of 30 inches (±1 inches) (760 mm, ±25 mm) per blow. Measure the drop at least daily to ensure that the drop is correct. To ensure a free-falling hammer, no more than 2 1/4 turns of the rope may be wound around the cathead (see ASTM D1586). The number of blows applied in each 6-inch (0.15 m) increment is counted until one of the following occurs:

- a total of 50 blows have been applied during any one of the 6-inch (0.15 m) increments described above;
- a total of 100 blows have been applied;
- there is no advancement of the sampler during the application of ten successive blows of the hammer (i.e., the spoon is "bouncing" on a stone or bedrock); or
- the sampler has advanced the complete 18 or 24 inches (0.45 or 0.60 m) without the limiting blow counts occurring as described above.

In some cases where the limiting number of blow counts has been exceeded, CRA may direct the driller to attempt to drive the sampler more if collection of a greater sample length is essential.

On the field form, record the number of blows required to drive each 6-inch (0.15 m) increment of penetration. The first 6 inches is considered to be a seating drive. The sum of the number of blows required for the second and third 6 inches (0.15 m) of penetration is termed the "standard penetration resistance" or the "N-value".

Note: If the borehole has sloughed and there is caved material in the bottom, the split spoon may push through this under its own weight, but now the spoon is partially "pre-filled". When the spoon is driven the 18 or 24 inches representing its supposedly empty length, the spoon fills completely before the end of the drive interval. Two problems arise:

1. the top part of the sample is not representative of the in-place soil at that depth; and

2. the SPT value will be artificially higher toward the bottom of the drive interval since the spoon was packed full. These conditions should be noted on the field log.

The sampler is then removed from the borehole and unthreaded from the drill rods. The open shoe (cutting end) and head of the sampler are partially unthreaded by the drill crew and the sampler is transferred to the geologist/engineer work surface.

Note: A table made out of two sawhorses and a piece of plywood is appropriate, or a drum, both covered with plastic sheeting.

The open shoe and head are removed by hand, and the sampler is tapped so that the tube separates.

Note: Handle each split spoon with clean disposable gloves if environmental issues are being investigated.

Measure and record the length of sample recovered making sure to discount any sloughed material that is present on top of the sample core.

Caution must be used when conducting SPT sampling below the groundwater table, particularly in sand or silt soils. These soils tend to heave or "blow back" up the borehole due to the difference in hydraulic pressures between the inside of the HSA and the undisturbed soil. To equalize the hydraulic pressure, the inside of the HSA must be filled with water or drilling mud. The drilling fluid level within the boring or hollow-stem augers needs to be maintained at or above the in situ groundwater level at all times during drilling, removal of drill rods, and sampling. Since heave or blow back is not always obvious to the driller, it is essential that the water level in the borehole always be maintained at or above the groundwater level.

Heaving conditions and the use of water or mud should be noted on the field logs.

SPT sampling below the water table in sands and silt occasionally results in low SPT values being obtained due to the heaving effect disturbing the soil especially if the water level in the hole has not been maintained at the in situ water level. Suspect low N values should be noted on the field logs. If it is critical to have accurate N values below the water table, other methods can be employed, such as conducting a dynamic cone penetration test. This quick and easy test involves attaching a cone shaped tip to the end of the drill rods, and driving the tip into the ground similar to the SPT method, except that the borehole is not pre-augered. Cones may be driven 20 to 40 feet through a formation without augering. Blow counts are recorded for each foot (0.3 m) of advancement.

A variation of split barrel sampling involves the use of a longer barrel in conjunction with hollow stem augers. The sampling barrel is installed inside the auger with a swivel attachment to limit rotation of the barrel. After completion of a 5-foot auger penetration, the auger is left in place and the barrel retrieved from the borehole. The sampler should be handled and the sample retrieved in the same way as described above for SPT sampling.

Thin-Walled Samplers (Shelby Tubes)

Thin-walled samplers are used to collect relatively undisturbed samples (as compared to split-spoon samples) of soft to stiff clayey soils. Shelby tubes are commonly used. The Shelby Tube has an outside diameter of 2 or 3 inches and is 3 feet long. These undisturbed

samples are used for certain laboratory tests of structural properties (consolidation, hydraulic conductivity, shear strength) or other tests that might be influenced by sample disturbance. Procedures for conducting thin-walled tube sampling are provided in ASTM D1587, and are briefly described below.

- the soil deposit being sampled must be cohesive in nature, and relatively free of sand, gravel, and cobble materials, as contact with these materials will damage the sampler;
- clean out the borehole to the sampling elevation using whatever method is preferred that will ensure the material to be sampled is not disturbed. If groundwater is encountered, maintain the liquid level in the borehole at or above groundwater level during the sampling operation;
- bottom discharge bits are not permitted. Side discharge bits may be used, with caution. Jetting through an open-tube sampler to clean out the borehole to sampling elevation is not permitted. Remove loose material from the center of a casing or hollow-stem auger as carefully as possible to avoid disturbance of the material to be sampled;
- place the sample tube so that its bottom rests on the bottom of the hole. Advance the sampler into the formation without rotation by a continuous and relatively rapid motion; usually hydraulic pressure is applied to the top of the drill rods;
- determine the length of advance by the resistance and condition of the formation, but the length shall never exceed 5 to 10 diameters of the tube in sands and 10 to 15 diameters of the tube in clays;
- in no case should the length of advance be greater than the sample-tube length minus an allowance for the sampler head and a minimum of 3 inches for cuttings.
- the tube may be rotated to shear the bottom of the sample 2 to 3 minutes after pressing in, and prior to retrieval to ensure the sample does not slide out of the tube. Lift the weight of the rods off of the tube prior to rotating.
- withdraw the sampler from the formation as carefully as possible in order to minimize disturbance of the sample;
- package and transport the sample in accordance with Paragraph ix).

On occasion it may be required that extraction of the sample from the tube be conducted in the field. The following procedure should be followed.

- a sample extruder, which consists of a clamp arrangement to hold the tube and a hydraulic ram to push the sample through the tube, is usually mounted on the side of the rig. To prevent cross-contamination, be certain that the extruder is field cleaned between each sample;
- the sample is then extruded into a carrying tray; these are often made from a piece of 4-inch or 6-inch diameter PVC pipe cut lengthwise. Be certain that the carrying tray is field cleaned between each sample. The sample is carried to the work station to describe the sample, trim the potentially cross contaminated exterior, and place it in the appropriate container. and
- the Shelby tube may then be thoroughly field cleaned and decontaminated for reuse. Since they are thin-walled, the tubes are easily damaged, crimped, or otherwise

distorted during handling or pushing. The Shelby Tube should be inspected before use and any which are significantly damaged should be rejected.

Geoprobe Macro-Core Soil Sampler

The operation of the Macro-Core Soil Sampler consists of "pushing" the sampler into the subsurface and then retrieved using a Geoprobe soil probing machine. The collected soil core is removed from the sampler along with the used liner. Once the liner has been removed and the sampler deconned a new liner is inserted and the sampler reassembled. The clean sampler is then advanced back down the same hole to collect the next soil sample. The Macro-Core sampler can be used in either the open-tube or closed-point sampling mode. The open-core is most commonly used. It is used in stable soils. In unstable soils, the piston rod point system prevents collapsed soil from entering the sampler as it is advanced back down the hole. Once at the sample depth, the piston rod is unthreaded and removed The sampler is then driven into the subsurface to fill the sampler with soil.

FIELD NOTES

- Samplers should be diligent in taking field notes during the sampling program. The boring logs shall note the depth of sample, the length of the core and the depth of any features of the soil such as changes in physical properties, color changes, the presence of roots, etc. If chemical odors are noted or unusual color patterns detected, theses should also be noted. Intentionally smelling the sample is not an acceptable practice because of safety reasons. Care should be made to make note of anything that could prove to be important in interpreting the results obtained. Some pollutants leave a staining pattern, it should be recorded as well. See SOP 1.4 and SOP 2.6 for further details.

DECONTAMINATION

- Decontamination of large equipment is difficult, but not impossible. Drillers do not appreciate the detail required to clean up their drill rigs, and do not like having to use dry coupling instead of grease. Precleaned split-spoons or the use of liners can help in preventing contamination of the sample. The augers can be cleaned with high pressure steam cleaners. See SOP 9.0 Equipment Decontamination for further details.

EQUIPMENT

- Drilling Equipment
- Subsurface Boring Log

- Tape Measure
- Water Level Probe

REFERENCE

1. American Society for Testing and Materials (1991), Standard D1452-80, "Practice for Soil Investigation and Sampling by Auger Borings", Annual Book of ASTM Standard, Section 4, Volume 04.08.

2. Environmental Protection Agency (1986), RCRA Ground-Water Monitoring Technical Enforcement Guidance Document, OSWER-9950.1.
3. Environmental Protection Agency (1987), A Compendium of Superfund Field Operations Methods, EPA/540/P-87/001.
4. ASTM D1452-80 Practice for Soil Investigation and Sampling by Auger Borings.
5. ASTM D1586-84 Test Method for Penetration Test and Split-Barrel Sampling of Soils
6. ASTM D1587-94 Practice for Thin Walled Tube Geotechnical Sampling of Soils
7. ASTM D2488-93 Practice for Description and Identification of Soils (Visual-Manual Procedure)
8. ASTM D4700-91 Guide for Soil Sampling from the Vadose Zone

REMEDIATION SECTION	PROCEDURE # SOP - 6.2
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SURFACE WATER SAMPLING FOR LABORATORY ANALYSIS

INTRODUCTION

Surface water sampling locations for water quality studies may be selected based on many factors, including: study objectives; the location of point source discharges, non-point source discharges and tributaries; the presence of structures (bridges, dams, etc.), and accessibility.

Before any sampling is conducted, the first requirement is to consider suitable sampling locations. Bridges and piers are normally good choices for surface water sampling since they provide access and permit water sampling at any point across the width of the water body. Sampling locations should be selected in accordance with the Work Plan and discussed with the Project Manager.

Wading for water samples in lakes, ponds, and slow-moving rivers and streams must be done with caution since bottom deposits are easily disturbed. Samples must be collected without entrained suspended sediments. All surface water samples are to be collected commencing with the most downstream sample to avoid sediment interference with other samples. A life vest and safety line will be worn in all cases where footing is unstable or where water is fast moving or over 3 feet (0.85 m) in depth. A second person may also be required for most of the sampling scenarios.

Prior to entering select areas it may be necessary to acquire property access permission from the land owner. Access permission must be acquired in advance of the sampling program and may require a written agreement.

PROCEDURES REFERENCED

- Equipment Decontamination (SOP-9.0)

SAMPLING RATIONALE/APPROACH

i) Rivers, Streams, and Creeks

Surface water samples should usually be collected in areas of the surface water body that are representative of the surface water body conditions. Representative samples can usually be collected in portions of the surface water body that have a uniform cross section and flow rate. Since mixing is influenced by turbulence and water velocity, the selection of a site immediately downstream of a riffle area (e.g., fast flow zone) will ensure good vertical mixing. These locations are also likely areas for deposition of sediment since the greatest deposition occurs where stream velocity slows.

A site that is clear of immediate point sources (e.g., tributaries and industrial and municipal effluents) is preferred for the collection of surface water samples unless the sampling is being performed to assess these sources.

Tributaries should be sampled as near the mouth as is feasible. However, it is important to select the sample location taking into consideration the impact that the downstream receiving water body has on the tributary flow and sediments. The downstream water body may change the water quality (salinity), temperature, or turbidity in the tributary near its mouth.

Sediment samples shall be collected along a cross-section of a river or stream in order to adequately characterize the bed material or as described in the Work Plan. A common procedure is to sample at quarter points along the cross-section of the sampling site selected. Samples may be composited as described in the Work Plan. Samples of dissimilar composition should not be combined.

In some instances sediment sampling may be performed along the shore only; depending upon the study needs.

ii) Lakes, Ponds, and Impoundments

The water in lakes, ponds, and impoundments has a much greater tendency to stratify than water in rivers and streams. The lack of mixing may require that more samples be obtained. An extreme turbidity difference may occur where a highly turbid river enters a lake. Therefore, each layer of the stratified water column may need to be considered separately. Stratification is caused by water temperature differences; the cooler, heavier water is beneath the warmer water.

Sample selection also should adequately represent the conditions of the lagoon or settling pond. Attention must be given to identify intakes and outflows within the

lagoon or settling pond which may provide biased sample representation. Sample locations with adjacent structures (i.e., banks, piers, etc.) may also provide biased samples within active lagoons or settling ponds, as the potential for boundary flow and eddies exist.

The number of water sampling sites on a lake, pond, or impoundment will vary with the purpose of the investigation, as well as the size and shape of the basin. In ponds and small impoundments, a single sample should be collected at the deepest point. In naturally formed ponds, the deepest point is usually near the center. In impoundments the deepest point is usually near the dam.

In lakes and larger impoundments, several subsamples may be composited to form a single sample. These vertical sampling locations are often taken along a grid.

In lakes with irregular shape, with several bays and coves that are protected from the wind, additional samples may be needed to represent water quality at various points in the lake. Additional samples may be taken where discharges, tributaries, and other such factors are suspected of influencing water quality.

When collecting sediment samples in lakes, ponds, and reservoirs, samples should be collected at approximately the center of the water body or as directed by the Work Plan. This is also the case for reservoirs that are formed by the impoundment of rivers or streams. The coarse grained sediments are deposited near the headwaters of the reservoir, and the fine grained sediments near the center. The shape, inflow pattern, and circulation must be considered when selecting sediment sampling sites in lakes and reservoirs.

In all instances, the sampling locations should be properly documented with field notes and photographs, as appropriate.

SAMPLING TECHNIQUES

Any equipment or sampling technique(s) used to collect a sample is acceptable as long as it provides a sample which is representative of the stream being sampled and is consistent with the Work Plan. Typically sample aliquots are collected from the area of concern directly, or a compositing approach is considered using a plastic bucket to collect a representative sample, then individual aliquots are collected from the sample bucket.

When collecting surface water samples, direct dipping of the sample container into the stream is acceptable unless the sample bottles contain preservatives. If the bottles are preserved, then precleaned unpreserved bottles should be used to collect the sample. The water sample should then be transferred to the appropriate preserved bottles. When collecting samples, submerse the inverted bottle to the desired sample depth and then tilt

the opening of the bottle upstream to fill. When composting across a stream and/or water channel is typically performed using a pre-rinsed 1 to 2 L plastic bottle collecting sub-samples for final mixing sample aliquot collection. VOC's must not be collected from the compositing bucket and are sampled directly from the stream cross section.

Wading may cause bottom sediment deposits to be re-suspended and therefore could result in a biased sample. Wading is acceptable if the stream has a noticeable current and the samples are collected directly into the bottle while pointed upstream. If the stream is too deep to wade or if the sample must be collected from more than one water depth, additional sampling equipment will be required. Samples should be collected approximately 6 inches (15 cm) below the surface with the sample bottles completely submerged. This will keep floating debris from entering the sample bottles. Floating debris could result in unrepresentative analytical data.

Sample collection when the flow depth is minimal (i.e., <1 inch (<2.5 cm)) will require special consideration to prevent sediment disturbance. Sampling might be conducted with a container then transferred to the appropriate glassware, or collection may be permissible with a peristaltic pump using a 'fixed' suction line, secured to prevent sediment collection. A small excavation in the stream bed to create a 'sump' for sample collection may be permissible but should be prepared well in advance of the sample collection event to allow sediment settlement.

Teflon bailers may be used for surface water sampling if it is not necessary to collect a sample at a specified interval. A top-loading bailer with a bottom check-valve is sufficient for many studies. As the bailer is lowered through the water, water is continually displaced through the bailer until a desired depth is reached, at which point the bailer is removed. This technique is not suitable where strong currents are encountered (because the ball may not seat effectively), or where a discrete sample at a specific depth is required.

If discrete samples are required from a specific depth, and the parameters to be measured do not require a Teflon-coated sampler, a standard Kemmerer, or Van Dorn sampler may be used. The Kemmerer sampler is a brass cylinder with rubber stoppers that leave the ends of the sampler open while being lowered in a vertical position to allow for passage of water through the cylinder. The Van Dorn sampler is plastic and is lowered in a horizontal position. In each case, a messenger is sent down a rope when the sampler is at the required depth to cause the stoppers to close the cylinder. The sampler is then raised to the surface. Water is removed through a valve to fill respective sample bottles. Dissolved oxygen (DO) sample bottles can be properly filled by allowing overflow using a rubber tube attached to the valve. When performing multiple depth sampling, care should be taken not to stir up the bottom sediment.

A glass beaker or stainless steel scoop may be used to collect samples if the parameters to be analyzed are not interfered with. The beaker or scoop should be rinsed three times with

the sample water prior to collection of the sample. All field equipment should follow standard cleaning procedures.

EQUIPMENT/MATERIALS

- Sampling device (Plastic bucket, pump, depth integrated sampler (D15))
- Flow measurement device (velocity meter, survey equipment, measuring tape)
- Sampling materials (sample containers, log book, cooler, chain-of-custody)
- Camera
- Work Plan
- Health and Safety Plan

REFERENCE

1. ASTM D4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents
2. ASTM D4581 Guide for Measurement of Morphologic Characteristics of Surface Water Bodies
3. ASTM D5906 Guide for Measuring Horizontal Positioning During Measurements of Surface Water Depths
4. ASTM D5073 Practice for Depth Measurement of Surface Water
5. ASTM D5413 Test Methods for Measurement of Water Levels in Open-Water Bodies
6. Greenberg, A.E., R.R. Trussell, and C.S. Clesceri(eds). 1985. Standard methods for the examination of water and wastewater. 16th Edition. American Public Health Association, Washington, DC. p.37.

REMEDIATION SECTION	PROCEDURE #: SOP-6.3
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GROUNDWATER SAMPLE COLLECTION FOR LABORATORY ANALYSIS

INTRODUCTION

This procedure is for the collection of groundwater samples for laboratory analysis.

The following describes two techniques for groundwater sampling: "Low Stress/Low Flow Methods" and "Typical Sample Methods."

"Low Stress/Low Flow" methods will be employed when it is critical to collect groundwater samples truly representative of the groundwater present, not impacted by sediment/colloid presence. Analysis typically sensitive to turbidity/sediment issues are Polychlorinated-biphenyls (PCBs), Semi-volatile compounds (SVOCs) and metals.

The "Typical Sample Methods" will be employed where the collection of parameters less sensitive to turbidity/sediment issues are being collected (volatile organics - VOCs, and general chemisty).

NOTE: If Non-aqueous phase liquids (NAPL) (light or dense) are detected in a monitoring well, groundwater sample collection will not be conducted and the Project Manager must be contacted to determine a course of action.

PROCEDURES REFERENCED

- Water Level Measurements (SOP-5.1)
- Equipment Decontamination (SOP-9.0)

PREPARATORY REQUIREMENTS

- Verify well identification and location using borehole log details and location layout figures. Note the condition of the well and inform the Project Manager of any necessary repair work required.
- Prior to opening the well cap, measure the breathing space above the well casing with a PID to establish baseline levels. Repeat this measurement once the well cap is opened. If either

of these measurements exceeds the air quality criteria in the health and safety plan, field personnel should adjust their PPE accordingly.

- Prior to commencing the groundwater purging/sampling tasks, a water level must be obtained to determine the well volume for hydraulic purposes. Refer to the Water Level Measurements Procedure (SOP 5.1)for details. In some settings it maybe necessary to allow the water level time to equilibrate. This condition exists if a water tight seal exists at the well cap and the water level has fluctuated above the top of screen; creating a vacuum or pressurized area in this air space. Three water level checks will verify static water level conditions or changing conditions.
- Calculate the water volume in the well. Typically overburden well volumes consider only the quantity of water standing in the well screen and riser; bedrock well volumes are calculated on the quantity of water within the open corehole and within the overburden casing.
- Estimate natural groundwater flow rate into well to determine the approximate pumping rate for purging/sampling activities.

WELL PURGING AND STABILIZATION MONITORING (LOW STRESS/LOW FLOW METHOD)

- The GM method of preference for groundwater sampling will be the low stress/low flow method described below.
- Bladder pumps/submersible variable rate pumps (i.e., Grundfos™ Rediflo or equivalent) are typically employed.
- Slowly lower the pump, safety cable, tubing and electrical lines into the well to the depth specified by the project requirements. The pump should be placed in the well approximately 24 hours prior to purging. The pump intake must be at the mid-point of the well to prevent disturbance and resuspension of any sediment in the screen base.
- Before starting the pump, measure the water level again with the pump in the well leaving the water level measuring device in the well when completed.
- Purge the well at 100 to a maximum of 500 milliliters per minute (mL/min). During purging, the water level should be monitored approximately every 5 minutes, or as appropriate. A steady flow rate should be maintained that results in drawdown of 0.3 feet or less. The rate of pumping should not exceed the natural flow rate conditions of the well being sampled. Care should be taken to maintain pump suction and to avoid entrainment of air in the tubing. Record adjustments made to the pumping rates and water levels immediately after each adjustment.
- During the purging of the well, monitor and record the field indicator parameters (pH, temperature, conductivity, oxidation-reduction (redox) reaction potential (ORP), dissolved oxygen (DO), and turbidity) approximately every five minutes. Stabilization is considered to be achieved when the final groundwater flow rate is achieved, and three consecutive readings for each parameters are within the following limits:
  - pH ±0.1 pH units of the average value of the three readings;
  - temperature ±3 percent of the average value of the three readings;

- conductivity ±0.005 milliSiemen per centimeter (mS/cm) of the average value of the three readings for conductivity <1 mS/cm and ±0.01 mS/cm of the average value of the three readings for conductivity >1 mS/cm;
- ORP ±10 millivolts (mV) of the average value of the three readings;
- DO ±10 percent of the average value of the three readings; and
- turbidity ±10 percent of the average value of the three readings, or a final value of less than 5 nephelometric turbidity units (NTU).

- Should stabilization not be achieved for all field parameters, purging is continued until a maximum of 20 well screen volumes have been purged from the well. Since low-flow purging (LFP) likely will not draw groundwater from a significant distance above or below the pump intake, the screen volume is based upon a 5-foot (1.4 m) screen length. After purging 20 well screen volumes, purging is continued if the purge water remains visually turbid and appears to be clearing, or if stabilization parameters are varying slightly outside of the stabilization criteria listed above and appear to be approaching stabilization.
- If low-turbidity samples are critical to the project goals, purging will be extended until turbidity has been reduced to 5 NTU or less.
- The pump must not be removed from the well between purging and sampling.

WELL PURGING AND STABILIZATION MONITORING (TYPICAL METHOD)

- Typically peristaltic pumps or bladder pumps or submersible pumps are preferred. In most cases bailer use is not desirable.
- Pump placement is typically performed at the mid-point of the screen.
- Purge the well until three consecutive well volume measurements of temperature and specific conductivity are approximately plus or minus 10 percent and if the pH values are within 1 pH unit of the last three value averages, and the groundwater turbidity values are less than 5 NTU. If stabilization has not occurred within the first five well volumes removed, continue purging and monitoring until eight well volumes have been pumped.
- Groundwater turbidity may be evaluated by a visual examination for sediment/silt presence or use of a nephlometer. Work Plan Specific goals may exist for turbidity values which may require extending the purging, or require an alternate pumping system.
- Monitoring well purging is accomplished by using in-place pumps or by either a peristaltic, bladder or other appropriate pump, depending on the well depth. The pump/hose assembly or bailer used for purging should be lowered into the top of the standing water column and not deep into the column. Typically pump placement at the mid-point of the screen is adequate.

SAMPLING TECHNIQUES

- If an alternate pump is utilized, the first pump discharge volumes should be discarded to allow the equipment a period of acclimation to the groundwater.
- Samples are typically collected directly from the pump with the groundwater being discharged directly into the appropriate sample container. Avoid handling the interior of the bottle or bottle cap and don new gloves for each well sampled to avoid contamination of the sample.
- Order of sample collection:
  - Volatile organic compounds
  - Semi-volatile organic compounds
  - Total organic carbon (TOC)
  - Total organic halogens (TOX)
  - Extractable organics
  - Total metals
  - Dissolved metals
  - Phenols
  - Cyanide
  - Sulfate and chloride
  - Nitrate and ammonia
  - Radionuclides
- For low stress/low flow sampling, samples should be collected at a flow rate between 100 and 250 mL/min and such that drawdown of the water level within the well does not exceed the maximum allowable drawdown of 0.3 feet.
- The pumping rate used to collect a sample for VOCs should not exceed 100 mL/min. Samples should be transferred directly to the final container 40 mL glass vials completely full and topped with a teflon cap. Once capped the vial must be inverted and tapped to check for headspace/air presence (bubbles). If air is present the sample vial will be discarded, and re-collected until free of air.
- Field filtration will be performed if dictated by the project Work Plan.
- Sample labels/sample identification
- All samples must be labeled with:
  - A unique sample number
  - Date and time
  - Parameters to be analyzed
  - Project Reference ID
  - Samplers initials
- Labels should be secured to the bottle and should be written in indelible inks.

EQUIPMENT/MATERIALS

- pH meter, Conductivity meter, Dissolved Oxygen (DO) meter, Oxidation-reduction (redox) reaction potential (ORP) meter, Nephlometer, Temperature guage
- Field filtration units (if required)
- Purging/sampling equipment
  - Peristaltic Pump (not suitable for VOCs[1]/SVOCs or depths >25 feet);
  - Suction Pumps (not suitable for LFP, VOCs/SVOCs, or depths >25 feet);
  - Submersible Pumps (suitable for VOCs/SVOCs only at low flow rates);
  - Air Lift Pumps (not suitable for VOCs/SVOCs);
  - Bladder Pumps (suitable for LFR and VOCs/SVOCs);
  - Inertia Pumps (gaining acceptability for VOCs/SVOCs generally not suited for GM programs); and
  - Bailers (not suitable for LFP generally not suited for GM programs).
- Water Level Probe
- Sampling Materials (containers, log book/forms, coolers, chain-of-custody)
- Work Plan
- Health and Safety Plan

NOTE[1]: PERISTALTIC PUMP USE FOR VOC COLLECTION IS NOT ACCEPTABLE ON EPA/RCRA SITES; THIS TECHNIQUE HAS GAINED ACCEPTANCE IN SELECT AREAS WHERE IT IS PERMISSIBLE TO COLLECT VOCS USING A PERISTALTIC PUMP AT A LOW FLOW RATE (EX. MICHIGAN).

FIELD NOTES

- Field notes must document all the events, equipment used, and measurements collected during the sampling activities. SOP 1.5 describes the data/recording procedure for field activities. The log book should document the following for each well sampled:
  - Identification of well
  - Well depth
  - Static water level depth and measurement technique
  - Sounded well depth
  - Presence of immiscible layers and detection/collection method
  - Well yield – high or low
  - Purge volume and pumping rate
  - Time well purged
  - Measured field parameters

- Purge/sampling device used
- Well sampling sequence
- Sampling appearance
- Sample odors
- Sample volume
- Types of sample containers and sample identification
- Preservative(s) used
- Parameters requested for analysis
- Field analysis data and method(s)
- Sample distribution and transporter
- Laboratory shipped to
- Chain of custody number for shipment to laboratory
- Field observations on sampling event
- Name collector(s)
- Climatic conditions including air temperature
- Problems encountered and any deviations made from the established sampling protocol.

A standard log form for documentation and reporting groundwater purging and sampling events are presented Attachment 2 of this FSP.

GROUNDWATER/DECON FLUID DISPOSAL

- Groundwater disposal methods will vary on a case-by-case basis but may range from:
  - Off-site treatment at private treatment/disposal facilities or public owned treatment facilities
  - On-site treatment at Facility operated facilities
  - Direct discharge to the surrounding ground surface, allowing groundwater infiltration to the underlying subsurface regime
  - Direct discharge to impervious pavement surfaces, allowing evaporation to occur
- Decon fluids should be segregated and collected separately from wash waters/groundwater containers. Often small volumes of solvents used during the day can be allowed to evaporate if left in an open pail. In the event evaporation is not possible or practical, off-site disposal arrangements must be made.

REFERENCE

1. ASTM D5474 Guide for selection of Data Elements for Groundwater Investigations
2. ASTM D4696 Guide for pore-liquid sampling from the vadose zone
3. ASTM D5979 Guide for conceptualization and characterization of groundwater systems

4. ASTM D5903 Guide for planning and preparing for a groundwater sampling event
5. ASTM D4448 Standard guide for sampling groundwater wells
6. ASTM D6001 Standard guide for direct push water sampling for geo-environmental investigations.
7. USEPA Low-flow (minimal drawdown) ground-water sampling procedures (EPA/540/S-95/504)
8. USEPA RCRA Groundwater Monitoring: Draft Technical guidance (EPA/530-R-93-001)

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NON-AQUEOUS PHASE LIQUID (NAPL) MONITORING AND SAMPLE COLLECTION FOR LABORATORY ANALYSIS

INTRODUCTION

This procedure is for the monitoring the presence of dense and light non-aqueous phase liquids (DNAPL and LNAPL), and collection of NAPL samples for laboratory analysis in monitoring, observation, and extraction wells.

It should be noted that groundwater sampling and analysis should not be performed in locations where NAPL has been identified.

PROCEDURES REFERENCED

- Water Level Measurements (SOP - 5.1)
- Equipment Decontamination (SOP - 9.0)

NAPL LEVEL MEASUREMENTS

- Conduct well identification, inspection, and opening in accordance with the Water Level Measurement Procedures (SOP 5.1).
- NAPL level measurements are best conducted using a dual phase interface probe. The interface probe uses an optical liquid sensor, in conjunction with an electric circuit to detect the top of a phase-separated liquid and the interface between the phase layer and water (water level). The procedure for use of this probe is:
- For LNAPL:
  - Lower the probe tip into the center of the well until discontinuous beeping is heard (this indicates the top of the LNAPL has been detected). Grasp the calibrated tape at the reference point and note reading. Confirm the reading by slowly raising and lowering the probe to the level of the phase layer.
  - Once the top of the phase layer is confirmed, slowly lower the probe until a continuous sound is heard. This indicates that the water level has been encountered. Grasp the tape at the reference point and note the reading. Confirm this water level measurement.
  - Decontaminate the submerged end of the tape and probe prior to the next use in accordance with the Work Plan requirements.
- For DNAPL:
  - Lower the probe tip in the center of the well to the bottom of the well, a discontinuous beeping will be heard if DNAPL is present. Grasp the calibrated tape at the reference point and note reading.
  - Once the bottom of the well is confirmed, slowly raise the probe until a continuous sound is heard. This indicates that the water level has been encountered and represents the top

of the DNAPL layer. Grasp the tape at the reference point and note the reading. Confirm this water level measurement.
  - Decontaminate the submerged end of the tape and probe prior to the next use.
- Alternative NAPL measurement methods exist in the event an interface probe is unavailable or not functioning properly. These methods tend to be less accurate than the interface probe but may be used to establish an estimated NAPL measurement.
  - Clear Bailer – A clear bottom-loading bailer may be used to estimate NAPL thickness if floating or denser than water. If NAPL presence is suspected, the bailer is carefully lowered to the location of suspected NAPL presence (top of water column/base of water column), and slowly removed and examined for NAPL. If present, the column of NAPL within the clear bailer can be measured to estimate the NAPL thickness within the groundwater column.
  - Weighted Cord – Primarily used for DNAPL measurements, a weighted "cotton" string or cord may be lowered to the base of the well and inspected upon retrieval. Typically, the lower DNAPL layer will "coat" the string indicating the approximate thickness of this layer.

WELL NAPL SAMPLING

- Prior to sampling, the level of NAPL in the well should be measured as identified above.
- Various sampling devices can be employed to acquire fluid samples from the top and bottom of the well, including the following:
  - Bottom-loading bailer;
  - Double check value bailer (produces most reliable results);
  - Peristaltic pump for shallow wells (<25 feet in depth); or
  - Inertia pump for deeper wells (up to 300 feet in depth).
- Transfer NAPL to sample containers for shipment to laboratory. NAPL can be sampled to evaluate the physical properties of the fluid or to evaluate chemical composition.
- Decontaminate equipment prior to next use.

Note: Groundwater sampling shall not be performed in locations where NAPL is present.

EQUIPMENT/MATERIAL

- Interface Probe;
- Bottom-loading bailer;
- Double check valve bailer;
- Peristaltic pump;
- Inertia pump
- Work Plan
- Health and Safety Plan

REFERENCE

1. Cohen, Robert M., Mercer, James W. (GeoTrans, Inc.), Robert S. Kerr Environmental Research Laboratory "DNAPL Site Evaluation" Office Research and Development. U.S. Environmental Protection Agency
2. Cohen, R.M., Brayda, A.P., Shaw, S.T., and Spaulding, C.P.; Fall 1992 "Evaluation of Visual Methods to Detect NAPL in Soil and Water", Groundwater Monitoring Review, Vol. 12 No. 4, pp. 132-141.

REMEDIATION SECTION	PROCEDURE # SOP - 6.6
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SAMPLE HANDLING AND SHIPPING

INTRODUCTION

Sample management is the continuous care given to each sample from the point of collection to receipt at the analytical laboratory. Good sample management ensures that samples are properly recorded, properly labeled, not lost, broken, or exposed to conditions which may affect the sample's integrity.

All sample submissions must be accompanied with a chain-of-custody (COC) document to record sample collection and submission.

The following sections provide the minimum standards for sample management.

PROCEDURES REFERENCED

A) Field Handling

Prior to entering the field area where sampling is to be conducted, especially at sites with defined exclusion zones, the sampler should ensure that all materials necessary to complete the sampling are on hand.

If samples must be maintained at a specified temperature after collection, proper coolers and ice/cool-packs must be brought out to the field. Consideration should be given to keeping reserve cooling media on hand if sampling events will be of long duration. Conversely, when sampling in extremely cold weather, proper protection of water samples, trip blanks, and field blanks must be considered.

Personnel performing groundwater sampling tasks must check the sample preparation and preservation requirements to ensure compliance with the Work Plan QAPP. Typical sample preparation may involve pH adjustment (i.e., preservation), sample filtration and preservation, or simply cooling to 4°C. Sample preparation requirements vary from site to site and vary depending upon the analytical method for which the samples will be analyzed.

The sampling personnel must also confirm before the sample event, the amount of bottle filling required for the respective sample containers. VOC samples must not have any

headspace within the sample collection vial; whereas when collecting select analytes (i.e., metals) a headspace must be provided to allow addition of the required preservative.

B) Sample Labeling

Samples must be properly labeled as soon as practical after collection.

Note that the data shown on the sample label is the minimum data required. The sample label data requirements are listed below for clarity.

i) Project name.
ii) Sample number.
iii) Sampler's initials.
iv) Date of sample collection.
v) Time of sample collection.
vi) Analysis required.
vii) Preservatives.

The Work Plan Quality Assurance/Quality Control (QA/QC) specification should be reviewed to determine any additional requirements.

Quite often the analytical laboratory supplying the containers will provide blank sample labels. If these are adequate and convenient they can be used.

Under certain field conditions it is impractical to complete and attach sample labels to the container at the point of sample collection. However, to ensure that samples are not confused, a clear notation should be made on the container with a permanent marker indicating the last three digits of the sample number. If the containers are too soiled or small for marking, the container can be put into a "zip-lock" bag which can then be labeled.

No one sample number format is adequate for every type of sampling activity. Prior to the start of every project or sub-sampling event within the project, Project Managers and field personnel should devise a sample number format. Sample number formats should be as simple and short as possible. Simple number formats will reduce transcription errors by both Consultants and lab personnel. The sample number format should be comprehensive enough to allow for easy location of detailed sample data within the Site log books. Sample format must also be consistent with any future data management activities.

Unless otherwise instructed, labels should not contain specific names of the sample source (i.e., "Well No. 16"). Provision of such specific data on the label can produce biased lab results.

Sample Labels/Sample Identification

All samples must be labeled with:

- a unique sample number (never to be re-used, nor likely to be);
- date and time;
- parameters to be analyzed;
- job number; and
- sampler's initials.

Labels should be secured to the bottle and should be written in indelible inks. It is also desirable to place wide clear tape over the label before packing in a cooler for label protection during transportation.

The unique sample identification number may follow the format recommended below, or a specific sample protocol for labeling may be specified in the project Work Plan.

JOB NUMBER		DAY/MONTH/YEAR
	A 1886 WDG -07/11/94-AB	
SERIES	SAMPLER'S INITIALS	SAMPLE IDENTIFICATION DESIGNATION

This format has been selected to maximize the information content of the sample number. Minor modifications are certainly reasonable.

i) Series is a letter which designates a group of samples. This might include sample round, or might designate sample type (e.g., sediment, soil, volatile analysis, Round 2 Lower Aquifer wells), or sample source. For example, "A" might mean samples of influent to some treatment system, "B" might mean samples of effluent. Letters should be used, not numbers. Series is optional.

ii) Job number together with the series number, will allow easier tracking of samples.

iii) Sampler's initials will allow identification of the sampler, and so allow all project personnel to contact the correct person for information regarding that sample and its collection. The use of three initials is requested. Special arrangements will need to be made if two individuals have the same initials.

iv) Sample date will allow monitoring of actual holding time of samples and should ensure that all sample numbers are unique, even if sample location designation is used in a system, as opposed to assigned at random.

v) Sample identification designation will identify the sample, and can be any numerical or letter designation.

The decision of how to assign sample numbers should be made at the beginning of a job or phase, and should be consistent throughout the job.

C) Packaging

When possible, sample container preparation and packing for shipment should be completed in a well organized and clean area, free of any potential cross-contaminants.

Sample containers should be prepared for shipment as follows:

i) Containers should be wiped clean of all debris/water using paper towels (paper towels must be disposed of with other contaminated materials).
ii) Clear, wide packing tape should be placed over the sample label for protection.

While there is no one "best" way to pack samples for shipment, the following packing guidelines should be followed.

i) Plan time to pack your samples (and make delivery to shipper if applicable). Proper packing and manifesting takes time. A day's worth of sampling can be easily wasted due to a few minutes of neglect when packing the samples.
ii) Always opt for more coolers and more padding rather than crowd samples. The cost associated with the packing and shipment of additional coolers is usually always small in comparison with the cost of having to re-sample due to breakage during shipment.
iii) Do not bulk pack. Each sample must be individually padded.
iv) Large glass containers (1 liter and up) require much more space between containers.
v) Ice is not a packing material due to the reduction in volume when it melts.

The following is a list of standard guidelines which must be followed when packing samples for shipment.

i) When using ice for a cooling media, always double bag the ice in "Zip-Lock" bags.
ii) Double-check to ensure trip and temperature blanks have been included for all shipments containing VOCs, or where otherwise specified in the QA/QC plan.
iii) Enclose the Chain-of-Custody form in a "Zip-Lock" bag.
iv) Ensure custody seals (two, minimum) are placed on each cooler. Coolers with hinged lids should have both seals placed on the opening edge of the lid. Coolers with "free" lids should have seals placed on opposite diagonal corners of the lid. Place clear tape over custody seals.

v) Ensure that all "Hazardous Material" stickers/markings have been removed from coolers being used which previously contained such materials.

Note: Never store sterile sample containers in enclosures containing equipment which use any form of fuel or volatile petroleum based product. An alternate means of secure storage must be planned for. When conducting sampling in freezing conditions at sites without a heated storage area (free of potential cross contaminants), trip blanks and temperature blanks not being used in a QA/QC role should be isolated from coolers immediately after receipt. Trip and temperature blanks should be double-bagged and kept from freezing.

D) Chain-of-Custody Records

Chain-of-custody forms will be completed for all samples collected. The form documents the transfer of sample containers.

The chain-of-custody record, completed at the time of sampling, will contain, but not be limited to, the sample number, date and time of sampling, and the name of the sampler. The chain-of-custody document will be signed and dated by the sampler when transferring the samples.

Each sample cooler being shipped to the laboratory will contain a chain-of-custody form. The chain-of-custody form will consist of four copies which will be distributed as follows: The shipper will maintain a copy while the other three copies will be enclosed in a waterproof envelop within the cooler with the samples. The cooler will then be sealed properly for shipment. The laboratory, upon receiving the samples, will complete the three remaining copies. The laboratory will maintain one copy for their records. One copy will be returned to the Field QA/QC Officer upon receipt of the samples by the laboratory. One copy will be returned with the data deliverables package.

Chain-of-custody (COC) records are legal documents. They must be completed and handled accordingly.

The following list provides guidance for the completion and handling of all COCs.

i) COCs used should be Consultant standard forms or those supplied by the analytical laboratory. Do not use any COC forms from other labs, even if the heading is blocked out.
ii) COCs must be completed in black ball-point ink only.
iii) COCs must be completed neatly using printed text.
iv) If a simple mistake is made, line out the error with a single line and initial and date next to it.
v) Each separate sample entry must be sequentially numbered.

vi) The use of "Ditto" or quotation marks to indicate repetitive information in columnar entries should be avoided. If numerous repetitive entries must be made in the same column, place a continuous vertical arrow between the first entry and the next different entry.

vii) When more than one COC form is used for a single shipment, each form must be consecutively numbered using the "Page ___ of ___" format.

viii) If necessary, place additional instructions directly onto the COC. Do not enclose separate loose instructions.

ix) Include a contact name and phone number on the COC in case there is a problem with the shipment.

x) Do not indicate the source of the sample as this may produce a biased lab result.

xi) Before using an acronym on a COC, define clearly the full interpretation of your designation [i.e., Polychlorinated Biphenyls - (PCBs)].

E) Shipment

In all but a few cases the QA/QC plan for the field work will require shipment of samples by overnight carrier. A great many problems can be avoided by proper advance planning.

Prior to the start of the field sampling, the carrier should be contacted to determine if pickup can be made at the field site location. If pickup at the field site can be made, the "no-later-than" time for having the shipment ready must be determined.

If no pickup is available at the site, the nearest pickup or drop-off location should be determined. Again, the "no-later-than" time for each location should be determined.

Sufficient time must be allowed not only for packaging but also for delivery of samples if this becomes necessary. Driving at high rates of speed in order to make the drop time is unacceptable.

Sample shipments must not be left at unsecured or questionable drop locations (i.e., if the cooler will not fit in a remote drop box do not leave the cooler unattended next to the drop box).

Some overnight carriers do not in fact provide "overnight" shipment to/from some locations. Do not assume; call the carrier in advance before the start of the field work.

Copies of all shipment manifests must be maintained in the field file.

SAMPLING RATIONALE/APPROACH

SAMPLING TECHNIQUES

EQUIPMENT/MATERIALS

REFERENCE

1. American Society of Testing and Materials (1991), Standard D2113-83 (87), "Diamond Core Drilling for Site Investigation", Annual book of ASTM Standards, Section 4, Volume 04.08.

REMEDIATION SECTION	PROCEDURE # **SOP 8.0**
WORLDWIDE FACILITIES GROUP	EFFECTIVE DATE:
GENERAL MOTORS CORPORATION	
REVISION #: **0**	REVISION DATE:

Field Instruments – Use And Calibration

INTRODUCTION

A significant number of field activities involve usage of electronic instruments to monitor for environmental screening and heath and safety purposes. It is imperative the instruments are used and maintained properly to optimize their performance and minimize the potential for inaccuracies in the data obtained, and to insure worker's health and safety is not compromised.

This SOP provides guidance on the usage, maintenance and calibration of electronic field equipment, whether for equipment owned by the Consultant or Contractor, or equipment obtained from a rental agency.

PROCEDURES REFERENCED

- 1.5 - Data Recording – field books/ digital recording

PROCEDURE

- All monitoring equipment will be in proper working order, and operated for the purpose for which it was intended, in accordance manufacturer's recommendations.

- Field personnel will be responsible for insuring the equipment is maintained and calibrated in the field to extent practical, or returned for office or manufacturer maintenance or calibration if warranted. Calibration is discussed in greater detail below.

- A copy of the Operating Instructions, Maintenance and Service manual for each instrument used on a project will be kept on site at all times.

- Instruments will be operated only by personnel trained in the proper usage and calibration. In the event certification of training is required, personnel will have documentation of such certification with them on site at all times.

- Personnel must be aware that certain instruments are rated for operation within a limited range of conditions such as temperature and humidity. Usage of such instruments in conditions outside these ranges will only proceed with proper approval by a project manager and/or Health and Safety supervisor as appropriate.

- Instruments that contain radioactive source material, such as x-ray fluorescence analyzers or moisture-density gauges require specific transportation, handling and usage procedures that are generally associated with a license from the Nuclear Regulatory Commission (NRC) or an NRC-Agreement State. Under no circumstance will operation of such instruments be allowed on site unless by properly authorized and trained personnel, using the proper personal dosimetry badges or monitoring instruments.

- <u>Calibration:</u>

  Calibration of an electronic instrument is critical to insure it is operating properly for it's intended use. Such instruments are often sensitive to changes in temperature or humidity, or chemical vapors in the working atmosphere, and as a result their response and ability to monitor conditions and provide data can change significantly.

  Calibration: Calibration of instruments shall be performed in accordance with the manufacturer's recommendations. This includes the following parameters:

  - Frequency
  - Use of proper calibration Gases or Chemical Standards
  - Requirements for Factory Calibration

  Calibration Gas Safety: Several instruments such as photoionization detectors (PIDs), flame ionization detectors (FIDs), oxygen meters, explosimeters, combustible gas indicators and many others require use of calibration gasses contained in compressed gas cylinders. Many of these gases are combustible or explosive. Care shall be taken to minimize the potential for injury from the use of such compressed gases. Transport, handling and storage of cylinders, where necessary, shall be performed in accordance with applicable DOT regulations and site requirements.

  Calibration will only be performed in areas free of sources of spark, flame or excessive heat. Smoking will not be allowed in the vicinity of calibration gas usage areas.

  Documentation of Calibration: Instrument Calibration activities will be documented. The attached form SOP 8.0-01 will be used to record applicable

calibration and maintenance activities. In addition, protocol for documentation outlined in SOP 1.5 will be followed.

- Intrinsically Safe Requirements

  Certain work locations may be such that dangerous, ignitable or explosive conditions exist. In such cases, it may be necessary to utilize only equipment that is rated as "Intrinsically Safe." Intrinsically safe instrumentation is designed with limited electrical and thermal energy levels to eliminate the potential for ignition of hazardous mixtures.

  For site work requiring operation of monitoring instruments in Class I, Division I locations (as defined by the National Fire Protection Agency (NFPA)) only instrumentation rated as Intrinsically Safe will be used. Such equipment (including all accessories and ancillary equipment) must be rated to conform to Underwriter's Laboratories (UL) Standard 913, for use in a Class I, Division 1 Groups A, B, C, and D locations. It is also recommended the equipment conform with CSA Standard 22.2, No. 157-92.

- Upon completion of the field activities, equipment shall be returned to the possession of the Consultant, Contractor or Rental Agency accompanied by a written summary of any problems encountered with its use or calibration.

- Equipment shall be properly prepared for shipping, including insuring that residual gases (if applicable) are removed from the instrument, and accompanying containers of compressed gases or fluids are properly labeled and sealed.

- Equipment Decontamination

  Equipment that comes in contact with Site media (water level meters, water quality meters) must be cleaned **before** removal from the site to ensure that chemicals are not transferred to other sites. It is the responsibility of the person who requisitioned the equipment to ensure appropriate cleaning before returning the equipment. Equipment decontamination procedures are typically site-specific for unique site compounds.

EQUIPMENT

- Monitoring equipment specific to work plan tasks.
- Manufacturer's instructions, operation and maintenance information.
- Associated calibration gases, aqueous standards, etc.

- Appropriate shipping containers to facilitate transport without damage to equipment.

REFERENCE

- Underwriter's Laboratories, Inc. (http://www.ul.com/hazloc/define.htm) Standard UL 913.
- National Fire Protection Agency (http://www.nfpa.org/index.html)
- Canadian Standards Association (CSA) (http://www.csa.ca) Standard 22.2 No. 157

REMEDIATION SECTION	PROCEDURE # 9.0
WORLDWIDE FACILITIES GROUP	EFFECTIVE DATE:
GENERAL MOTORS CORPORATION	
REVISION #: 0	REVISION DATE:

Equipment Decontamination

INTRODUCTION

This procedure describes decontamination of field equipment potentially exposed to contaminants. Proper decontamination is required to reduce the risk of transfer of contaminants from areas of contamination to other areas and to minimize the potential for cross-contamination that would compromise sample quality. The degree of decontamination required will be dependent on the nature of the activity, equipment used and on the amount of exposure to contaminants.

PROCEDURES REFERENCED

- 2.0 - Subsurface Investigations
- 5.0 - Aquifer Characterization
- 6.0 - Sample Collection for Laboratory Analysis
- 8.0 - Field Instruments – Usage/Calibration
- 12.0 – Waste Characterization

PROCEDURE

A. General Procedure Discussion

Decontamination activities must be performed in a controlled area outside any exclusion zones established on the site. Care must be taken to minimize the potential for transfer of contaminated materials to the ground or onto other materials. Regardless of the size or nature of the equipment being decontaminated, the process will utilize a series of steps that involve removal of gross material (dirt, grease, oil etc.), washing with a detergent, and multiple rinsing steps. In lieu of a series of washes and rinse steps, steam cleaning with low-volume, high-pressure equipment (i.e. steam cleaner) is acceptable.

Drill rigs, backhoes and other exploration equipment must be decontaminated prior to initiating site activities, in-between exploration locations to minimize cross- contamination potential, and prior to mobilizing off site after completion of site work. Heavy equipment is generally best deconned with a combination of steam-cleaning equipment and detergent

scrubbing. Particular attention should be paid to parts in direct contact with contaminants, e.g. shovels, tires, augers, drilling decks, etc

Control and containerization of all decontamination fluids is critical. A decontamination pad must be constructed that is appropriate for the size and type of equipment being decontaminated. At a minimum, the decontamination pad will have the following elements:

- an impermeable barrier capable of containing decon fluids;
- a low point where fluids will collect and can be pumped into appropriate containers;
- durability to withstand equipment such as vehicle and foot traffic;
- appropriate ancillary equipment such as racks to place decontaminated equipment to drain without further exposure to contaminated fluids;
- Labels to alert personnel as to the potential presence of contaminated materials.

B. Decontamination of Non-Dedicated Specific Sampling Equipment

The following specific decon procedure is recommended:

- Brush loose soil off of equipment;
- Wash equipment with laboratory grade detergent (i.e. Alconox or equivalent);
- Rinse with tap water (three rinses minimum);
- Rinse equipment with reagent grade methanol for VOC samples (this requirement may not be appropriate for sites where methanol is a contaminant of concern);
- Rinse with distilled water;
- Rinse equipment with nitric acid for metal samples (especially important for sites with potentially high metals concentrations;
- Rinse equipment with distilled water; and
- Allow water to evaporate before reusing equipment

Decontamination waste fluids may be minimized by utilizing spray bottles (marked as to contents). Collect rinseate in a separate bucket for waste characterization.

C. Decontamination of Monitoring Equipment

Because monitoring equipment is difficult to decontaminate, care should be exercised to *prevent* contamination. Sensitive monitoring instruments should be protected when they are at risk of exposure to contaminants. This may include enclosing them in plastic bags allowing an opening for the sample intake. Ventilation ports should not be covered.

If contamination does occur, decon of the equipment will be required; however, immersion in decon fluids is not possible. As such, care much be taken to wipe the instruments down with detergent-wetted wipes or sponges, and wiped with deionized water-wetted wipes or sponges.

D. Disposal of Wash Solutions and Contaminated Equipment

All contaminated wash water, rinsates, solids and materials used in the decon process that cannot be effectively decontaminated (such as polyethylene sheeting) will be containerized and disposed of in accordance with applicable regulations and GM requirements. All containers will be labeled with an indelible marker as to contents and date of placement in the container, and any appropriate stickers required (such as PCBs).

Sampling of containerized wastes will be performed immediately upon completion of the investigations to minimize storage time on site. Storage of decon wastes on site will not exceed 90 days under any circumstances.

EQUIPMENT

Decontamination equipment and solutions are generally selected based on ease of decontamination and disposability.

- Polyethylene sheeting;
- Metal racks to hold deconned equipment;
- Soft-bristle scrub brushes or long-handle brushes for removing gross contamination and scrubbing with wash solutions;
- Large galvanized wash tubs, stock tanks, or wading pools for wash and rinse solutions;
- Plastic buckets or garden sprayers for rinse solutions;
- Large plastic garbage cans or other similar containers lined with plastic bags can be used to store contaminated clothing;
- Contaminated liquids and solids should be segregated and containerized in DOT-approved plastic or metal drums, appropriate for offsite shipping/disposal if necessary.

REFERENCE

- ASTM D5088 - Practice for Decontamination of Field Equipment Used at Non-Radioactive Waste Sites

REMEDIATION SECTION	PROCEDURE #SOP - 12.0
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GENERAL MOTORS CORPORATION	
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WASTE CHARACTERIZATION - INVESTIGATION-DERIVED WASTES (IDW)

INTRODUCTION

The following procedure describes the techniques for characterization of investigation derived waste (IDW) for disposal purposes. IDW may consist of soil cuttings (augering, boring, well installation soils, test pit soils), rock core or rock flour (from coring, reaming operations), groundwater (from well development, purging and sampling activities), personal protective equipment (spent gloves, tyvecs), (PPE) and disposal equipment (DE).

This procedure applies when disposition of investigation soils and/or groundwater is required in accordance with the project Work Plan. Generally, this procedure is applicable to Facilities where the Project Manager has assessed the areas of investigation and has developed a waste handling plan. In some areas and/or sections within a Facility it may be permitted to return test pit soil to the source area (RCRA guidance allows waste management techniques within an area of concern without 'triggering' new points of waste generation if the material is not a RCRA waste). Soil and groundwater should be handled by this characterization/disposal procedure. These practice is consistent with USEPA procedure for IDW at RCRA facilities and CERCLA sites (reference 1, 2).

Investigative soil and groundwater will not be considered a listed waste (in most circumstances) unless there is specific knowledge concerning the chemical source as a listed waste, chemical origin and timing of chemical introduction to the subsurface. Consequently waste sampling and characterization is performed to determine if the wastes exhibit one, or more characteristics of hazardous waste. Once the waste characterization is complete, RCRA regulations apply if determined hazardous. If determined to be non-hazardous solid wastes, solid waste regulations apply.

The disposal of soil cuttings and/or purged groundwater must be reviewed on a case by case basis prior to initiation of field activities.

The following outlines the waste characterization procedures to be employed when IDW disposal is required.

PROCEDURES

The procedures for handling and characterization of field activity generated wastes are:

A.) Soil/rock cuttings - Soils removed from boring activities and well construction tasks (including, rock flour from bedrock coring) will be contained within an approved container, suitable for transportation and disposal.

- Once placed into the approved container, any free - liquids (i.e., groundwater) will be poured off for disposal as waste fluids, or solidified within the approved container using a solidification agent such as speedy-dri (or equivalent). No free liquid as determined by the "paint filter test" shall be present.
- Contained soils will be screened for the presence of Volatile Organic Compounds (VOCs), using a Photo-ionization detector (PID); this data will be logged for future reference.
- Once screened, full and closed the container will be labeled in accordance with the Facility labeling requirements and placed into the Facility container storage area. At a minimum the following information will be shown a each container label: date of filling/generation, Facility name, source of soils (i.e., borehole or well), and Facility contact. If necessary, the exterior of the container will be cleaned to remove any lose dirt/cuttings.
- Prior to container closure, representative samples from a percentage of the containers will be collected for waste characterization purposes and submitted to the project laboratory. The waste characterization sampling scheme will be dictated by the Work Plan and establish the volume of soils required for analysis (depending on parameters required), the number of containers considered representative, the homogenization procedure, volatile analysis collection procedure (if required) and preparation handling requirements. Typically at a location where an undetermined site specific parameter group exists, sampling and analysis may consist of the full RCRA Waste Characterization (ignitability, corrosivity, reactivity, toxicity), or a subset of the above based upon data collected, historical information and generator knowledge.

B.) Groundwater - Well construction development, purging and sampling groundwater which requires disposal will be contained. Containment may be performed in 55-gallon drums, tanks suitable for temporary storage (i.e., nalgene or Facility provided tanks 500 to 1,000 gallons) or if large volumes of groundwater are anticipated, drilling "Frac" tanks may be utilized (20,000 gallons ±), or tanker trailer (5,000 to 10.000 gallons ±). In all cases the container/tank used for groundwater storage must be clean before use such that cross-contamination does not occur.

C.) Decon Waters/Decon Fluids - Decon waters and/or fluids will be segregated, contained and disposed accordingly.

- Decon waters may be disposed of with the contained groundwater once analytical results have been acquired. Depending on the extent of chemistry present it may be appropriate to discharge the decon waters to the Publicly Owned Treatment

Works (POTW); or discharge to an On-Site treatment system; or send off-Site for treatment.

- Spend Solvent/Acid Rinses - Solvents used during decon activities should be segregated and disposed separately from the groundwater/decon water. Often if only small amounts of solvents are involved these can be left to evaporate. If large volumes are involved then containerization, labeling and storage is required. Acid rinses may be combined with the groundwater/decon water for disposal.
- PPE/DE - a number of disposal options exists for spent PPE/DE generated from investigation tasks. The options typically employed are:
  - i) Immediately disposed of within on-site dumpster/municipal trash; or
  - ii) If known to be contaminated with RCRA hazardous waste, dispose off-Site at a RCRA-Subtitle C facility, or TSCA facility, if applicable; or alternatively decontaminated PPE/DE and dispose on-site within dumpster/municipal trash; or
  - iii) contained and store until the final remedy is implemented.

WASTE CHARACTERIZATION PROCEDURE

EQUIPMENT/MATERIALS

REFERENCE

1. USEPA RCRA Guidance and Policies: Management of Remediation Waste Under RCRA (October 1998), and Management of Contaminated Media (October 1998).
2. USEPA CERCLA Guidance (Options Relevant to RCRA Facilities): Guide to Management of Investigation - Derived Wastes (January 1992).

ATTACHMENT 2

FIELD FORMS

Form SOP 1.2 - 01

RECORD OF FIELD MONITORING

Page of

PROJECT _____ PROJECT MGR. _____
LOCATION _____ FIELD REP _____
CLIENT _____ DATE _____
CONTRACTOR _____

Project Description: _____

Task Description: _____

Instrument Type: 1) _____ Serial Number: _____
(OVA, LEL, PID, etc) 2) _____ Serial Number: _____

Calibration and/or operational check completed as per manufacturers instructions: ☐ Yes ☐ No

Time completed: _____

Weather conditions: _____

Reading Type:[1]	Level: (ppm or indicate units)	Time:	PPE Level:[2]

1. Key to Reading Types:
   Breathing Zone-BZ
   Perimeter-P
   Surface-S

2. EPA Levels B, C or D

Comments: _____

Form SOP 1.3-01

UNDERGROUND UTILITY CLEARANCE SUMMARY

Project: _____

Location: _____

Utility Clearance Organization: _____

Phone Number: _____

Date Contacted: _____ **Contacted By:** _____

Date Scheduled For Field Work Start Up: _____

On Site Meeting Called? / Date: _____

Utility Company Response Summary:

Company	Date	Utilities Present?	Utilities Marked?	Remarks

Other Comments: _____

Form SOP 5.1-01	# GROUNDWATER MONITORING REPORT	WELL NUMBER Page of

PROJECT _____ PROJECT MGR. _____
LOCATION _____ FIELD REP. _____
CLIENT _____ DATE _____
ELEVATION REFERENCED TO: _____

Date	Time	Elapsed Time (days)	Depth of Water from ( ) In Ft.	Elevation of Water	Remarks	Read By

Form SOP 8.0-01

INSTRUMENT CALIBRATION RECORD

PROJECT _____ PROJECT MGR. _____

LOCATION _____ FIELD REP _____

CLIENT _____ DATE _____

Instrument	Date Calib-rated	By	Standard Used	Decontamination, Maintenance or Repair Performed	Remarks

Other Remarks _____

STRATIGRAPHY LOG (OVERBURDEN)

PAGE ____ OF ____

PROJECT NAME _____
PROJECT NUMBER _____
CLIENT _____
LOCATION _____

DRILLING CONTRACTOR _____
DRILLER _____
SURFACE ELEVATION _____
WEATHER (A.M.) _____
(P.M.) _____

HOLE DESIGNATION _____
DATE/TIME STARTED _____
DATE/TIME COMPLETED _____
DRILLING METHOD _____
CRA SUPERVISOR _____

STRATIGRAPHIC INTERVALS (DEPTHS IN ft/m BGS)			SAMPLE DESCRIPTION	SAMPLE DETAILS								PID / FID (ppm)	CHEMICAL ANALYSIS	GRAIN SIZE
FROM	AT	TO	ORDER OF DESCRIPTORS: SOIL TYPE SYMBOL(S) – MAIN COMPONENT(S), (NATURE OF DEPOSIT), SECONDARY COMPONENTS, RELATIVE DENSITY/CONSISTENCY, GRAIN SIZE/PLASTICITY, GRADATION/STRUCTURE, COLOUR, MOISTURE CONTENT, SUPPLEMENTARY DESCRIPTORS NOTE: PLASTICITY DETERMINATION REQUIRES THE ADDITION OF MOISTURE IF THE SAMPLE IS TOO DRY TO ROLL (INDICATE IF MOISTURE WAS ADDED OR NOT).	SAMPLE #	SAMPLING METHOD	PENETRATION RECORD SPLIT SPOON BLOWS (RECORD N-VALUES & RECOVERIES) 6"	6"	6"	6"	SAMPLE INTERVAL			

NOTES AND COMMENTS

DEPTH OF BOREHOLE CAVING _____ DEPTH OF FIRST GROUNDWATER ENCOUNTER _____ TOPSOIL THICKNESS _____

WATER LEVEL IN OPEN BOREHOLE ON COMPLETION _____, AFTER ____ HOURS ____

COMPLETION DETAILS: _____

NOTE: FOR EACH SPLIT-SPOON SAMPLE, RECORD BLOW COUNTS, N-VALUE, SAMPLE RECOVERY LENGTH, AND SAMPLE INTERVAL.

CRA

SOIL CLASSIFICATION SYSTEM (MODIFIED U.S.C.S.)

MAJOR DIVISIONS			GROUP SYMBOL	TYPICAL DESCRIPTION
HIGHLY ORGANIC SOILS			PT	PEAT AND OTHER HIGHLY ORGANIC SOILS
COARSE-GRAINED SOILS (MORE THAN HALF BY WEIGHT LARGER THAN NO. 200 SIEVE SIZE)	GRAVELS MORE THAN HALF OF COARSE FRACTION LARGER THAN NO. 4 SIEVE SIZE	CLEAN GRAVELS	GW	WELL GRADED GRAVEL, GRAVEL-SAND MIXTURES, < 5% FINES
			GP	POORLY GRADED GRAVELS AND GRAVEL-SAND MIXTURES, < 5% FINES
		DIRTY GRAVELS	GM	SILTY GRAVELS, GRAVEL-SAND-SILT MIXTURES, > 12% FINES
			GC	CLAYEY GRAVELS, GRAVEL-SAND-CLAY MIXTURES, > 12% FINES
	SANDS MORE THAN HALF OF COARSE FRACTION SMALLER THAN NO. 4 SIEVE SIZE	CLEAN SANDS	SW	WELL GRADED SANDS, GRAVELLY SANDS, < 5% FINES
			SP	POORLY GRADED SANDS, OR GRAVELLY SAND, < 5% FINES
		DIRTY SANDS	SM	SILTY SANDS, SAND-SILT MIXTURES > 12% FINES
			SC	CLAYEY SANDS, SAND-CLAY MIXTURES > 12% FINES
FINE-GRAINED SOILS (MORE THAN HALF BY WEIGHT PASSES NO. 200 SIEVE SIZE)	SILTS BELOW "A" LINE ON PLASTICITY CHART; NEGLIGIBLE ORGANIC CONTENT		ML	INORGANIC SILTS AND VERY FINE SAND, ROCK FLOUR, SILTY SANDS OF SLIGHT PLASTICITY
			MH	INORGANIC SILTS, MICACEOUS OR DIATOMACEOUS, FINE SANDY OR SILTY SOILS
	CLAYS ABOVE "A" LINE ON PLASTICITY CHART; NEGLIGIBLE ORGANIC CONTENT		CL	INORGANIC CLAYS OF LOW PLASTICITY, GRAVELLY, SANDY, OR SILTY CLAYS, LEAN CLAYS
			CI	INORGANIC CLAYS OF MEDIUM PLASTICITY, SILTY CLAYS
			CH	INORGANIC CLAYS OF HIGH PLASTICITY, FAT CLAYS
	ORGANIC SILTS & ORGANIC CLAYS BELOW "A" LINE ON PLASTICITY CHART		OL	ORGANIC SILTS AND ORGANIC SILTY CLAYS OF LOW PLASTICITY
			OH	ORGANIC CLAYS OF HIGH PLASTICITY

CONVENTIONAL SOIL DESCRIPTIONS

NON-COHESIVE (GRANULAR) SOIL

RELATIVE DENSITY	BLOWS PER FOOT (N-VALUE)
Very loose	less than 5
Loose	5 to 9
Compact	10 to 29
Dense	30 to 50
Very Dense	greater than 50

COHESIVE (CLAYEY) SOIL

CONSISTENCY	BLOWS PER FOOT (N-VALUE)
Very Soft	0 to 2
Soft	3 to 4
Firm	5 to 8
Stiff	9 to 15
Very Stiff	16 to 30
Hard	greater than 30

GRAIN SIZE CLASSIFICATION

COBBLES	Greater than 3 inches (76 mm)
GRAVEL	3 in. to No. 4 (4.76 mm)
Coarse Gravel	3 in. to 3/4 in.
Fine Gravel	3/4 in. to No. 4 (4.76 mm)
SAND	No. 4 (4.76 mm) to No. 200 (0.074 mm)
Coarse Sand	No. 4 (4.76 mm) to No. 10 (2.0 mm)
Medium Sand	No. 10 (2.0 mm) to No. 40 (0.42 mm)
Fine Sand	No. 40 (0.42 mm) to No. 200 (0.074 mm)
SILT	No. 200 (0.074 mm) to 0.002 mm
CLAY	Less than 0.002 mm

NOTE: The "No. ___ " refers to the standard sieve sizes.

COMPONENT PERCENTAGE DESCRIPTORS

Noun(s) (e.g. SAND and GRAVEL)	35 to 50%
Adjective (e.g. SANDY)	20 to 35%
With	10 to 20%
Trace	Less than 10%

SOIL STRUCTURE TERMS

Stratified	Blocky
Laminated	Lenses/Seams
Fissured	Homogeneous

CRA

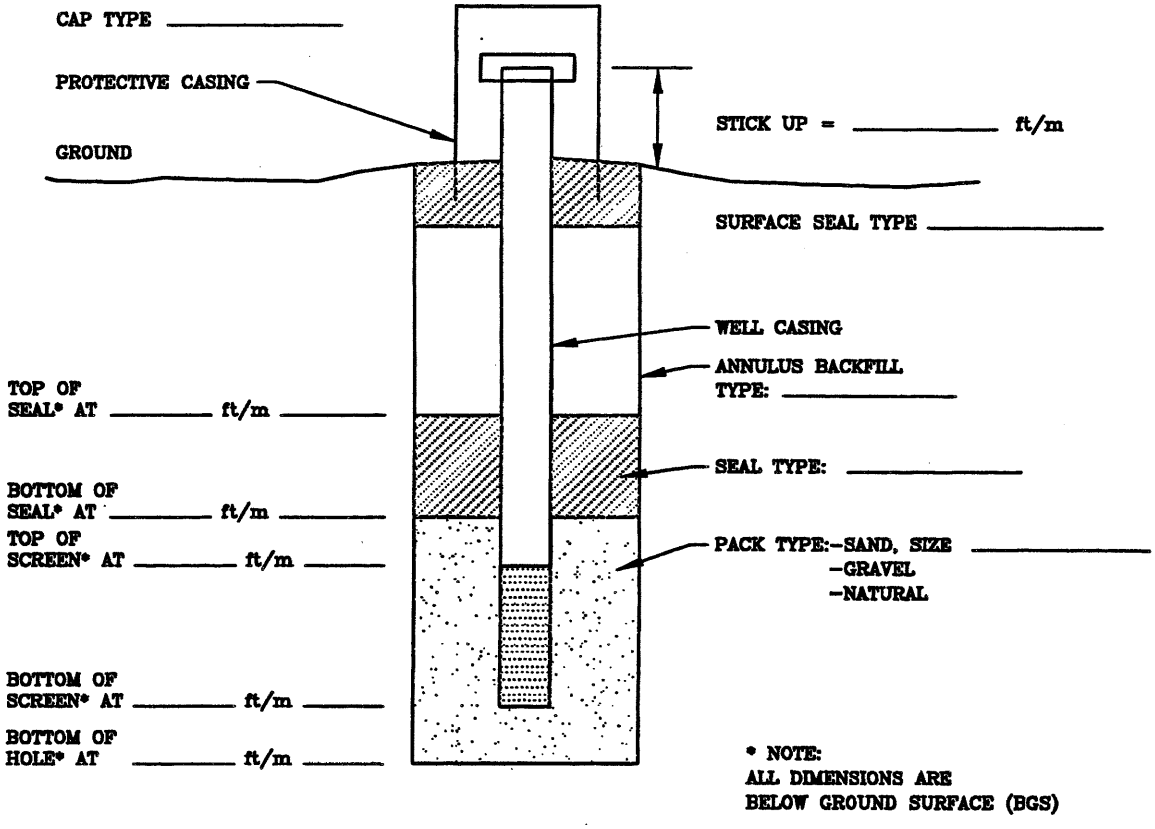
OVERBURDEN INSTRUMENTATION LOG

PROJECT NAME _____ HOLE DESIGNATION _____

PROJECT NUMBER _____ DATE COMPLETED _____

CLIENT _____ DRILLING METHOD _____

LOCATION _____ CRA SUPERVISOR _____



SCREEN TYPE: ☐ continuous slot ☐ perforated ☐ louvre ☐ other: _____

SCREEN MATERIAL: ☐ stainless steel ☐ plastic ☐ other: _____

SCREEN LENGTH: _____ ft/m SCREEN DIAMETER: _____ in/cm SCREEN SLOT SIZE: _____

WELL CASING MATERIAL: _____ WELL CASING DIAMETER: _____ in/cm

HOLE DIAMETER: _____

DEVELOPMENT: METHOD: _____ DURATION: _____

CRA

BEDROCK DRILL AND STRATIGRAPHIC LOG

PROJECT NAME _____ HOLE DESIGNATION _____ PAGE _____ OF _____

PROJECT NUMBER _____ DATE COMPLETED _____ ELEVATIONS:

CLIENT _____ DRILLING METHOD _____ TOP OF CASING: _____

LOCATION _____ SUPERVISOR _____ GROUND _____

SAMPLE DESCRIPTION	STRAT CHANGE DEPTH	RUN NO.	SAMPLE INTERVAL		% RECOV	RQD%	% WATER RETURN	LAB	TEST INTERVAL	
			FROM	TO					FROM	TO

BEDROCK STRATIGRAPHIC CORE LOG

PAGE ____ OF ____

PROJECT NAME _____
PROJECT NUMBER _____
CLIENT _____
LOCATION _____
_____

DRILLING CONTRACTOR _____
DRILLER _____
BEDROCK SURFACE ELEV. _____
CORE SIZE _____
CORING METHOD _____

HOLE DESIGNATION _____
DATE STARTED _____
DATE COMPLETED _____
CRA SUPERVISOR _____

DEPTH FROM	RUN No.	CORE RECOVERY %	RQD	DEPTH TO	GENERAL DESCRIPTION ROCK TYPE, COLOUR, TEXTURE, ETC. FORMATION: _____	STRENGTH	WEATHERING	DISCONTINUITIES							OCCASIONAL FEATURES (VUGS, ALTERATION PRODUCTS ETC.)	DRILLING OBSERVATIONS
								No. OF SETS	TYPE / S	ORIENTATION	SPACING	ROUGHNESS	APERATURE	FILLING		

CRA

MONITORING WELL RECORD FOR LOW-FLOW PURGING

Project Data:

Project Name: _____ Date: _____

Ref. No.: _____ Personnel: _____

_____

Monitoring Well Data:

Well No.: _____ Screen Length (ft): _____

Measurement Point: _____ Depth to Pump Intake (ft)[1]: _____

Constructed Well Depth (ft): _____ Well Diameter, D (in): _____

Measured Well Depth (ft): _____ Well Screen Volume, V_s (mL)[2]: _____

Depth of Sediment (ft): _____ Initial Depth to Water (ft): _____

Time	*Pumping Rate (mL/min)*	*Depth to Water (ft)*	*Drawdown from Initial Water Level*[3] *(ft)*	*pH*	*Temperature °C*	*Conductivity (mS/cm)*	*ORP (mV)*	*DO (mg/L)*	*Turbidity (NTU)*	*Volume Purged, Vp (mL)*	*No. of Well Screen Volumes Purged*[4]

Notes:

(1) The pump intake will be placed at the well screen mid-point or at a minimum of 2 ft above any sediment accumulated at the well bottom.

(2) The well screen volume will be based on a 5-foot screen length, $V_s=p*(D/2)^2*(5*12)*(2.54)^3$

(3) The drawdown from the initial water level should not exceed 0.3 ft.

(4) Purging will continue until stabilization is achieved or until 20 well screen volumes have been purged (unless purge water remains visually turbid and appears to be clearing, or unless stabilization parameters are varying slightly outside of the stablization criteria and appear to be stablizing), No. of Well Screen Volumes Purged= Vp/Vs.

WELL PURGING FIELD INFORMATION FORM

JOB# _____ - _____

SITE/PROJECT NAME: _____ WELL# _____

WELL PURGING INFORMATION

PURGE DATE (MM DD YY) _____

SAMPLE DATE (MM DD YY) _____

WATER VOL. IN CASING (LITRES/GALLONS) _____

ACTUAL VOLUME PURGED (LITRES/GALLONS) _____

PURGING AND SAMPLING EQUIPMENT

PURGING EQUIPMENT.....DEDICATED Y N (CIRCLE ONE)

SAMPLING EQIPMENT.....DEDICATED Y N (CIRCLE ONE)

PURGING DEVICE [ ] A - SUBMERSIBLE PUMP B - PERISTALTIC PUMP C - BLADDER PUMP D - GAS LIFT PUMP E - PURGE PUMP F - DIPPER BOTTLE G - BAILER H - WATERRA®

SAMPLING DEVICE [ ]

X- _____ PURGING OTHER (SPECIFY)

X- _____ SAMPLING OTHER (SPECIFY)

PURGING DEVICE [ ] A - TEFLON B - STAINLESS STEEL C - POLYPROPYLENE D - PVC E - POLYETHYLENE

SAMPLING DEVICE [ ]

X- _____ PURGING OTHER (SPECIFY)

X- _____ SAMPLING OTHER (SPECIFY)

PURGING DEVICE [ ] A - TEFLON B - TYGON C - ROPE D - POLYPROPYLENE E - POLYETHYLENE F - SILICONE G - COMBINATION TEFLON/POLYPROPYLENE

SAMPLING DEVICE [ ] X- _____ (SPECIFY)

X- _____ PURGING OTHER (SPECIFY)

X- _____ SAMPLING OTHER (SPECIFY)

FILTERING DEVICES 0.45 [ ] A - IN-LINE DISPOSABLE B - PRESSURE C - VACUUM

FIELD MEASUREMENTS

WELL ELEVATION _____ (m/ft)

DEPTH TO WATER _____ (m/ft)

GROUNDWATER ELEVATION _____ (m/ft)

WELL DEPTH _____ (m/ft)

pH	TURBIDITY	CONDUCTIVITY	SAMPLE TEMPERATURE
_____ (std)	_____ (ntu)	_____ (µm/cm) AT 25°C	_____ (°C)
_____ (std)	_____ (ntu)	_____ (µm/cm) AT 25°C	_____ (°C)
_____ (std)	_____ (ntu)	_____ (µm/cm) AT 25°C	_____ (°C)
_____ (std)	_____ (ntu)	_____ (µm/cm) AT 25°C	_____ (°C)
_____ (std)	_____ (ntu)	_____ (µm/cm) AT 25°C	_____ (°C)

FIELD COMMENTS

SAMPLE APPEARANCE: _____ ODOR: _____ COLOR: _____ TURBIDITY: _____

WEATHER CONDITIONS: WIND SPEED _____ DIRECTION _____ PRECIPITATION Y/N OUTLOOK _____

SPECIFIC COMMENTS _____

I CERTIFY THAT SAMPLING PROCEDURES WERE IN ACCORDANCE WITH APPLICABLE CRA PROTOCOLS

CRA

DATE _____ PRINT _____ SIGNATURE _____

ATTACHMENT 2

LABORATORY STANDARD OPERATING PROCEDURES (SOP)

ATTACHMENT 2

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	SOP Title	SOP ID
I.	**LABORATORY SOPs**	
A.	Support Procedures	
i)	Sample Receiving and Sample Control	NC-SC-0005
ii)	Glassware Washing	NC-QA-0014
iii)	Nonconformance and Corrective Action	CORP-QA-0010
iv)	Total Solids, Percent Moisture, Ash and Total Volatile Solids	NC-WC-0004
B.	Organics Preparation and Analysis	
i)	Determination of Volatile Organics by GC/MS Based on Methods 8260B, 624, and 524.2.	CORP-MS-0002NC
ii)	Extraction and Cleanup of Organic Compounds from Waters and Soils, Based on SW-846 3500 Series, 3600 Series, 8151A and 600 Series Methods.	CORP-OP-0001NC
iii)	GC/MS Analysis Based on Methods 8270C and 625.	CORP-MS-0001NC
iv)	Gas Chromatographic Analysis Based on Method 8000B, 8021B, 8081A, 8082, 8151A, 8310, 8141A, 8015B, 608, 610 and Wisconsin DNR Modified DRO Method.	CORP-GC-0001NC
C.	Inorganics Preparation and Analysis	
i)	Acid Digestion of Aqueous Samples by SW846 and MCAWW 200 Series Methods	CORP-IP-0003NC
ii)	Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analyses, SW-846 Method 6010B and EPA Method 200.7	CORP-MT-0001NC
iii)	Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW-846 7470A and MCAWW 245.1	CORP-MT-0005NC

ATTACHMENT 2

TABLE OF CONTENTS (CONT'D)

SOP Title		SOP ID
iv)	Acid Digestion of Soils, SW-846 Method 3050B	CORP-IP-0002NC
v)	Preparation and Analysis of Mercury in Solid Samples By Cold Vapor Atomic Absorption Spectroscopy, SW-846 7471A and MCAWW 245.5	CORP-MT-0007NC
vi)	Total Organic Carbon (TOC) and Total Inorganic Carbon (TIC)	NC-WC-0017
vii)	Cyanide Preparation Method	NC-WC-0032
viii)	Cyanide Automated, Pyridine-Barbituric Acid Method	NC-WC-0031
ix)	Determination of Inorganic Anions by Ion Chromatography	NC-WC-0084

STL STANDARD OPERATING PROCEDURE

TITLE: SAMPLE RECEIVING AND SAMPLE CONTROL

(SUPERSEDES: REVISION 6, 09/02/99)

Prepared by:		6/27/01 Date
Reviewed by:	Technology Specialist	6-27-01 Date
Approved by:	Quality Assurance Manager	6/28/01 Date
Approved by:	Environmental Health and Safety Coordinator	6-28-01 Date
Approved by:	Laboratory Director	7-2-01 Date

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1 SCOPE AND APPLICATION

1.1 It is the responsibility of Sample Receiving and Control personnel to perform the procedures described herein in full compliance with this SOP.

1.2 It is the responsibility of the Laboratory Director, QA Manager, and departmental Supervisor of the facility to assure that the procedures described are performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable personnel to perform this SOP correctly.

1.3 Analyst

1.3.1 It is the responsibility of the analyst to provide the correct request(s) for bottles to the sample custodian by using the QuantIMS program, PSR024.01 (as described in section 5.3) and to return all bottles to the custodian.

1.4 Sample Custodian

1.4.1 It is the responsibility of the sample custodian or designee to fill all bottle requests in a timely fashion and document the transfers on the request/return forms (Appendix 17.1.6).

1.4.2 It is the responsibility of the sample custodian or designee to correctly document the bottle information required (Appendix 17.1.6).

1.4.3 It is the responsibility of the sample custodian or designee to ensure that the returned bottle is the same as the one relinquished and to return it to the proper storage area.

1.4.4 This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2 SUMMARY OF METHOD

2.1 Not applicable.

3 DEFINITIONS

3.1 Sample Custodian refers to sample control personnel or designee.

3.2 Refer to the glossary in the Laboratory Quality Manual (LQM), latest version

4 INTERFERENCES

4.1 Not applicable.

5 SAFETY

5.1 Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

5.2 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately. {VITON gloves may be worn when halogenated solvents are used for extraction or sample preparation, nitrile gloves may be used when other solvents are handled. [Note: VITON is readily degraded by acetone; all solvents will readily pass through disposable latex rubber gloves.]}

5.3 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. {The following specific hazards are known} The following specific hazards are known:

5.3.1 The following materials are known to be corrosive: Sulfuric Acid, Nitric Acid, Hydrochloric Acid, Sodium Hydroxide.

5.4 Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5 The preparation of standards and reagents {and glassware cleaning procedures that involve solvents such as methylene chloride} will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6 All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported immediately to a laboratory supervisor.

5.7 This document accurately reflects current standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

6 EQUIPMENT AND SUPPLIES

6.1 Thermometers

6.2 PPE such as gloves, lab coats, safety glasses, etc.

6.3 Utility knives

6.4 PH paper

6.5 Copier, printer, computer and label generator

6.6 Carts

7 REAGENTS AND STANDARDS

7.1 Not applicable.

8 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Not applicable.

9 QUALITY CONTROL

9.1 Nonconformance and Corrective Action

9.1.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10 CALIBRATION AND STANDARDIZATION

10.1 Not applicable.

11 PROCEDURE

11.1 Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.

11.2 The procedures listed in this document describe the responsibilities of Sample Control personnel in ensuring that data is transmitted correctly from the client samples to all personnel involved with sample analysis and review.

11.3 The intent of the sample custodian maintenance program is to show custody of individual bottles based on workorder number as well as bottle control numbers. This program acts as an internal chain of custody.

11.4 The sample control group opens each cooler and removes the enclosed sample documents. Coolers are prioritized based on rush status and expirable tests. If a COC (chain-of-custody) is marked rush, the following occurs:

24 hour TAT – flag with red folder
48 hour TAT – flag with blue folder
72 hour TAT – flag with yellow folder
1 week TAT – flag with green folder

11.5 If tests on the COC are expirable, the cooler is marked with a manila folder.

11.6 Rush and expirable coolers are to be unpacked first and logged as soon as possible. The lab groups are to be notified with any rush 72 hours or less.

11.7 The following information on the Cooler Receipt/ Narrative Form (Appendix 17.2.1).

11.7.1 Presence of the custody seals on the outside of the cooler

11.7.2 Presence of the custody papers inside the cooler

11.7.3 The custody papers were properly filled out (ink, signed, match labels)

11.7.4 The custody papers were signed in the appropriate place

11.7.5 Presence of the shipper's packing slip

11.7.6 Presence of packing material information: if yes, type of packing material

11.7.7 Conditions of samples at receipt (chilled, etc.).

11.7.7.1 If temp vial is present, it is used to take the temperature. The temperature of the temp vial is taken as soon as it is removed from the cooler. If a temp vial is not present, proceed to section 11.7.7.2.

11.7.7.2 When a temp vial is not present the temp is taken by placing the thermometer probe between the coolant and the sample(s). If a coolant is not present, proceed to section 11.7.7.3.

11.7.7.3 If no coolant is in the cooler, the thermometer probe is placed between two sample bottles and the temperature recorded.

11.7.7.4 If the temperature is outside 4°C ± 2°C, the anomaly is recorded on the cooler receipt form. The project manager is contacted when the temperature is >6°C.

11.7.8 Bottles sealed in separate plastic bags

11.7.9 Condition of bottles upon receipt (good condition, broken, etc.)

11.7.10 Complete bottle labels (date, time, client ID)

11.7.11 Information on bottle labels and tags agree with custody papers

11.7.12 Correct bottles used for the tests indicated

11.7.13 VOA bottles were checked for the presence of air bubbles. Any bubbles exceeding 6 mm in diameter are narrated and the PM is contacted.

11.7.14 Sufficient amount of sample sent in each bottle

11.7.15 Samples were received via overnight courier, client drop off, or other means

11.7.16 pH's are taken, on all preserved samples less Volatiles, TOC, and TOX by removing sample lids and using a droplet of sample from in the lid to test the pH. The pH's are then recorded on the cooler receipt form. The pH paper strips are then discarded.

11.7.16.1 In determining if the sample is preserved to the correct pH for Navy samples, the sample custodian or receiving personnel must take an aliquot of sample out of the sample container either by pouring out a small aliquot or using a disposable pasteur pipette and place a drop of the sample on a pH paper strip.

11.7.17 Purchased prepared vials of preservatives are used if samples are not at the correct pH. The pH is adjusted by adding the appropriate preservative in 5 mL increments up to a maximum of 20 mL or unless there is a reaction. Sulfides are preserved with 5 mL Sodium hydroxide and 1 mL Zinc acetate. The pH adjustment is noted on the cooler receipt form.

11.7.18 The concentrations of the preservatives used:

4N Sodium Hydroxide
1N Zn Acetate
1:1 HCL (18%)
1:4 $HN0_3$ (18%)
1:2 H_2SO_4 (33%)

11.7.18.1 If the pH of Navy samples are adjusted, the sample custodian or receiving personnel must record on the cooler receipt form the laboratory assigned reagent ID number.

11.8 The Sample Control person is to remove all sample containers. Any broken, leaking, or dirty sample containers are to be placed inside the fume hood. Dirty sample containers are to be cleaned appropriately, so as not to contaminate the sample storage area. The Sample Control person is to wear disposable latex gloves, safety glasses, and a lab coat while handling any samples.

11.9 Any volatile sample(s) suspected (e.g., odor) or known (client information or site history) to be high in volatile concentration, the volatiles department will be contacted for pick-up and segregation of sample(s).

11.10 The Sample Control person is to examine all documents and compare information from sample container labels and Chain-of-Custody Records to insure that there is no discrepancy between documents, ensuring that all documents are properly completed and signed.

11.11 If any problems or discrepancies are noted during the sample receiving process that compromise sample integrity, such as limited sample volume, sample identification cannot be determined from the COC, incorrect pH levels (or preservatives if known), or broken, leaking samples, the Project Manager is immediately notified. They in turn will contact the client in an attempt to solve discrepancies.

11.12 Expirable tests (hold time 48 hours or less) must be written on the top of the bottles from which they are to be analyzed. If more than one method exists for the analysis, the method must also be written. These bottles designated for expirable tests will be given directly to the wet chem lab group.

11.13 All solid /soil samples received are split off for the total solid analysis. Splitting these samples is the responsibility of the receiving group. A small portion (approx. 5-10 grams) from each sample is put into a small plastic snap-top container designated for the TS analysis. If the TS container is not labeled with a QuantIMS label at the time of splitting off, the TS container must be labeled with handwritten client ID. Containers designed for VOC analysis must not be opened/used for TS aliquot. When only one solid container is received for VOC analysis, receiving group must **not** split off because of possible contamination or possible lost volatiles. An empty TS container with a QuantIMS label is given to the VOC analysis group for each solid. The VOC group will aliquot for the TS when they open container for analysis. TS plastic containers are placed into baggies by lot (project) and the baggies are put into a box inside the walk-in cooler door.

11.14 If all samples recorded on the Chain-of-Custody Record were received by the laboratory and there are no problems observed with the sample shipment, the Sample Control person will sign the Chain-of-Custody Record in the "Received for Laboratory by:" box on the document. If problems are noted, sign for shipment and note the problems. All discrepancies are recorded on Cooler Receipt Form.

11.15 A quote must reflect what is on the chain-of-custody. Any discrepancies must be resolved by the project manager. Likewise, if there is not an associated quote in QuantIMs, the samples are placed on hold until a quote is completed by the project manager.

11.16 In the event that a project is on hold until the next day or longer, all associated paperwork is placed into a black folder and all samples and black folder are put into cold storage. Any project that is on hold must be recorded on the dry-erase board posted on walk-in cooler door.

11.17 The Sample Control person will enter each sample into the laboratory computer (QuantIMS), where a unique lot number is assigned to each project received, and sequential sample numbers are designated for each client identification within the lot.

11.17.1 Lot Numbers: The lot number is nine characters in length and is based on the date of receipt. Lot number A9J010121 is described as follows:

A - STL location where the samples were received.

(A = North Canton, B = Tampa, C = Pittsburgh, etc.)

9 - Last digit of the year (i.e. 1999).

J - Month (i.e. A = January, B = February, J = October, etc.)

01 - The next 2 numeric characters identify the day of the month, in this case, the first day of the month.

0121 - The next 4 numeric characters are the sequential assignment of numbers specific to each lot received. Each day the first lot logged in receives the number "0101", the second lot receives the number "0102", etc..

For example:

If four bottles were submitted under Client ID numbers AB100-AB103 and the laboratory identification number generated by the computer is A9K100101, then the assigned laboratory number recorded on the Sample Log-In Sheet would be as follows.

Client ID Sample Number	Assigned Laboratory Number
AB 100	A9K100101-001
AB 101	A9K100101-002
AB 102	A9K100101-003
AB 103	A9K100101-004

11.17.2 Sample Numbers: The samples in each lot are assigned a sample number that is attached to the lot number and are reset at each new lot. For example: the first and second samples in the lot above are labeled A9J010121-001 and A9J010121-002.

11.17.3 Sample Suffixes: Each sample also has a 1 character field (which is not a required field for all samples) called the suffix which identifies the sample as specified below.

Client Sample	no suffix
Method Blank	B
Laboratory Control Sample	C
Laboratory Control Sample Duplicate	L
Matrix Spike	S
Matrix Spike Duplicate	D
Sample Duplicate	X
Serial Dilution	P
Sample Confirmation	Y
Post Digestion Spike	Z
Re-analysis	I

Example: A9J010121-001X is a sample duplicate for sample A9J010121-001.

11.17.4 Work Order Numbers: Each test requested by the client for an individual sample receives an individual 8 digit work order number assigned by QuantIMS. Work order number A5WE1-2-1C is described as follows:

A5WE1 - In addition to the three digit sample identification described in 4.7.2 (i.e. - 001 and - 002), the first 5 characters of the work order number also identifies each unique sample. This identification is generated in QuantIMS using a sequential logic that is beyond the scope of this SOP to describe.

2 - The "modifier" indicates the type of run. In this case this is the second time the sample had to be run. If it needs reprepped and run again, the number would indicate a "3". The original analysis work order number assigns "1" to the modifier position.

1C - The "suffix" is the identification of the specific test for that sample. The suffix in this case is not always sequential, but is unique to the test to be performed on the sample.

Example: A5WE1-2-1C is the assigned 8 digit work order number for the reanalysis of the chloride test on the sample A5WE1. A5WE1-1-05 could be the 8 digit work order number for the analysis of SW846 8270 on sample A5WE1.

11.17.5 Each sample container with the same client ID has a unique number. Each container will be labeled with the same 5 digit work order number and then will contain a suffix beginning with – 001, -002, -003, etc. For example, if 5 containers are submitted from the same sample point and the LIMS-generated work order number is CREE4, LIMS will generate 5 labels: CREE4-001, CREE4-002, CREE4-003, CREE4-004, CREE4-005. The specific bottle number is the number used for sample request/removal paperwork.

11.17.6 Expirable tests must be given to the lab groups the day they are received. The expirable test/method is written on top of the bottle and the bottle must be put in the red bin designated for expirables. The work order or sample ID must be recorded on the expirable logsheet – also must record the test to be run, special method if necessary and the initials of the person relinquishing the sample. The wet chem lab group checks this bin throughout the day and is responsible for signing out the sample container when they take it.

11.17.7 Once all sample containers have been properly labeled and all the information has been recorded by the Sample Lot Summary, the Sample Control person will place the samples into the proper storage locations. These locations are as follows:

11.17.8 Organic extractable samples (Semivolatiles, Pesticides/PCBs) are to be placed into the walk-in refrigerators located in Sample Receiving.

11.17.9 Volatile samples are to be stored in the double-door refrigerator located in the Sample Custodian area.

11.17.9.1 Samples known or suspected to be of high concentration are not stored in these refrigerators. The applicable volatile group is contacted to obtain the samples for segregation.

11.17.10 Inorganic samples are to be placed into the walk-in refrigerators located in Sample Receiving.

11.18 Preserved metal samples are placed in a non-refrigerated room located in the Sample Custodian area. Metals samples that need to be lab filtered and preserved are stored in the walk-in cooler. All metals for Navy projects are stored in the walk-in cooler.

11.19 One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.20 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.21 For clients who request a show of sample transfer from sample receipt to storage, a sample control record is completed (see Appendix 17.2.4 or Appendix 17.2.5). STL LIMS will generate this record for Expanded Deliverable and CLP designated samples. This record is also referred to as an internal chain of custody (COC). The form is completed as follows:

11.21.1 Laboratory sample number - record/list the individual five digit work order number in this column. List all pertaining samples in the project lot. Use an additional sheet if necessary. Do not record multiple lots on a sample control record.

11.21.2 Transferred by - record name of person making the transfer

11.21.3 Date - record date of sample transfer

11.21.4 Entered - "X" or "√" since samples are already logged into the LIMs.

11.21.5 Removed - leave blank since samples are not being removed

11.21.6 Reason - record "storage"

11.21.7 Date returned - leave blank

11.22 The completed sample control record is attached to the summary package. The sample control record can be manually printed using the SAM S31 command in QuantIMS. Note: The report package for the lot must be "D" (Expanded Deliverable) or "C" (CLP) for the report to print.

11.23 Samples are requested by an analyst through the QuantIMS program PSR024.01. This program is accessed from the Sample Receiving Menu (SAM) by selecting option S24 (Sample Removal Requests).

Bottles are requested based on method code, workorder number, prep code, QA batch number, lot number, or method group.

11.24 Each bottle has an associated lot number, sample workorder number, and bottle suffix number (Appendix 17.2.9).

11.25 When an analyst requests bottles and exits the request program, a requisition (Appendix 17.2.6) prints in the custodian's work area. This requisition identifies the requestor and the method/parameter requested. The sample custodian fills the requisition, recording each bottle number on the form. The bottle(s) is/are signed out to the custody of the requestor by indicating the name and the date of transfer. The request is relinquished with the custodian's initials and accepted by the requestor's initials.

11.26 When bottles are returned, the sample custodian records the return on the original requisition form and re-enters custody of the bottles to the sample control area. The custodian initials and dates the return on the form (Appendix 17.2.6).

11.27 When sample bottles are consumed in the analysis process, the empty container is returned to the custodian. The custodian marks a "C" by the appropriate sample on the request form to indicate a consumed sample (Appendix 17.2.6).

11.28 Samples returned after the custodian has left at the end of the day are placed in the walk-in cooler by a custodian designee and recorded as received by the custodian the next working day.

11.28.1 All request forms remain in the sample custodian area while samples are being analyzed. The final form is also kept in the sample custodian area after samples have been analyzed and reported.

11.28.2 When a sample custodian is not available, a designee gets the samples, and all request paperwork is completed properly.

11.29 Subcontracting of samples

11.29.1 Samples that are logged but not analyzed at the laboratory are subcontracted to different laboratories for analysis including other STL facilities.

11.29.2 The LIMs system will automatically print a Sample Analysis Requisition for these samples upon completion of the log-in process (Appendix 17.2.7).

11.29.3 This form contains information necessary for sample analysis. The original form is sent to the subcontracted laboratory and a copy is attached to the summary package. The Sample Analysis Requisition form must have a relinquished signature with a date and time. Any additional information necessary for sample analysis must be handwritten on the form (e.g. list of compounds, homogenizing of samples, limited quantity, etc.). In order to track subcontracted samples, the lab purchase order number on the Sample Analysis Requisition form must be recorded in the subcontracted sample PO book located in the receiving log-in area.

11.29.4 For samples sent to a non QuantIMS lab a chain of custody form is filled out and accompanies the samples.

11.29.5 A sample analysis request (SAR) can be printed from LIMS by entering the Lot Number. The SAR form can be found on Main Screen on receiving computer terminals.

12 DATA ANALYSIS AND CALCULATIONS

12.1 Not applicable.

13 METHOD PERFORMANCE

13.1 Training Qualifications:

13.1.1 The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.1.2 The only personnel authorized to execute this SOP are the Sample Log-In persons.

14 POLLUTION PREVENTION

14.1 This method does not contain any specific modifications that serve to minimize or prevent pollution.

15 WASTE MANAGEMENT

15.1 All samples may be disposed of 30 days after the report date except for those samples associated with special client retention. Shelves are purged in chronological order. All lots on a specific shelf must be noted. If a lot can be disposed of, a disposal date must be recorded.

15.2 Disposal dates are recorded on a print-out of lots according to a storage location. When clearing samples that cannot yet be disposed of because of special client retention, samples are boxed or stored on carts. Stored samples must have a specific date listed in which samples can be disposed of, or a note that indicates "SAVE" and client name or reason.

15.3 All lots assigned to a certain shelf can be obtained from QuantIMS.

15.3.1 UTL – "Enter"

15.3.2 Type "wrk" – "Enter"

15.3.3 Type "2" by option

15.3.4 Choose one query name and "Enter"

15.3.5 SLOIS for metals shelves

15.3.6 CLOIS for cooler shelves C1 – C272

15.3.7 WLOIS for cooler shelves W1-163

15.3.8 Type "1" next to "select records"

15.3.9 Enter the shelf location 3x where prompted (appears on two different screens). Then hit "Enter".

15.3.10 Dates need to be updated every four to six months so shelf locations only print most recent lots assigned.

15.3.11 "F3" to run query and "Enter".

15.3.12 At save definition type "Y" and run option "2" and "Enter".

15.4 Solid samples that can be disposed of are boxed and labeled "SOLIDS NOW."

15.5 Liquid wastes that can be disposed of are boxed and labeled "LIQUID NOW." These boxes are taken to the facility waste building and drummed by the Health and Safety Coordinator or designee.

15.6 Water samples that can be disposed of are placed on carts and purged down the drain in one of the following areas: sample receiving, wet chemistry, or extractions.

15.7 Solvent waste must be disposed of in clearly labeled waste cans.

15.8 Acid waste must be collected in clearly labeled acid waste containers. Solid materials (gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.9 Refer to the Laboratory Sample and Waste Disposal plan.

15.10 Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16 REFERENCES

16.1 STL Quality Management Plan (QMP), current version.

16.1.1 STL Laboratory Quality Manual (LQM), current version.

16.2 Associated SOPs and Policies, latest version

16.2.1 QA Policy, QA-003

16.2.2 Navy/Army SOP, NC-QA-0016

17 MISCELLANEOUS

17.1 Wherever "Sample Control" is mentioned in all SOPs, it is assumed to include the sample custodian or any alternate that is designated by the Sample Control Coordinator.

17.2 Appendix:

17.2.1 Cooler Receipt Form/Narrative

17.2.2 Preservative Preparation

17.2.3 Preservative Requirements

17.2.4 Internal Chain of Custody – CLP

17.2.5 Internal Chain of Custody – Regular Production Organic LIMS generated

17.2.6 Sample Custodian Removal Request

17.2.7 Sample Analysis Requisition

17.2.8 Subcontracted PO Logbook

17.2.9 Laboratory Generated Bottle Label

17.1.1

Example

Cooler Receipt/Narrative Form

STL Cooler Receipt Form/Narrative

North Canton Facility

Client:_____ Project:_____ Quote#:_____

Cooler Received on:_____ Opened on:_____ by: _____

(Signature)

Fedx ☐ Client Drop Off ☐ UPS ☐ Airborne ☐ Other:_____

Cooler ☐ Safe ☐ Foam Box ☐ Client Cooler ☐ Other:_____

STL Shipper No#:_____

1. Were custody seals on the outside of the cooler and intact? Yes ☐ No ☐

   If YES, Quantity_____ Location_____

   Were the custody seals signed and dated? Yes ☐ No ☐ NA ☐

2. Shipper's packing slip attached to this form? Yes ☐ No ☐

3. Were custody papers included inside the cooler and relinquished? Yes ☐ No ☐

4. Did you sign the custody papers in the appropriate place? Yes ☐ No ☐

5. Packing material used:

Peanuts ☐ Bubble Wrap ☐ Vermiculite ☐ Foam ☐ None ☐ Other :_____

6. Cooler temperature upon receipt _____ °C (see back of form for multiple coolers/temp)

METHOD: Temperature Vial ☐ Between Coolant & Sample Container ☐ Against Bottles ☐

COOLANT: Wet Ice ☐ Blue Ice ☐ Dry Ice ☐ Water ☐ None ☐

7. Were all the bottles sealed in separate plastic bags? Yes ☐ No ☐

8. Did all bottles arrive in good condition (Unbroken)? Yes ☐ No ☐

9. Did all bottle labels and tags agree with the custody papers? Yes ☐ No ☐

10. Were samples at the correct pH? Yes ☐ No ☐ NA ☐

11. Were correct bottles used for the tests indicated? Yes ☐ No ☐

12. Were air bubbles >6 mm in any VOA vials? Yes ☐ No ☐ NA ☐

13. Was a sufficient amount of sample sent in each bottle? Yes ☐ No ☐

Contacted PM _____ Date:_____ by:_____ via Voice Mail ☐ Verbal ☐ Other ☐

Concerning:_____

√	MACRO	MACRO
1. CHAIN OF CUSTODY		
	SR1A	Samples were received under proper custody procedures and without discrepancies.
	SR1B	The chain of custody and sample bottles did not agree. The following discrepancies occurred_____
2. SAMPLE CONDITION		
	SR2A	Sample(s)_____ were received or requested after the recommended holding time had expired.
	SR2B	Sample(s) _____ were received with insufficient volume
	SR2C	Sample(s)_____ were received in a broken container.
3. SAMPLE PRESERVATION		
	SR3A	Sample(s) _____ were further preserved in sample receiving to meet recommended pH level(s).
	SR3B	Sample(s)_____were received with bubble > 6 mm in diameter (cc: PM)
4. NCM		
	SR4A	NCM has been generated. Refer to Clouseau for details
5. Other Anomalies (see below or back)		

STL Cooler Receipt Form/Narrative

North Canton Facility

Client ID	pH	Date	Initials

Discrepancies Cont.

Macro Name:

Macro Name:

Macro Name:

Macro Name:

Other Anomalies:

17.1.2

Preservative Preparation

Preservative Preparation

(If purchased preservative solution vials are not used)

1:1 Hydrochloric Acid (18%): Slowly add 1000 mL concentrated HCl to 1000 mL reagent water and mix. Store in a well labeled plastic coated acid bottle.

1:2 Sulfuric Acid (33%): In a 2000 mL beaker, SLOWLY and CAREFULLY add 500 mL concentrated H_2SO_4 to 1000 mL reagent water and mix. A cool water bath may be needed to cool the solution and beaker. Store in a well labeled plastic acid bottle.

NOTE: *All preparations must be performed in a hood and proper personal protective equipment must be worn. All reagents and final preservative solution must be documented in applicable reagent logbooks.*

17.1.3

Preservative Requirements

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative[1,2]	Volume	Parameter	Container	Preservative [1,2]	Volume
Asbestos	P	None	250 mL	Radiological Alpha, Beta, Radium	P	HNO_3	4 L
Acidity	P	None	250 mL	Hardness	P	HNO_3	250 mL
Alkalinity (Sep)	P	None	250 mL	Metals	P	HNO_3	1 L
BOD	P	None	250 mL	Dissolved Metals*	P	HNO_3	1 L
Carbonaceous BOD	P	None	250 mL	Total Organic Carbon (TOC)	G	HCl	2 x40 mL
Bromide (Br)	P	None	250 mL	Chemical Oxygen Demand	P	H_2SO_4	250 mL
Chloride (Cl)	P	None	250 mL	Total Organic Halogens	G	H_2SO_4	250 mL
Chromium, $^{6+}$	P	None	250 mL	COD	P	H_2SO_4	250 mL
R. Chlorine	P	None	100 mL	Ammonia Nitrogen (NH_3)	P	H_2SO_4	500 mL
Color	P	None	50 mL	TKN	P	H_2SO_4	1L
Conductivity	P	None	250 mL	Nitrate/Nitrite	P	H_2SO_4	250 mL
Corrosivity	P	None	250 mL	Oil & Grease	G	H_2SO_4	1 L
Dissolved Oxygen	G	None	300 mL	Phenols	G	H_2SO_4	1 L
Fecal Coliform	P	None	125 mL	Total Phosphorus	P	H_2SO_4	250 mL
Flashpoint	G	None	100 mL	TON	P	H_2SO_4	1 L
Fluoride	P	None	250 mL				
Nitrate	P	None	250 mL	TRPH - IR 418.1	G	HCl	2 L
Nitrite	P	None	250 mL	VOC 601	G	HCl	3x40 mL
pH	P	None	50 mL	VOC 8010	G	HCl	3x40 mL

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative[1,2]	Volume	Parameter	Container	Preservative [1,2]	Volume
Elemental PO_4	G	None	250 mL	VOC 624	G	HCl	8x40 mL
Orthophosphate	P	None	250 mL	BTEX 8020	G	HCl	3x40 mL
TDS	P	None	250 mL	VOC 8240	G	HCl	3x40 mL
TSS	P	None	250 mL	THM/502.2	G	HCl	2x40 mL
Total Solids	P	None	250 mL	502.2	G	HCl & Asc. Acid	2x40 mL
TVS	P	None	250 mL	VOC 624	G	HCl	3x40 mL
T. Coliform	P	None	125 mL	VOC 602	G	HCl	3x40 mL
Settleable Solids	P	None	1L	465 C & D	G	HCl	4x40 mL
Silica	P	None	250 mL	BTEX 8021	G	HCl	3x40 mL
Sulfate	P	None	250 mL	VOC	G	HCl	3x40 mL
Sulfite	P	None	250 mL	VOC 8260	G	HCl	3x40 mL
Surfactants (MBAS)	P	None	250 mL	VOC and VOA	G	HCl	3x40 mL
Turbidity	P	None	250 mL	VOC 8010/8020	G	HCl	3x40 mL
TPH-GC	G	None	2 L				
				Total Cyanide	P	NaOH[3]	250 mL
BNAs	G	None	2 L	Amenable Cyanide	P	NaOH	250 mL
BNA + Dioxin	G	None	2 L	Free Cyanide	P	NaOH	250 mL
PNA/PAH	G	None	2 L	Sulfide	P	Zn Acetate & NaOH	1 L
Pesticides	G	None	2 L	Formaldehyde	G	None	500 mL
Reactive Cyanide	P	None	1 L	Carbonate	P	None	250 mL
Reactive Sulfide	P	None	1 L	Bicarbonate	P	None	250 mL
PCB	G	None	2 L	TPH - Diesel (Ext.)	G	None	2 L

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative[1,2]	Volume	Parameter	Container	Preservative [1,2]	Volume
Pesticides + PCBs	G	None	2 L	TPH - Gasoline (P&T)	G	HCl	2x40 mL
Herbicides	G	None	2 L	Glycols 8015	G	None	2x40 mL
OPPs	G	None	2 L	BTEX & MTBE	G	HCl	3x40 mL
				601/602	G	HCl	3x40 mL

* Filtered in field

[1] HCl, HNO_3, and H_2SO_4 to pH < 2. NaOH to pH > 12

[2] Temperature = 4°C ± 2°C except for aqueous metals

[3] Samples to be analyzed for Cyanide should be field-filtered for Residual Chlorine. If Residual Chlorine is detected, ascorbic acid (0.6 g) should be added.

17.1.4

Example

Internal Chain of Custody - CLP

STL North Canton

Sample Control Record

Client: _____ Case Number/SDG: _____

Storage Location: _____

Laboratory Sample Number	Transferred By	Date	Entered	Removed	Reason	Date Returned
1.						
2.						
3.						
4.						
5.						
6.						
7.						
8.						
9.						
10.						

17.1.5

Internal Chain of Custody

Regular Production-Organic

STL North Canton

Sample Control Record

Client: _____ Case Number/SDG: _____

Storage Location: _____

Laboratory Sample Number	Transferred By	Date	Entered	Removed	Reason	Date Returned
11.						
12.						
13.						
14.						
15.						
16.						
17.						
18.						
19.						
20.						

17.1.6

Example

Sample Custodian Removal Request Form

(LIMS Generated)

17.1.7

Example Sample Analysis Requisition

17.1.8

Example Subcontracted PO Logbook

17.1.9

Laboratory Generated Bottle Label

STL North Canton
STANDARD OPERATION PROCEDURE

TITLE: GLASSWARE WASHING

(SUPERSEDES: REVISION 3, DATED 01/11/01)

Prepared by: _____ 7/03/01
Date

Reviewed by: Patrick O'Meara 7/11/01
Technology Specialist Date

Approved by: _____ 7/3/01
Quality Assurance Manager Date

Approved by: _____ 7-10-01
Environmental Health and Safety Coordinator Date

Approved by: _____ 7-6-01
Laboratory Director Date

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1. PURPOSE

1.1. The procedure listed in this document will describe the standard operating procedures for washing glassware in the laboratories.

1.2. This document accurately reflects current standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

2. RESPONSIBILITIES

2.1. It is the responsibility of the analyst to perform the analysis described herein in full compliance with this SOP.

2.2. It is the responsibility of the Laboratory Director, QA Manager, and departmental Supervisor of the facility to assure that the analysis described is performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable the analyst to perform this SOP correctly.

3. SAFETY

3.1. Procedures shall be carried out in a manner that protects the health and safety of all STL North Canton associates.

3.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately. {VITON gloves may be worn when halogenated solvents are used for extraction or sample preparation, nitrile gloves may be used when other solvents are handled. [Note: VITON is readily degraded by acetone; all solvents will readily pass through disposable latex rubber gloves.]}

3.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

3.3.1. The following materials are known to be corrosive: **Nitric Acid, Hydrochloric Acid, Chromic Acid**

3.3.2. The following material is known to be flammable: **Acetone**.

3.4. Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

3.5. The glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operation will permit.

3.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported immediately to a laboratory supervisor.

3.7. Glassware in contact with chemicals used in analytical procedures may be toxic or carcinogenic. Therefore, each piece of glassware should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals should be reduced to the lowest possible level.

3.8. Hands must not be placed in the glassware while washing. An appropriate scrub brush must be used to clean the inside of glassware. This will prevent the breakage of glassware while trying to force hands in or out of apparatus.

4. PROCEDURES

4.1. Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.

4.2. Metals

4.2.1. Dirty glassware is taken to a central location and thoroughly rinsed. Any ink on the outside of the glassware is removed with acetone.

4.2.2. The glassware is immersed in a hot, soapy solution of water and laboratory detergent. An appropriate scrub brush or pad is used to scrub the glassware.

4.2.3. Rinse the glassware thoroughly three times with hot tap water and then rinse once with 1:1 nitric acid. Appropriate protective wear should be worn. If the glassware is still visibly dirty, or if spotting or beading occurs, repeat 4.2.2.

4.2.4. Rinse the glassware three times with analyte-free water and place it in a clean drying area.

4.2.5. After air drying, the glassware should be spot and stain free. If it is not, then repeat the entire procedure. Seldom used items should be stored to minimize contamination.

4.3. Wet Chemistry

4.3.1. After using a piece of glassware, it is thoroughly rinsed with hot tap water and carried to the dirty glassware area. If it is visibly dirty, it should be washed immediately.

4.3.2. The glassware is immersed in a hot, soapy solution of water and laboratory detergent. An appropriate scrub brush or pad is used to scrub the glassware. A mechanical, laboratory dishwasher equipped with a DI water rinse is also approved for glassware cleaning.

4.3.3. Rinse the glassware thoroughly three times with hot tap water. If the glassware is still visibly dirty, or if spotting or beading occurs, repeat 4.3.2.

4.3.4. After air drying, the glassware should be spot and stain free. If it is not, then repeat the entire procedure.

4.3.5. Place the glassware in a designated clean storage location free from interferences or contamination.

4.4. Semi-volatile Organics

4.4.1. If high level contamination is suspected perform the appropriate high level cleaning before proceeding with the standard cleaning process described in Section 4.4.2 and following.

4.4.1.1. Non-polar organics: if the glassware was most recently *wet* with a solvent, then rinse 3 times with that solvent, if *wet* with water, then rinse 3 times with acetone. Collect rinses in the appropriate solvent waste container. Allow the remaining solvent to evaporate from the glassware in a ventilation hood to reduce analyst exposure to the solvent.

4.4.1.2. Organic acids (e.g. phenols, carboxylic acids such as some herbicides): rinse 3 times with 1 M NaOH. Collect rinses in the water sample waste container.

4.4.1.3. Organic bases (e.g. amines [fish smell]): rinse 3 times with 1 M H_2SO_4. Collect rinses in the water sample waste container.

4.4.2. After using a piece of glassware, it is thoroughly rinsed with hot tap water and carried to the dirty glassware area. If it is visibly dirty, it should be washed or soaked immediately rather than allow the residue to harden and become more difficult to wash.

4.4.3. The glassware is immersed in a hot, soapy solution of water and laboratory detergent (recommended pH > 10). An appropriate scrub brush is used to scrub the glassware. Change the wash water (when it becomes cold, visibly dirty or is used on glassware with oil or sediment residue) by emptying, rinsing the inside of the sink with hot water and refill with hot water and detergent.

4.4.4. Rinse the glassware three times vigorously with hot tap water. If the glassware is still visibly dirty, or if spotting or beading occurs, rinse with 1:1 HCl and repeat 4.4.3.

4.4.4.1. If the HCl rinse is not effective, rinse or soak the item in chromic acid. Return as much chromic acid to the storage bottle as practical. Rinse the item with reagent water, collecting the rinses in an acid waste container. Chromic acid is very corrosive and must be handled with great care. Chromic acid can be reused until it begins to turn green. Spent chromic acid and chromic acid rinse water are transferred to the metals waste container.

4.4.5. After completing the hot tap water rinse, rinse the glassware three times with deionized water and place it in a clean drying area.

4.4.6. Place cleaned glassware used for semi-volatiles in a muffle oven for approximately one hour at 400°C. Do not heat glassware with visible residue. Baked on residue is much more difficult to remove.

4.4.7. Clean glassware is inverted (where applicable) and stored prior to use in a designated clean storage location.

4.4.8. Before use, glassware is to be pre-rinsed with the solvent inherent to a given procedure if stored more than one week.

4.5. Volatile organics

4.5.1. Rinse the glassware vigorously with hot tap water. If necessary, use a scrub brush to remove any remaining residue.

4.5.2. Place the cleaned glassware in the drying oven until dry or overnight.

4.5.3. Allow the cleaned and dry glassware to cool completely before use.

4.6. All Laboratory Groups

4.6.1. All glassware washing brushes must be hung on hooks when not in use to prevent contamination and ensure proper drying.

5. DEFINITIONS

5.1. Refer to the glossary in the Laboratory Quality Manual (LQM), latest version.

6. APPENDICES

6.1. See Table 1, Glassware Washing

6.2. Reagents

6.2.1. 1 M NaOH - 40±5 g sodium hydroxide dissolved in 1 L of reagent water.

6.2.2. 1 M H_2SO_4 - 55±5 mL concentrated sulfuric acid dissolved in 1 L of reagent water

6.2.3. Chromic acid – add a few milliliters of water to 40 g of potassium dichromate to make a paste, slowly add 600 mL of concentrated sulfuric acid while stirring.

Table 1 Glassware Washing

	Metals	Organics	Wet Chemistry
Wash	Hot Water, Detergent Solution	Hot Water, Detergent Solution	Hot Water Detergent Solution
Rinse	3 Times Tap Water 1 Time 1:1 Nitric Acid 3x Reagent Water	3 Times Tap Water 1 Time 1:1 Hydrochloric Acid* 3x Reagent Water	3 Times Tap Water 3x Reagent Water
Dry	Air	Muffle at 400°C for 1 hour	Air
Storage	Designated Cabinets & Shelves	Designated Cabinets & Shelves	Designated Cabinets & Shelves

*If the glassware is visibly dirty after washing in a hot detergent solution, the glassware is rinsed with 1:1 HCl and rewashed with a hot detergent solution.

Controlled Copy No. _____
Implementation Date _____

SOP No. CORP-QA-0010
Revision No. 2
Revision Date: 06/15/99
Page: 1 of 23

QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: NONCONFORMANCE AND CORRECTIVE ACTION SYSTEM

(SUPERSEDES: REVISION NO. 1)

Prepared by: Quanterra® QA Managers

Approved by: _____
Director of Quality Assurance

Approved by: _____
Director of Environmental Health and Safety

Approved by: _____
Vice President Operation Services

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1. PURPOSE

1.1. The purpose of this document is to establish procedures for the identification and documentation of nonconformances and the corrective actions taken as a result of these events. The Quanterra® Quality Assurance Management Plan (QAMP) requires documentation of instances of deviations from established control limits, approved standard operating procedure (SOPs), or client-specified requirements. The Nonconformance Memo (NCM) described in this procedure is used to document deviations from Quanterra® policies and procedures and documented client specifications including root causes and corrective actions taken to remedy the nonconformance. The NCM may be stored in electronic form in a database, or maintained using defined printed forms.

1.2. This document applies to procedures, services, analytical data, reports, or materials purchased by the laboratory or supplied by the laboratory to its clients. Nonconformances related to sample receiving activities may be documented separately from the NCM process by use of the Condition Upon Receipt (CUR) Form as described in a facility-specific SOP. Regardless of the type of system used to handle the sample receiving and client-related issues, including holding time violations (HTVs), the system must include and emphasize the immediate notification of the Project Manager (PM). This will allow the PM to initiate immediate client notification and resolution of how to proceed. See Section 5, Definitions, for further clarification of application.

1.3. Nonconformances can be identified by Quanterra® laboratory employees in the course of their daily operations or by external parties (i.e., customers and representatives of customers) through reviews of records, audit, or proficiency testing.

2. RESPONSIBILITIES

2.1. **Laboratory Associate:** During the course of their work, all employees are responsible for identifying and documenting problems, using a Nonconformance Memo, that might affect the quality of Quanterra's® product. They should also identify or attempt to seek out possible measures to correct the problem. By signing or initialing laboratory notebooks, forms, bench sheets, data reports, and other quality-related documents, associates are verifying that procedures have been followed. Any deviation that might render a measurement suspect shall be documented.

2.2. **Group Leader/Team Leader/Supervisor** (for purposes of this SOP, the term "Supervisor" will be used): Each supervisor is responsible for the review of NCMs to ensure that problems which might affect quality are adequately described and that personnel are assigned to correct them. Supervisors review hardcopy or electronic

versions of NCMs and forward them to the appropriate project manager. Together with project managers and Quality Assurance personnel, supervisors are responsible for determining the appropriateness of planned corrective actions.

2.3. **Project Manager (PM):** The project manager is responsible for relaying project requirements to staff so that special project requirements are understood and nonconformances recognized. The project manager communicates conformance problems to clients and documents decisions made with clients. The project manager ensures that short-term corrective actions for routine analytical QC failures are completed. An example would be making sure that repreparation and analysis of a sample was done. The project manager may withhold final reports to clients until corrective actions agreed to with the client have been completed.

2.4. **QA Manager:** The Quality Assurance manager or his or her designee is responsible for reviewing all initiated NCMs to ensure that actions taken are appropriate, and assisting in resolving QA/QC discrepancies. The QA staff will maintain a nonconformance tracking system to guarantee that each nonconformance is brought to closure. The system will also be used to monitor for trends that might indicate long-term quality problems. Systematic problems are investigated, NCMs issued and reviewed, and spot audits conducted to ensure that long-term corrective actions have been successfully completed. If review of an area reveals a significant problem with data quality, the Quality Assurance manager has the authority and responsibility to stop production in that laboratory area.

2.5. **Operations/Systems Manager :** The operations/systems manager shall ensure that corrective actions are correct and have been implemented. The operations/systems manager shall document this review and concurrence by signing the NCM as the responsible manager, if QA-required, for a specific corrective action. Along with the laboratory manager, the operations/systems manager shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work.

2.6. **Laboratory Manager:** The laboratory manager shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work. The laboratory manager is also responsible for the implementation of the NCM system in the laboratory.

2.7. **Corporate QA Director:** The Quanterra® Quality Assurance Director should be notified of any continuing nonconformances that are not properly addressed by operations or where the root cause cannot be identified.

3. SAFETY

3.1. Normal office dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.

3.2. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra® associates.

3.3. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra® associate. The situation must be reported **immediately** to a laboratory supervisor.

4. PROCEDURE

4.1. When to Initiate a Nonconformance Memo

4.1.1. Lab associates are to prepare a hard copy or initiate an electronic nonconformance memo (NCM) whenever procedures, services, data, reports, electronic disk deliverables (EDDs), or standard materials deviate from established specifications. All nonconformances with the exception of matrix-related failures require an NCM (see definitions of nonconformance, anomalies, and deficiencies in Section 5 and Section 1.2 for exceptions). ALL holding time violations (HTVs) require an NCM.

4.1.2. All standard operating procedures (SOPs) shall be followed. By signing or initialing laboratory notebooks, bench sheets, data reports, and other quality-related documents, employees are verifying that the SOPs have been followed with the exceptions of the pre-approved deviations (as described in QAPjPs Quality Assurance Summaries, or equivalent systems). Any intentional deviation from an SOP must be pre-approved by the QA manager and Supervisor. Any deviation from an SOP or client requirement not previously approved must be documented using the NCM process.

4.1.3. An NCM is to be completed for each instance of a nonconformance. A single NCM can be used for a single event affecting multiple lot numbers and samples, but normally a separate NCM would be initiated for different nonconformance issues. If the nonconformance involves projects for multiple project managers, then the NCM will need to be routed to each project manager.

4.2. Determining Which NCM Process to Use

4.2.1. Laboratories equipped with Quanterra's® standard Laboratory Information Management System (QuantIMS) shall use the NCM database application developed for this purpose, **Clouseau.**

4.2.2. Laboratories not equipped with QuantIMS shall use a paper-based NCM process or an equivalent electronic system.

4.3. How to Process the NCM using Clouseau

4.3.1. Initiating the NCM

4.3.1.1. While properly logged into a PC where Clouseau has been installed, start the Clouseau program.

4.3.1.2. At the Main Menu, select Create NCM.

4.3.1.3. Enter the information required at the top of the form. At a minimum select the Production Area, Classification (anomaly or deficiency), NCM Type, and NCM Description.

4.3.1.4. For NCMs that affected samples, indicate the relevant batch numbers, lots, samples, and tests by using the Tests/Samples option within Clouseau.

4.3.1.5. Complete the Comments/Details and Corrective Action sections of the Create NCM Form. These sections may be filled out jointly with the supervisor. Consult the project manager or the operations/systems manager and QA manager if the supervisor and associate are uncertain of corrective actions. Be objective and specific but brief. Include enough information that decisions to approve the NCM can be made easily (include pertinent QC information).

4.3.1.5.1. Design corrective actions to correct the immediate problem (short-term corrective actions) and to minimize the possibility of its recurrence (long-term corrective actions). Examples of corrective actions are modifications to nonconforming procedures, repair or replacement of deficient equipment, training personnel, and reanalysis of any affected samples.

4.3.1.5.2. Where operational corrective actions are required, they shall be supported with reference to recovery data, control charts, or other documentation.

4.3.1.6. If the corrective action involved retraining, the training must be documented with the signatures of the trainees and submitted to the QA staff before the NCM is considered closed.

4.3.1.7. Save the NCM using the appropriate command in Clouseau. When presented with the Send E-Mail form, the supervisor of the affected Production Area will be listed in the "Notify Now" box, while affected Project Managers and QA staff will be listed in the "Notify Later" box. Verify that these names are correct. If any personnel should be informed that are not listed, add their names to the "Notify Now" box by double-clicking on their names in the Personnel box.

4.3.1.8. Initiate the NCM notification process by selecting SEND EMAIL.

4.3.2. Supervisory Review and Approval

4.3.2.1. The supervisor will receive notification of the NCM via e-mail. The supervisor must log into a computer workstation where Clouseau has been installed, and run Clouseau.

4.3.2.2. Using the Review NCM form, select the NCM to be reviewed and click on OPEN.

4.3.2.3. With the NCM on screen, review the information provided. Add necessary comments or corrective actions to the appropriate areas. If the corrective action has not been determined, the situation must be referred to the project manager and the operations/systems manager for resolution to ensure client requirements can be satisfied. The QA staff should be consulted if there are questions as to how to proceed. If the above input was not needed, the operations/systems manager does not need to have every NCM routed to him or her. If, upon receipt and review of the NCM by the QA staff, it is felt the operations/systems manager needs to be made aware of the issues, the QA staff will notify the operations/systems manager using the Under Review/Send Email options of Clouseau.

4.3.2.4. If the nonconformance is hardware/equipment related, the item shall be nonconformance tagged and segregated, if possible, to ensure that it is not used until repaired. Refer to Section 4.4.4.8.

4.3.2.5. The supervisor will be responsible for the completion of the corrective action unless otherwise indicated. Enter the name of the person responsible for performing the corrective action if other than the supervisor. This is Operations' commitment to rectify the problem. The supervisor selects the "Approved" option on the Review NCM form and selects SAVE. This initiates the NCM routing process, which will now route the NCM to the project manager. The project manager must receive the NCM in a timely manner, generally within 48 hours. If the NCM is for a holding time violation, a project manager ***must*** be notified ***immediately***.

4.3.3. Project Manager Review, Client Notification, and Project Documentation

4.3.3.1. The project manager shall determine if client notification is required to either assist in the definition of corrective action or to notify the client of problems related to sample analysis. The project manager shall indicate using the Client Notification Form in Clouseau the date and method of notification and client's response .

4.3.3.2. With the NCM on screen, review the information provided. Add necessary comments or corrective actions to the appropriate areas. Notify the supervisor of any changes made to the corrective action plan.

4.3.3.3. The project manager selects the "Approved" option on the Review NCM form and selects SAVE. This initiates the NCM routing to the QA office. The QA office then must review the NCM in a timely manner, generally within 72 hours.

4.3.3.4. If the nonconformance involves analytical work in process, the **final** report cannot be released until a project manager has approved the NCM. If it is found that erroneous analytical data (e.g., from data validation comments or phone requests, etc.; inaccurate chromatograms, spectra, calculations, or final reports) have been released by the laboratory, this fact must be documented on an NCM and forwarded to the QA office. Prior to making the corrections, proper documentation shall be filled out and turned into the QA staff if corrections are needed in the database (QuantIMS). The original data shall be marked as unusable and maintained for historical purposes. The corrective action shall include prompt client notification and issuance of amended reports.

4.3.4. Quality Assurance Review and Trending

4.3.4.1. The QA staff shall review all NCMs for conformance with standard laboratory practices and Quanterra® policies.

4.3.4.2. NCMs will be reviewed to ensure that the corrective action was completed and effective in addressing the root cause of the nonconformity to prevent recurrence.

4.3.4.3. Clouseau's reporting and tracking system will be used to monitor for repetitive failures that might indicate systematic problems. Tracking records would (when applicable) include:

- NCM log number
- Date initiated
- Project number
- Lab sample ID numbers
- Method or parameter
- Nonconformance description
- Corrective action required
- Characterization as an anomaly or deficiency
- Closure of NCM

4.3.4.4. The QA staff shall identify repetitive quality issues that may be systematic in nature and may require corrective actions to prevent recurrence. Recurrent technical or Information Technology problems shall be referred to the appropriate technical group for corrective actions. Correction of systematic problems could take the form of modifications of nonconforming procedures, repair or replacement of deficient equipment, training or replacement of personnel. Findings and corrective actions from these investigations or audits shall also be documented. Resolution of corrective actions for systematic problems must be documented by the responsible laboratory area along with supporting evidence.

4.3.4.5. The QA staff shall conduct follow-up assessments to confirm that correction of systematic problems is successful. These assessments can be done as part of the monthly spot assessment program at the laboratory.

4.3.4.6. The approval of the QA manager or designee is required in Clouseau to indicate that the NCM has been closed.

4.3.4.7. The QA office may maintain a central file/record of all closed NCMs.

4.4. How to Process NCMs (Non-QuantIMS laboratories)

4.4.1. Initiate the NCM

4.4.1.1. Enter the information required at the top of the form (see Example Form in Attachment A; printed forms are available from QA). Names or initials must be uniquely identified by using three initials or by spelling out the last name. Use specific test codes (when available) rather than generic codes such as "VOAs" or "Anions." At a minimum enter Project ID, QuantIMS batch number or LIMS QC Lot number if appropriate, in addition to sample numbers. The laboratory generating the NCM is identified as part of the log number (e.g., AUS-97-123). Therefore, separate laboratory location identification is not required on the form.

4.4.1.1.1. If the NCM is for a holding time violation, the project manager ***must*** be notified immediately.

4.4.1.1.2. An instrument tag-out does not require the completion of the NCM if there are no client samples involved (e.g., instrument out of service to perform routine maintenance.) See section 4.2.5.

4.4.1.2. Check appropriate NCM boxes. If box 31—"Other"—is checked, supply a brief description of the problem. Attach further information as necessary.

4.4.1.3. Complete the reverse side of the form concerning root cause and corrective actions. These sections may be filled out jointly with the supervisor. Consult the project manager or the operations/systems manager and QA manager if the supervisor and associate are uncertain of corrective actions. Be objective and specific but brief.

Include enough information that decisions to approve the NCM can be made easily (include pertinent QC information).

4.4.1.3.1. Design corrective actions to correct the immediate problem (short-term corrective actions) and to minimize the possibility of its recurrence (long-term corrective actions). Examples of corrective actions are modifications to nonconforming procedures, repair or replacement of deficient equipment, training personnel, and reanalysis of any affected samples. Additional sheets documenting corrective action may be attached to the NCM. Reference the attachment on the NCM.

4.4.1.3.2. Where operational corrective actions are required, they shall be supported with reference to recovery data, control charts, or other documentation.

4.4.1.4. If the corrective action involved retraining, the training must be documented with the signatures of the trainees and submitted to the QA staff.

4.4.1.5. Instrument/Equipment Nonconformance Tag

4.4.1.5.1. Instruments and equipment which habitually fail to meet calibration criteria or are out of service due to needed repair or other reasons must be marked with a tag indicating the nonconforming condition. (See Example in Attachment B.)

4.4.1.5.2. If the reason for the nonconformance tag caused sample data to be impacted, initiate an NCM and mark box 30. Identify the instrument by name and/or identification number and briefly describe the problem. This information can be recorded on page 2 under "Problem Description/Root Cause". Submit the NCM to the group supervisor.

4.4.1.5.3. Upon receipt of the NCM, QA assigns the NCM number and provides the number to the analytical group so that the nonconformance tracking number can be recorded in the instrument's maintenance log.

4.4.1.5.4. The corrective action will be to either permanently remove the instrument from service or to have the instrument repaired. If an instrument is repaired, its reliability must be

demonstrated through successful recalibration before the nonconformance can be closed. The nonconformance tag remains in effect during the demonstration period. Record this information in the instrument maintenance logbook. Reference the successful calibration on the tag and return the tag to the QA staff for closure of the NCM.

4.4.1.6. Submit the form to the supervisor responsible for the area indicated at the top of the form.

4.4.2. Supervisory Review, Approval, and Signature

4.4.2.1. Review the information provided. Complete or amend the descriptions of root cause and corrective action. If the corrective action has not been determined, the situation must be referred to the project manager and the operations/systems manager for resolution to ensure client requirements can be satisfied. The QA staff should be consulted if there are questions as to how to proceed. If the above input was not needed, the operations/systems manager does not need to have every NCM routed to him or her. If, upon receipt and review of the NCM by the QA staff, it is felt the operations/systems manager needs to be made aware of the issues, the QA staff will notify and document the notification of the operations/systems manager on the form.

4.4.2.2. If the nonconformance is hardware/equipment related, the item shall be nonconformance tagged and segregated, if possible, to ensure that it is not used until repaired.

4.4.2.3. The supervisor will be responsible for the completion of the corrective action unless otherwise indicated. Enter the name of the person responsible for performing the corrective action if other than the supervisor. This is Operations' commitment to rectify the problem. This will be verified by the QA staff and/or the operations/systems managers.

4.4.2.4. The supervisor signs and dates the "Corrective Action Approved By" line and forwards the original form to the project manager. The project manager must receive the NCM in a timely manner, generally within 48 hours. If the NCM is for a holding time violation, a project manager ***must*** be notified ***immediately***.

4.4.3. Project Manager Review, Client Notification, and Project Documentation

4.4.3.1. The project manager shall determine if client notification is required to either assist in the definition of corrective action or to notify the client of problems related to sample analysis. The project manager shall indicate on the NCM whether client notification is required or not.

4.4.3.2. Record the result of the client contact or leave blank if client contact was not required. Sign and date the NCM form. This must be done whether client notification is completed or not. The signature documents the project manager as having read and reviewed the NCM.

4.4.3.3. Document any changes to the corrective action plan, and notify the supervisor. The project manager's signature on the front of the NCM serves as documentation that the PM has read and reviewed the NCM and is aware of the corrective action plan.

4.4.3.4. The project manager will forward the original to the QA office within 72 hours for review and closure. The PM may keep a copy for his or her own personal use, but the original must be forwarded to the QA office.

4.4.3.5. If the nonconformance involves analytical work in process, the **final** report cannot be released until a project manager has signed the NCM.

4.4.3.6. If it is found that erroneous analytical data (e.g., from data validation comments or phone requests, etc.; inaccurate chromatograms, spectra, calculations, or final reports) have been released by the laboratory, this fact must be documented on an NCM (items 25 and 26 on NCM form) and forwarded to the QA office. Prior to making the corrections, proper documentation shall be filled out and turned in to the QA staff if corrections are needed in the database (LIMS or QuantIMS). The original data shall be marked as unusable and maintained for historical purposes. The corrective action shall include prompt client notification and issuance of amended reports.

4.4.3.7. After the QA staff has closed the NCM, the original of the closed NCM will be filed either in the project file or QA files per laboratory procedure.

4.4.4. Quality Assurance Review and Tracking

4.4.4.1. The QA staff shall review all NCMs for conformance with standard laboratory practices.

4.4.4.2. NCMs will be reviewed to ensure that the corrective action was completed.

4.4.4.3. A tracking system will also be used to monitor for repetitive failures that might indicate systematic problems. Tracking records would (when applicable) include:

- NCM log number
- Date initiated
- Project number
- Lab sample ID numbers
- Method or parameter
- Nonconformance description and NCM box number
- Corrective action required
- Characterization as an anomaly or deficiency
- Closure of NCM

4.4.4.4. The QA staff shall identify repetitive quality issues that may be systematic in nature and may require corrective actions to prevent recurrence. Recurrent technical or Information Technology problems shall be referred to the appropriate technical group for corrective actions. Correction of systematic problems could take the form of modifications of nonconforming procedures, repair or replacement of deficient equipment, training or replacement of personnel. Findings and corrective actions from these investigations or audits shall also be documented. Resolution of corrective actions for systematic problems must be documented by the responsible laboratory area along with supporting evidence.

4.4.4.5. The QA staff shall conduct follow-up assessments to confirm that correction of systematic problems is successful. These assessments can be done as part of the monthly spot assessment program at the laboratory.

4.4.4.6. The signature of the QA manager or designee is required for corrective actions to indicate that the issue has been closed.

4.4.4.7. The QA office shall maintain a central file/record of all NCMs.

4.4.4.8. Instrument/Equipment Nonconformance Tag

4.4.4.8.1. Instruments and equipment which habitually fail to meet calibration criteria or are out of service due to needed repair or other reasons must be marked with a clearly visible tag or sign indicating the nonconforming condition. (See Example in Attachment B.)

4.4.4.8.2. If the reason for the nonconformance tag caused sample data to be impacted, initiate an NCM (electronic or hard copy). Identify the instrument by name and/or identification number and briefly describe the problem.

4.4.4.8.3. Upon saving the NCM, the NCM number indicated by Clouseau shall be recorded in the instrument's maintenance log.

4.4.4.8.4. The corrective action will be to either permanently remove the instrument from service or to have the instrument repaired. If an instrument is repaired, its reliability must be demonstrated through successful recalibration before the nonconformance can be closed. The nonconformance tag remains in effect during the demonstration period. Record the back to control information in the instrument maintenance logbook. Reference the successful calibration on the tag and return the tag to the QA staff for closure of the NCM.

5. DEFINITIONS

5.1. ***Nonconformance:*** an unplanned deviation from an established protocol or plan. The deviation may be the result of Quanterra's® actions, then termed a **deficiency**, or the result of events beyond the control of Quanterra®, then termed an **anomaly**.

A nonconformance exists when:

5.1.1. Any laboratory QC sample (e.g., method blank, laboratory control sample, duplicate laboratory control sample, matrix spike, matrix spike duplicate, and surrogate spike) component result is outside established control limits and demonstrate a **systematic** deficiency. Any matrix spike or matrix spike duplicate or sample related QC outside of established control limits attributed

to matrix effects must be documented, but an NCM is not required. A procedure is not performed as described in the applicable SOP or QA Policy, ***except*** in cases where the procedure has been performed according to a client-specified document Quanterra® has agreed to follow (e.g., EPA SOWs and QAPjPs).

5.1.2. A practice or procedure is not performed as described according to a client or project document that Quanterra® has agreed to follow.

5.1.3. Purchased materials or services are determined to be defective and their use would affect data quality.

5.1.4. ANY holding time violations (HTVs) occurs regardless of what or whose actions caused them including ACTS of GOD.

5.1.5. A formal NCM is not required for routine minor instrument maintenance, malfunctions, and power failures which can be documented in instrument maintenance logbooks.

5.2. ***Corrective action:*** Measures taken to rectify conditions adverse to quality and, where possible, to prevent their reoccurrence.

5.2.1. Corrective actions may vary from reporting the data as is—with appropriate documentation—to a complete reevaluation and restructure of a system.

5.2.2. Many corrective actions can be implemented immediately; however, some will take time to implement.

6. MISCELLANEOUS

6.1. Associated Reference Documents

6.1.1. Quanterra® Quality Assurance Management Plan (QAMP), current revision.

6.1.2. ANSI/ASME NQA-1, Chapter II, Basic Requirement 15 "Control of Nonconforming Items." Supplement 15S-1 "Supplementary Requirements for the Control of Nonconforming Items."

6.1.3. ANSI/ASQC Q94-1987, "Quality Management and Quality System Elements - Guidelines," Section 14.0 "Nonconformity" and Section 15.0 "Corrective Action."

6.1.4. Clouseau Program Documentation.

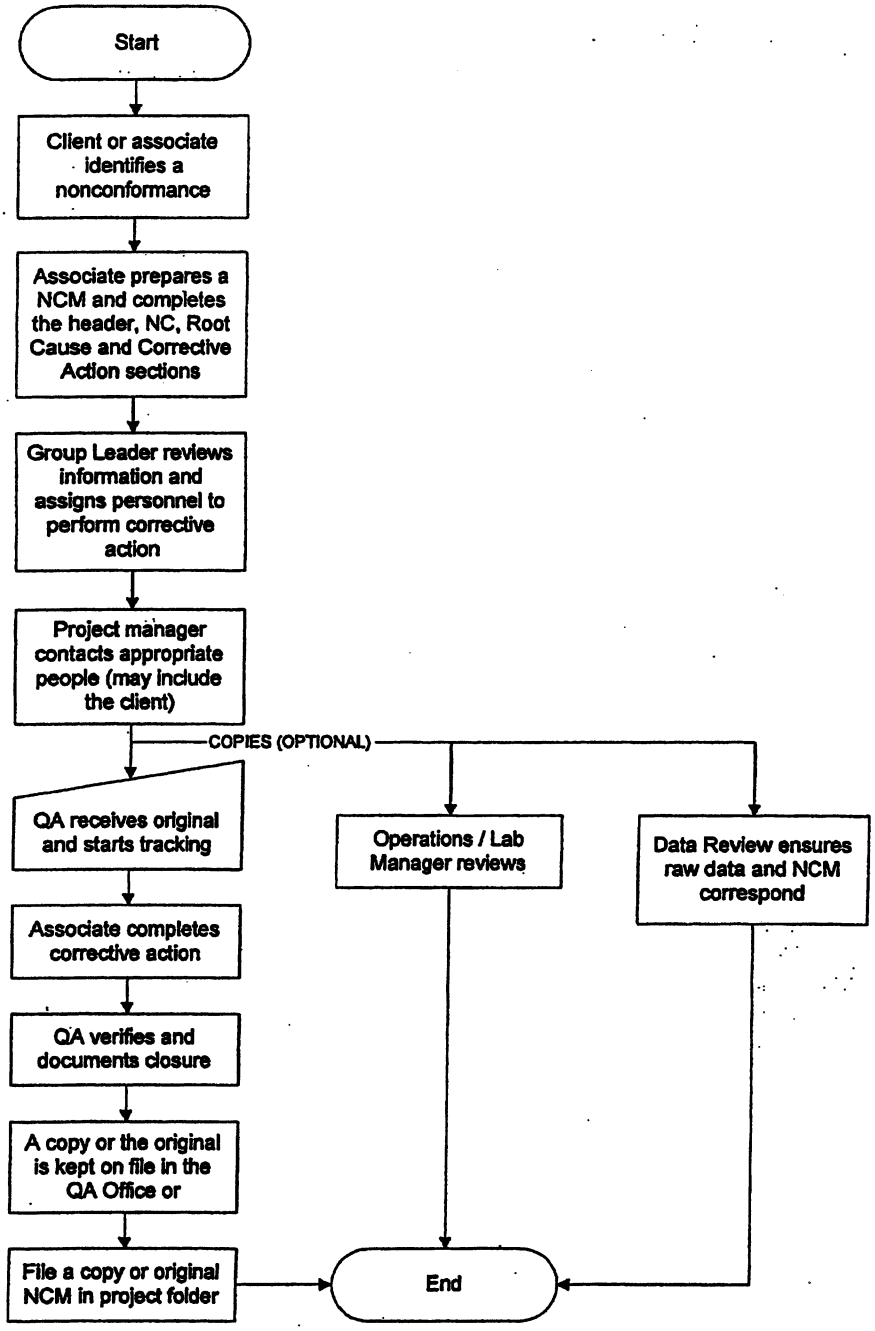
6.2. Appendices

6.2.1. Attachment A: Laboratory Nonconformance Memo (NCM).

6.2.2. Attachment B: Instrument/Equipment Nonconformance Tag Form

6.2.3. Attachment C: Nonconformance Memo Generated from Clouseau

6.3. Flow Chart for Internal NCM



ATTACHMENT A LOG #: _____

LABORATORY NONCONFORMANCE MEMO (NCM)

Quanterra® Incorporated

Project ID/Client: _____

NCM Initiated by/Date: _____ Project Manager: _____

Sample Numbers/QC batch or lot numbers _____

Tests: _____

Analytical Area (check appropriate area):

- ☐ Sample control
- ☐ Organic preparation
- ☐ Inorganic preparation
- ☐ GC
- ☐ HPLC
- ☐ GC/MS
- ☐ Wet chemistry
- ☐ Metals
- ☐ Reporting
- ☐ Data review
- ☐ Radiochemistry
- ☐ _____

Nonconformance (check appropriate area): *To be completed by analyst*

Holding Time Violations (exceeded by _____ days)

Category I: Laboratory Independent

- ☐ 1. Holding time expired in transit
- ☐ 2. Sample rec'd > 48 hrs after sampling. or ½ holding time has expired
- ☐ 3. Test added by client after expiration

Category II: Laboratory Dependent

- ☐ 4. Instrument failure
- ☐ 5. Analyst error
- ☐ 6. Log-in error
- ☐ 7. Miscommunication
- ☐ 8. Other (explanation required)_____

_____

Category III: Analysis Reruns (QA/QC)

- ☐ 9. Surrogates
- ☐ 10. Internal standards
- ☐ 11. Spike recoveries
- ☐ 12. Blank contamination

Category IV: Analysis Reruns (Confirmation)

- ☐ 13. Second column
- ☐ 14. Contamination check
- ☐ 15. Confirmation of matrix effects
- ☐ 16. Other (explanation required)_____

Category V: Analysis Reruns (Dilution)

- ☐ 17. Over calibration
- ☐ 18. Under calibration
- ☐ 19. Other (explanation required)_____

Quality Assurance/Quality Control

- ☐ 20. QC data reported outside of controls
- ☐ 21. Incorrect procedure used
- ☐ 22. SOP intentionally modified with QA and tech approval
- ☐ 23. Invalid instrument calibration
- ☐ 24. Received insufficient sample for proper analysis

Incorrect or Incomplete Client Deliverable

- ☐ 25. Hardcopy deliverable error
- ☐ 26. Electronic deliverable error

Reported Detection Limits Elevated Due to:

- ☐ 27. Sample matrix: Does not include high analyte content
- ☐ 28. Insufficient sample volume
- ☐ 29. Other (explanation required)

Miscellaneous

- ☐ 30. Instrument/equipment Tag-out
- ☐ 31.Other (explanation required)

_____

_____

_____

_____

Notification (check appropriate area): ☐ **Required** ☐ **Not Required** *To be completed by project manager*

Client notified by (name and date): _____

- ☐ In writing
- ☐ By facsimile
- ☐ By telephone
- ☐ Other (explain)

Client's name and response: _____

- ☐ Process "as is"
- ☐ Re-sample
- ☐ On hold until _____
- ☐ Other (explain)

Project manager (signature and date): _____

QUANTERRA® LABORATORY NONCONFORMANCE MEMO LOG #: _____

Corrective Action: *To be completed and reviewed by all associates involved*

Problem Description/Root Cause Author's initials and date: _____

Corrective Actions (Short Term) Author's initials and date: _____

Corrective Actions to Prevent Reoccurrence (Long Term) _____

Corrective Action approved by (Supervisor/Group Leader) and date: _____

Additional Comments: _____

Corrective Action to be completed by (if other than Supervisor/Group Leader): _____

Date Corrective Action is to be completed: _____

❑ Notified Ops/Sys Manager (Initials) _____

The Office of Quality Assurance maintains a copy or the original of this NCM indicating its final status.

ATTACHMENT B

EXAMPLE

INSTRUMENT/EQUIPMENT NONCONFORMANCE TAG FORM

QUANTERRA®

CAUTION

DO NOT USE

NONCONFORMING ITEM

NCM NUMBER _____

AFFECTED ITEM _____

_____

_____

_____ _____

ANALYST DATE

WORK MAY NOT PROCEED ON THIS ITEM UNTIL SUCCESSFUL CALIBRATION IS DOCUMENTED.

ATTACHMENT C

EXAMPLE

LABORATORY NONCONFORMANCE MEMO (GENERATED BY CLOUSEAU)

Quanterra

Clouseau

Nonconformance Memo

NCM #: E00053
NCM Initiated By: Kevin Freitag
Date Opened: 03/10/99
Date Closed: 03/18/99

Classification: Anomaly
Status: CLOSED
Production Area: Sample Control
Tests: 8260B
Lot #'s (Sample #'s): E9C100234 (9)
QC Batch: None.

Nonconformance: Sample receiving issues
Subcategory: Samples received did not match the COC

Problem Description / Root Cause

Name	Date	Description
Kevin Freitag	03/10/99	Sample was received but was not listed on COC. Sample was logged in as E9C100234-009.

Corrective Action

Name	Date	Corrective Action

Quality Assurance Verification

Verified By	Due Date	Status	Notes:
Sevdá Aleckson	N/A	Client approved, verification not required	

Client Notification Summary

Client	Project Manager	Date Notified	Response Date	How Notified
ARCADIS GERAGHTY & MILLER	Diane Suzuki	03/18/99	03/18/99	in writing

Response: No response saved
Response Details:

Approval History

Name	Date Approved:	Position
Kevin Freitag	03/10/99	Chemist
Linnea Coffey	03/18/99	Project Manager
Diane Suzuki	03/18/99	Project Manager
Sevdá Aleckson	03/18/99	Quality Assurance

Date Printed: 06/07/99

Page 1 of 1

Controlled Copy
Copy No:_____

Implementation Date: 08/24/00

SOP No: NC-WC-0004
Revision No: 3
Revision Date: 08/04/00

STL STANDARD OPERATING PROCEDURE

TITLE: TOTAL SOLIDS, PERCENT MOISTURE, ASH AND TOTAL VOLATILE SOLIDS

(SUPERSEDES: REVISION 2.0)

Prepared by:		8-2-00 Date
Reviewed by:	Technology Specialist	8-7-00 Date
Approved by:	Quality Assurance Manager	8-4-00 Date
Approved by:	Environmental Health and Safety Coordinator	8-13-00 Date
Approved by:	Laboratory Director	8-17-00 Date

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Total Solids, Total Volatile Solids, Percent Moisture, and Ash in waters, wastewaters, sludges, and solids. It is based on EPA 160.3, EPA 160.4, ASTM D2216-90, ASTM D1553-83, SM 2540E, USEPA CLP Statements of Work OLM01.9, OLM03.1, ILM04.0 and ILM03.0. The approximate working range for Total Solids is 10 - 20,000 mg/L for waters and 0.5 to 100% for non-waters.

1.2. The applicable method codes are SM (160.3), AN (160.4) , NJ (D2216-90), ES (OLM03.1), DS (ILM03.0), WN (OLM01.9), OV (ILM04.0) and H9 (SM2540E). The associated preparation code is 88.

1.3. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

2. SUMMARY OF METHOD

2.1. A homogenous sample is dried at 104 °C ± 1°C and the difference in the weight loss of the sample (non-water) or the weight gain of the vessel (water) represents the Total Solids.

3. DEFINITIONS

3.1. Refer to the glossary in the Quality Assurance Management Plan (QAMP).

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Samples high in concentrations of minerals must be dried longer, desiccated, and weighed quickly to prevent any excess weight added due to absorption of water from the atmosphere.

4.3. Non-homogeneous samples may give erratic results.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Analytical balance: capable of accurately weighing ± 0.0001 g

6.2. Drying Oven

6.3. Desiccators

6.4. Evaporating dishes: various

6.5. Tongue blades

6.6. Graduated cylinders: various

6.7. Sample containers

6.8. Top loading balance: capable of accurately weighing ± 0.01 g

6.9. Analytical balance: capable of accurately weighing ±0.0001 g

6.10. Aluminum pans

6.11. Beakers: glass, various

6.12. Muffle furnace

6.13. Conductivity meter

6.14. Volumetric flasks: various

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Reagent water

7.2. Standards

7.2.1. Sodium Chloride (NaCl): reagent grade

7.2.2. Total Solid Standard, 2,500 mg/L: Add 2.5 g sodium chloride (NaCl) to a 1000 mL volumetric flask and dilute to volume with reagent water. Mix well. Replace after three months or as needed.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored in plastic or glass containers at 4°C ± 2°C.

8.3. The holding time is seven days from sampling to the completion of analysis for water samples.

8.4. There is no recommended holding time for non-water samples.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.2. Corrective Action for Blanks

9.2.2.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.2.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS) (Water Samples Only)

9.3.1. One aqueous LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. A LCS consisting of 25 mL of the 2500 mg/L TS standard is analyzed with each analytical batch of samples.

9.3.3. Corrective Action for LCS

9.3.3.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.3.2. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Duplicates

9.4.1. Sample duplicates are performed at a frequency of 10% and must meet laboratory-specific limits for precision.

10. CALIBRATION AND STANDARDIZATION

10.1. Not Applicable

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Vessel Preparation - Water Samples

11.3.1. Place glass beakers or evaporating dishes in the drying oven (103°C - 105°C) for at least one hour and in the desiccator for at least one hour.

11.4. Sample Analysis - Water Samples

11.4.1. Label and accurately weigh the beaker or evaporating dish on an analytical balance to the nearest 0.1 mg. Record weight on the analytical logsheet.

11.4.2. Tare the vessel and add approximately 100 mL of the homogenized sample. Record the volume of the sample on the analytical logsheet.

NOTE: If the sample is known to contain > 2000 mg/L dissolved solids, it should be diluted. A prescreening may be performed using a conductivity meter in which any results >2000 umhos should require a dilution.

11.5. To determine proper dilutions:

$$\frac{2000 \times 100}{Conductivity} = mL\ Sample\ to\ Use$$

11.5.1. Place the vessel with sample in the drying oven (103° - 105°C) overnight or until dry and then place the vessel in the desiccator for at least one hour.

11.5.2. If the sample was not dried overnight, place the dish in the oven for one hour, the desiccator for one hour, and reweigh. Continue this process until a constant weight is reached, until loss of weight is less than 4% of the previous weight, or 0.5 mg, whichever is less.

11.5.3. If a TVS or Ash is required, go to Section 11.7

11.6. Non-water Samples

11.6.1.. For liquid, sludge, TVS and ash samples, label and weigh an evaporating dish on a top loading balance. Record the weight on the analytical logsheet. For solid samples (TS), the tare weight is 5.652 For solid samples, sample receiving will divide the soil sample into a pre-tared container with a label. Weigh and record the wet weight on the analytical logsheet or the computer spreadsheet. For liquids and sludges, transfer approximately 50 mL to the tared evaporating dish then weigh and record the actual weight on the analytical logsheet.

11.6.2. Place dishes with sample in the drying oven (103° - 105°C) until dry (minimum of 12 hours). Document the time samples were placed in the oven on the logsheet or spreadsheet.

11.6.3. Remove the samples from the drying oven when they are dry. Document the time samples were removed from the oven on the logsheet or spreadsheet.

11.6.4. Place the dishes in the desiccator for at least one hour. Weigh the sample and dish on the top loading balance. Record the weight on the analytical logsheet.

NOTE: If the drying time was not overnight, it may be necessary to repeat the drying, desiccating, and weighing cycle until a constant weight is achieved as described in Section 11.5.2.

11.6.5. Method Blank for solid samples (TS), use a new container with a label. Weigh and record the weight on the analytical logsheet or computer spreadsheet.

11.6.6. Sample Duplicate for solid samples (TS), sample receiving will supply the sample duplicate. If the sample duplicate is not supplied from receiving, split some sample off from another container into a new container with a label. Weigh and record the weight on the analytical logsheet.

11.6.7. If a Total Volatile Solid or Ash is required, go to Section 11.7. All other samples may be properly disposed in the solid hazardous waste container.

11.7. Total Volatile Solids (TVS) and Ash Samples

11.7.1. Follow Section 11.6 for non-water samples

11.7.2. After the final dried weight has been taken, place the evaporating dish and dried sample in the muffle furnace. Muffle the sample for one hour at 550°C ± 50°C.

NOTE: Open the oven slowly and allow to cool for at least one hour before removing the samples.

11.7.3. Remove the dish with sample and place it in the desiccator for at least one hour, then weigh it on the top loading balance. Record the weight on the analytical logsheet or computer spreadsheet.

11.8. Percent Moisture

11.8.1. Follow Section 11.6

11.9. Analytical Documentation

11.9.1. Record all analytical information in the analytical logbook/logsheet or computer spreadsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.9.2. All standards are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.9.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.9.4. Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Total Solids

$$\textit{Total Solids, mg/L (waters)} = \frac{(A - B) \times 1000 \times 1000}{C}$$

Where: ***A =*** ***Final weight of dried sample and dish, g***

B = ***Initial weight of dish, g***

C = ***Sample volume, mL***

D = ***Sample weight, g***

$$\textit{Total Solids, \% (non-waters)} = \frac{(A - B) \times 100}{(C - B)}$$

Where: ***A = Final weight of dried sample and dish, g***

B = ***Initial weight of dish, g***

C = Inital weight of wet sample and dish, g

12.2. Ash

$$\textit{Ash, mg / L (waters)} = \frac{(D - B) \times 1000 \times 1000}{\textit{Sample Volume, mL}}$$

$$\textit{Ash, \% (non - waters)} = \frac{(D - B) \times 100}{A - B}$$

Where: ***A = Weight of dried sample and dish (after Total Solid), g***

B = Weight of dish only, g

C = Weight of wet sample and dish, g

D = Weight of muffled sample and dish, g

12.3. Total Volatile Solids (TVS)

$$\frac{(A - D) \times 1000 \times 1000}{\text{Sample Volume, mL}} = \text{TVS, mg / L (waters)}$$

$$\frac{(A - D) \times 100}{(A - B)} = \text{TVS, \% (non - waters)}$$

Where: *A = Weight of dried sample and dish (after Total Solid), g*

B = Weight of dish only, g

C = Weight of wet sample and dish, g

D = Weight of muffled sample and dish, g

12.4. LCS % Recovery

$$\text{LCS \% Recovery} = \frac{\text{mg / L}}{\text{2,500 (true)}} \times 100$$

12.5. Dry Weight

$$\text{Dry Weight} = \frac{\text{Sample Test Result} \times 100}{\text{(\%) Total Solid Results}} = \text{Dry Weight}$$

12.6. Percent Moisture

$$\text{\%Moisture} = 100 - \text{\%TS (Section 12.1)}$$

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Solid materials (gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.2. Refer to the Laboratory Sample and Waste Disposal plan.

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1. References

16.1.1. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition, Solids, Method 2540E.

16.1.2. EPA 600, 1984, Total Residual Solids, Method 160.3

16.1.3. EPA 600, Total Volatile Residue, Method 160.4

16.1.4. Annual Book of ASTM Standards, Volume 04.08, 1990

16.1.5. USEPA Contract Laboratory Program, Statement of Work for Inorganics Analysis, Multi-media Multi-concentration, ILM02.1, ILM03.0, and ILM04.0

16.1.6. USEPA Contract Laboratory Program, Statement of Work for Organics Analysis, Multi-media Multi-concentration, OLM01.9 and OLM03.1

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Reporting limits

17.1.1. The lower reporting limit (RL) for Total Solids, TVS, and Ash is 10 mg/L for water and 0.5% for non-waters. The reporting limit for Method 160.3 is 10%.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

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Implementation Date: 06/05/01

SOP No. CORP-MS-0002NC
Revision No. 2.3
Revision Date: 05/23/01

STL STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF VOLATILE ORGANICS BY GC/MS BASED ON METHOD 8260B, 8260A, AND 624

(SUPERSEDES: REVISION 2.2, DATED 11/28/00)

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ANALYSIS OF VOLATILE ORGANICS BY METHOD 624 55

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1.0 SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Volatile Organic Compounds in waters, wastewater, soils, sludges and other solid matrices. Standard analytes are listed in Tables 5 and 6.

1.2. This SOP is applicable to method 8260B. It may also be used for analysis following method 8260A. Appendix A presents modifications to the procedures in the main SOP that are necessary for analysis of wastewater by method 624. The associated LIMS method codes are QK (8260B), DN (624), and MZ (8260A). Ohio VAP projects are distinguished by Program Code 2J. The following Prep Codes are used: 15 (5 mL purge), 25 (25 mL purge), 4B (Methanol preservation, EnCore™), 4D (Sodium Bisulfate preservation, EnCore™), 4P (Frozen, EnCore™), and 73 (5030A Methanol Prep).

1.3. This method can be used to quantify most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4. The method is based upon a purge and trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 5 to 200 µg/L for 5 mL waters, 1 to 40 µg/L for 25 mL purge waters, 5 to 200 µg/kg for low-level soils, and 250 to 25,000 µg/kg for medium-level soils. Reporting limits are listed in Tables 1 and 3.

1.5. Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates, and laboratory control spike samples.

2. SUMMARY OF METHOD

2.1. Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2. Aqueous samples are purged directly. Generally, soils are preserved by extracting the volatile analytes into methanol. If especially low detection limits are required, soil samples may be preserved with sodium bisulfate and purged directly.

2.3. In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature or at 40°C (40°C required for low level soils) and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbant

column where the volatile components are trapped. After purging is completed, the sorbant column (trap) is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components which are detected with a mass spectrometer.

2.4. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3. DEFINITIONS

3.1. Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. Using this method, each BFB analysis will normally start a new batch. Batches for medium level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort should be made to keep the samples together.

3.1.1. The Quality Control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. Refer to the STL QC Program document (QA-003) for further details of the batch definition.

3.2. Method Blank

3.2.1. A method blank consisting of all reagents added to the samples must be analyzed with each batch of samples. The method blank is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

3.3. Laboratory Control Sample (LCS)

3.3.1. Laboratory Control Samples are well characterized, laboratory generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

3.4. Surrogates

3.4.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Each sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

3.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

3.5.1. A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second aliquot of the same sample which is prepared and analyzed along with the sample and matrix spike. Matrix spikes and duplicates are used to evaluate accuracy and precision in the actual sample matrix.

3.6. Calibration Check Compound (CCC)

3.6.1. CCCs are a representative group of compounds which are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and % drift for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.7. System Performance Check Compounds (SPCC)

SPCCs are compounds which are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, pre-purged purified reagent water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants. In extreme cases the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

4.3. Matrix interferences may be caused by non-target contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.5. Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered an antifoaming agent (e.g., J.T. Baker's Antifoam B silicone emulsion) can be used. A blank spiked with this agent must be analyzed with the sample because of the non-target interferences associated with the agent.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

5.2. The Chemical Hygiene Plan (CHP) gives details about the specific health and safety practices which are to be followed in the laboratory area. Personnel must receive training in the CHP, including the written Hazard Communication plan, prior to working in the laboratory. Consult the CHP, the STL Health and Safety Policies and Procedures Manual, and available Material Safety Data Sheets (MSDS) prior to using the chemicals in the method.

5.3. Consult the STL Health and Safety Policies and Procedures Manual for information on Personal Protective Equipment. Eye protection that protects against splash and a laboratory coat must be worn in the lab. Appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately. Disposable gloves shall not be reused.

5.4. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined, therefore each chemical compound should be treated as a potential health hazard. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

5.4.1. Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include: Acrylonitrile, benzene, carbon tetrachloride, chloroform, 1,2-dibromo-3-chloropropane, 1,4-dichlorobenzene, and vinyl chloride.

5.4.2. Chemicals known to be flammable are: **Methanol.**

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor.

5.8. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the STL Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed by an annual refresher training.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes: 10 µL and larger, 0.006 inch ID needle.

6.2. Syringe: 5 or 25 mL glass with luerlok tip, if applicable to the purging device.

6.3. Balance: Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g

6.4. Glassware:

6.4.1. Vials: 20 mL with screw caps and Teflon liners.

6.4.2. Volumetric flasks: 10 mL and 100 mL, class A with ground-glass stoppers.

6.5. Spatula: Stainless steel.

6.6. Disposable pipets: Pasteur.

6.7. pH paper: Wide range.

6.8. Gases:

6.8.1. Helium: Ultra high purity, gr. 5, 99.999%.

6.8.2. Nitrogen: Ultra high purity, from cylinders of gas generators, may be used as an alternative to helium for purge gas.

6.8.3. Compressed air: Used for instrument pneumatics.

6.8.4. Liquid nitrogen: Used for cryogenic cooling if necessary.

6.9. Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.

6.9.1. Sample Purger: The recommended purging chamber is designed to accept 5 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low level soils are purged directly from a VOA vial.

6.9.2. Trap: A variety of traps may be used, depending on the target analytes required. For most purposes the Vocarb 3000 trap is suitable. Other traps, such as Vocarb 4000, or Tenax / Silica gel / Charcoal may be used if the Quality Control criteria are met.

6.9.3. Desorber: The desorber should be capable of rapidly heating the trap to 180°C. Many such devices are commercially available.

6.9.4. Sample Heater: A heater capable of maintaining the purge device at 40°C is necessary for low level soil analysis.

6.10. Gas Chromatograph/Mass Spectrometer System:

6.10.1. Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming.

6.10.2. Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:

6.10.2.1. Column 1: 105m x 0.53 ID Rtx-624 with 3 µm film thickness.

6.10.2.2. Column 2: 75 m x 0.53 ID DB-624 widebore with 3 µm film thickness.

6.10.2.3. Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 AMU every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-Bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.10.3. GC/MS interface: In general glass jet separators are used but any interface (including direct introduction to the mass spectrometer) that achieves all acceptance criteria may be used.

6.10.4. Data System: A computer system that allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as

the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

6.10.5. Cryogenic Cooling: Some columns require the use of liquid nitrogen to achieve the subambient temperature required for the proper separation of the gases.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Methanol: Purge and Trap Grade, High Purity

7.1.2. Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See section 9.4) Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable.

7.2. Standards

7.2.1. Calibration Standard

7.2.1.1. Stock Solutions: Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at -10° to -20°C.

7.2.1.2. Working standards: A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer or as recommended by the manufacturer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by more than 20% then corrective action is necessary. This may include steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem then a new initial calibration must be performed.

7.2.1.3. Aqueous Calibration Standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

7.2.1.4. If stock or secondary dilution standards are purchased in sealed ampoules they may be used up to the manufacturers expiration date.

7.2.2. Internal Standards: Internal standards are added to all samples, standards, and blank analyses. Refer to Table 7 for internal standard components.

7.2.3. Surrogate Standards: Refer to Table 8 for surrogate standard components and spiking levels.

7.2.4. Laboratory Control Sample Spiking Solutions: Refer to Table 9 for LCS components and spiking levels.

7.2.5. Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Table 9.

7.2.6. Tuning Standard: A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration of 25 ng/µL of 4-Bromofluorobenzene in methanol is prepared as described in Sections 7.2.1.1 and 7.2.1.2.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample collection.

8.2. Water samples are normally preserved at pH $\leq$ 2 with 1:1 hydrochloric acid. If residual chlorine is present, 2 drops of 10% sodium thiosulfate are added.

8.3. Solid samples are field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. At specific client request, unpreserved soil samples may be accepted.

8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take an EnCore™ sample. (The 5 g or 25 g sampler can be used, depending on client preference). Following shipment back to the lab the soil is preserved in methanol. This is the medium level procedure. If very low detection limits are needed (< 50 µg/kg for most analytes) then it will be necessary to use two additional 5 g EnCore™ samplers or to use field preservation.

8.5. Sample collection for medium level analysis using EnCore™ samplers.

8.5.1. Ship one 5 g (or 25 g) EnCore™ sampler per field sample position.

8.5.2. An additional bottle must be shipped for percent moisture determination.

8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25 g) sample into a tared VOA vial containing 5 mL methanol (25 mL methanol for the 25 g sampler). Obtain the weight of the soil added to the vial and note on the label.

8.5.4. Add the correct amount of surrogate spiking mixture. (Add 25 µL of 2500 µg/mL solution for a nominal 25 g sample, 5 µL for a nominal 5 g sample.) Refer to Section 17.8 for Michigan project criteria.

8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 500 µL of 50 µg/mL solution for a nominal 25 g sample, 100 µL for a nominal 5 g sample.) Reduce the volume of methanol added to ensure the final volume is 25 mL for nominal 25 g sample or 5 mL methanol for a nominal 5 g sample. Refer to Section 17.8 for Michigan project criteria.

8.5.6. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (50 µL of spike to 25 mL methanol or 10 µL spike to 5 mL methanol). Refer to Section 17.8 for Michigan project criteria.

8.5.7. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.5.8. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4 ±2°C until analysis.

8.6. Sample collection for medium level analysis using field methanol preservation

8.6.1. Prepare a 2 oz sample container by adding 25 mL purge and trap grade methanol. (If a 5 g sample is to be used, add 5 mL methanol to a 2 oz container or VOA vial).

8.6.2. Seal the bottle and attach a label.

8.6.3. Weigh the bottle to the nearest 0.01g and note the weight on the label.

8.6.4. Ship with appropriate sampling instructions.

8.6.5. Each sample will require an additional bottle with no preservative for percent moisture determination.

8.6.6. At client request, the methanol addition and weighing may also be performed in the field.

8.6.7. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.6.8. Add the correct amount of surrogate spiking mixture. (Add 25 µL of 2500 µg/mL solution for a nominal 25 g sample, 5 µL for a nominal 5 g sample.) Refer to Section 17.8 for Michigan project criteria.

8.6.9. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 25 µL of 50 µg/mL solution for a nominal 25 g sample, 100 µL for a nominal 5 g sample.) Reduce the volume of methanol added to ensure the final volume is 25 mL for nominal 25 g sample or 5 mL methanol for a nominal 5 g sample. Refer to Section 17.8 for Michigan project criteria.

8.6.10. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (500 µL of spike to 25 mL methanol or 100 µL spike to 5 mL methanol). Refer to Section 17.8 for Michigan project criteria.

8.6.11. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.6.12. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4+2°C until analysis.

8.7. Low level procedure

8.7.1. If low detection limits are required (typically < 50 µg/kg) sodium bisulfate preservation must be used. However, it is also necessary to take a sample for the medium level (field methanol preserved or using the EnCore™ sampler) procedure, in case the concentration of analytes in the soil is above the calibration range of the low level procedure.

8.7.2. A purge and trap autosampler capable of sampling from a sealed vial is required for analysis of samples collected using this method. (Varian Archon or O.I. 4552).

8.7.3. The soil sample is taken using a 5g EnCore™ sampling device and returned to the lab. It is recommended that two EnCore™ samplers be used for each field sample position, to allow for any reruns than may be necessary. A separate sample for % moisture determination is also necessary.

8.7.4. Prepare VOA vials by adding a magnetic stir bar, approximately 1 g of sodium bisulfate and 5 mL of reagent water.

8.7.5. Seal and label the vial. It is strongly recommended that the vial is labeled with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction. The label absolutely must not cover the neck of the vial or the autosampler will malfunction.

8.7.6. Weigh the vial to the nearest 0.1g and note the weight on the label.

8.7.7. Extrude the soil sample from the EnCore™ sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil and note on the label.

Note: Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case at a specific site, add 5 mL of water instead, and freeze at <-10°C within 48 hours, analyzed within 12 days after preserving with water, and stored at a 45 degree angle in the freezer.

Note: Freezing is not allowed for Ohio VAP soil samples.

8.7.9. Alternatively the sodium bisulfate preservation may be performed in the field. This is not recommended because of the many problems that can occur in the field setting. Ship at least two vials per sample. The field samplers must determine the weight of soil sampled. Each sample will require an additional bottle with no preservative for percent moisture determination, and an additional bottle preserved with methanol for the medium level procedure. Depending on the type of soil it may also be necessary to ship vials with no or extra preservative.

8.8. Unpreserved soils

8.8.1. At specific client request unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the lab. This is the old procedure based on method 5030A and method 8260A. It is no longer included in SW846 and is likely to generate results that are biased low, possibly be more than an order of magnitude.

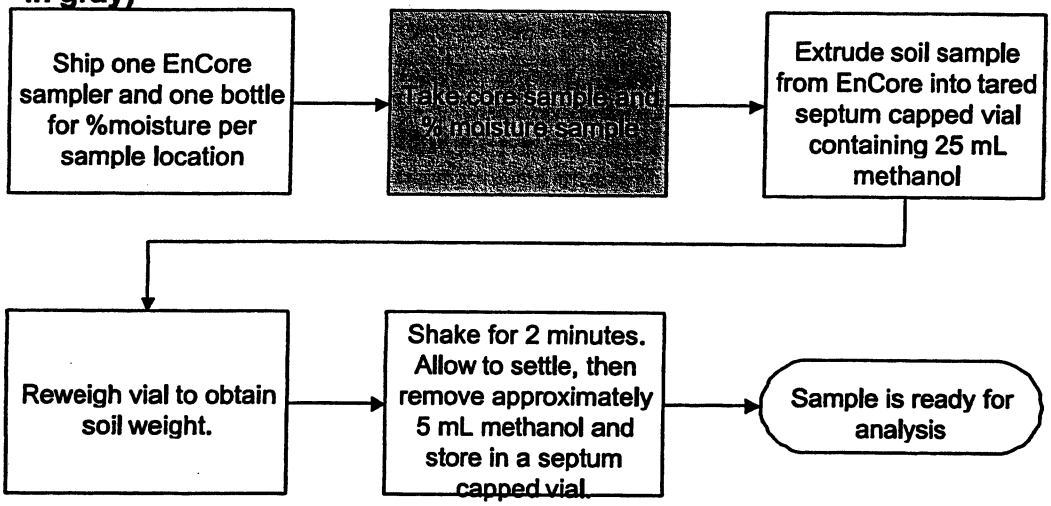
8.9. Aqueous samples are stored in glass containers with Teflon lined septa at 4°C +/- 2°C, with minimum headspace.

8.10. Medium level solid extracts are aliquoted into 2 - 5 mL glass vials with Teflon lined caps and stored at 4°C +/- 2°C. The extracts are stored with minimum headspace.

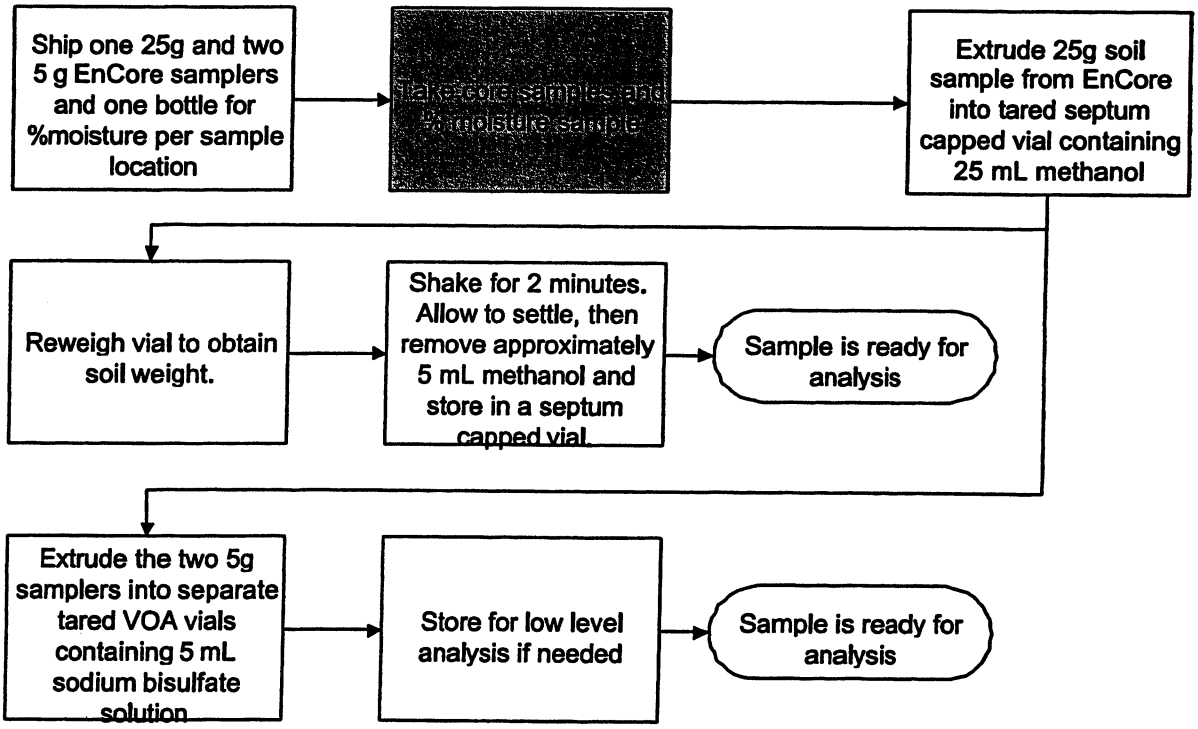
8.11. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14 day holding time. However they should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative). Maximum holding time for the EnCore™ sampler (before the sample is added to methanol or sodium bisulfate) is 48 hours.

8.12. A holding blank is stored with the samples. This is analyzed and replaced if any of the trip blanks show any contamination. Otherwise it is replaced every 14 days.

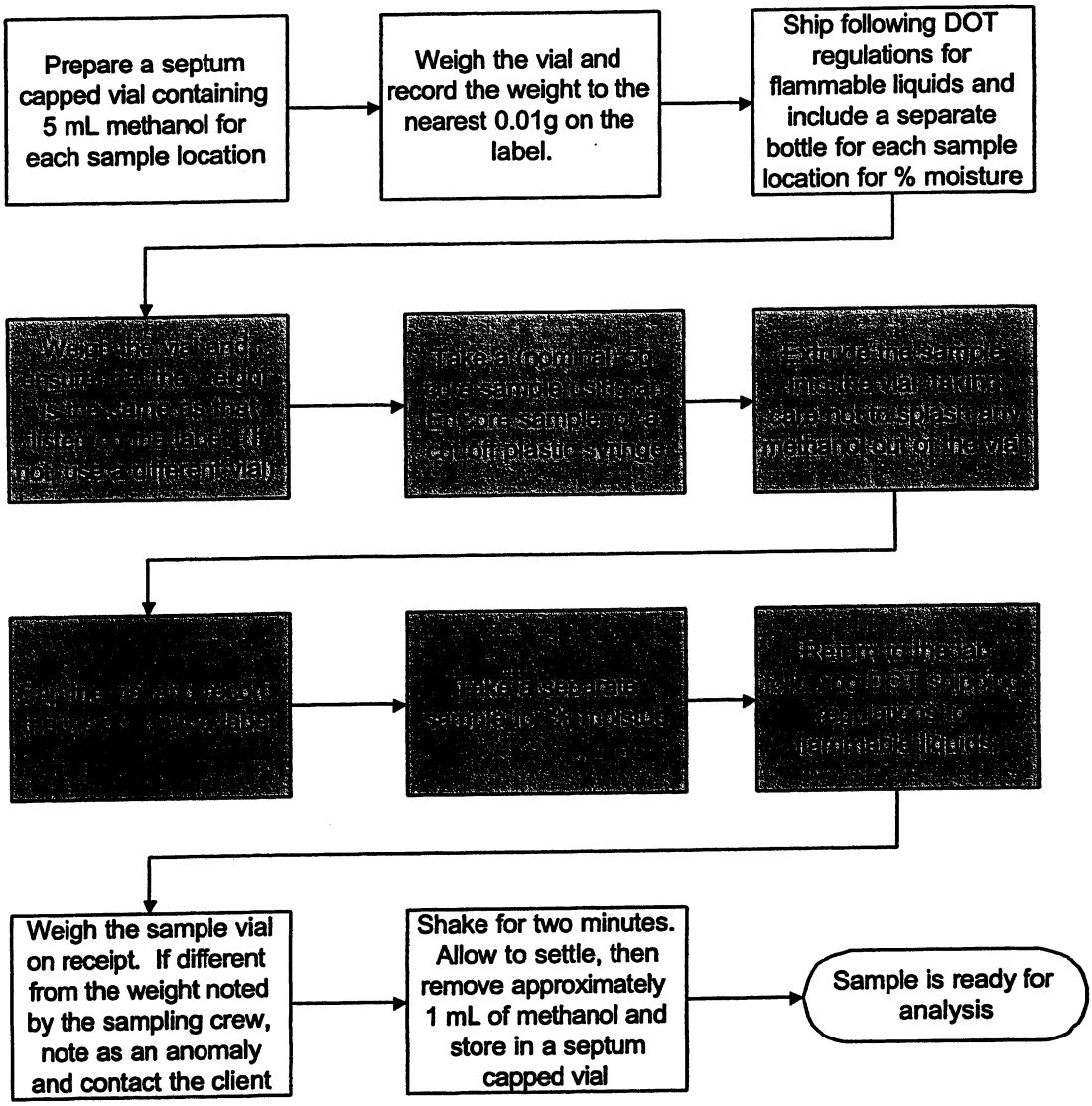
EnCore procedure when low level is not required (field steps in gray)



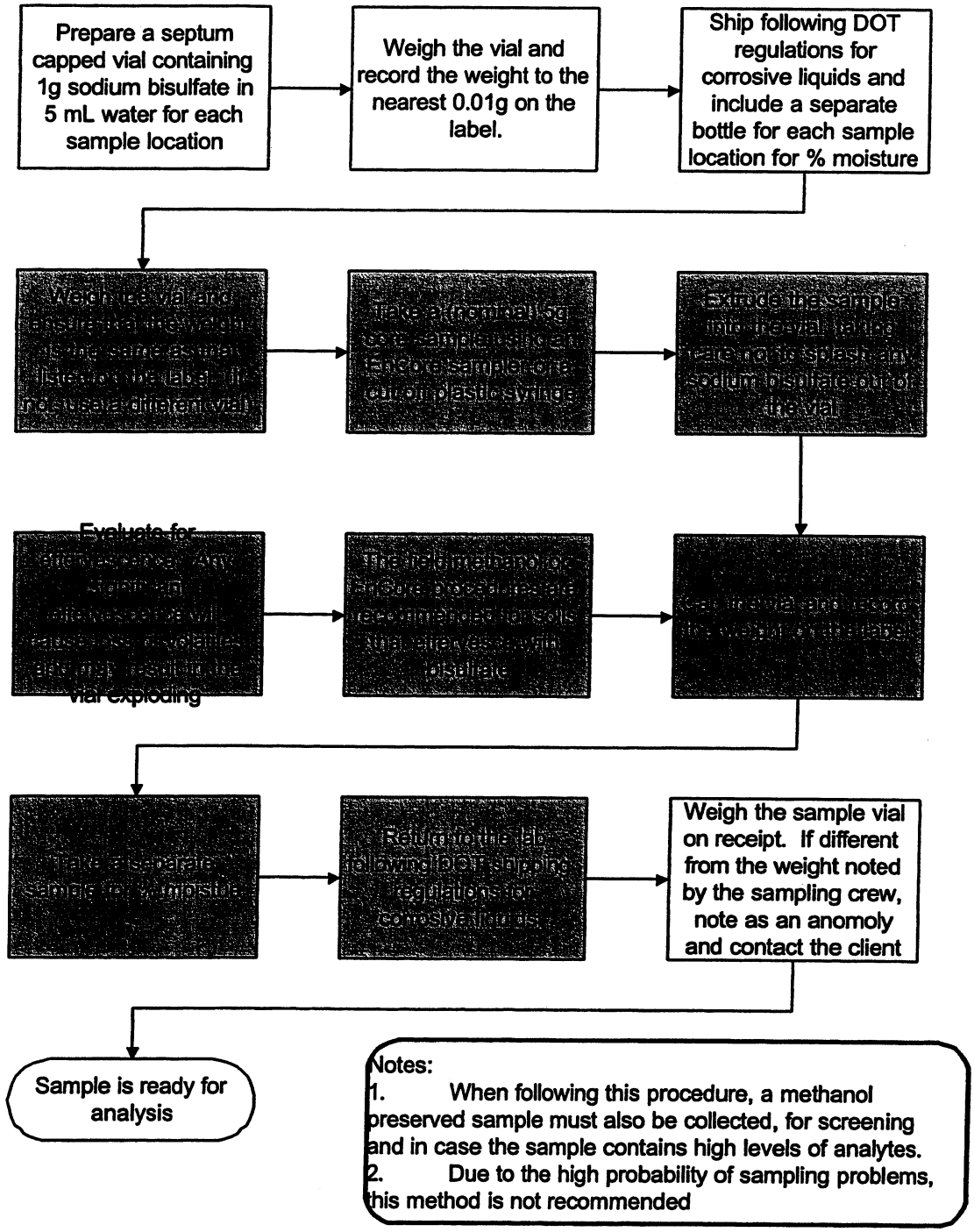
EnCore procedure when low level is required



Field methanol extraction procedure (field steps in gray)



Field bisulfate preservation procedure (field steps in gray)



9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration described in Section 13 and method detection limit (MDL) studies must be acceptable before analysis of samples may begin. MDLs should be analyzed for low and medium soils and aqueous samples.

9.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of a standard at the reporting limit and a single point calibration.

9.2. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery +/- 3 standard deviations for surrogates, matrix spikes and LCS. Precision limits for matrix spikes / matrix spike duplicates are 0 to mean relative percent difference + 3 standard deviations.

9.2.1. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.2. Refer to the QC Program document (QA-003) for further details of control limits.

9.3. Surrogates

Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Table 8. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.

- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.

- Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare/reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.3.1. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.

9.3.2. Refer to the STL QC Program document (QA-003) for further details of the corrective actions.

9.4. Method Blanks

9.4.1. For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. For low-level volatiles, the method blank consists of reagent water. For medium-level volatiles, the method blank consists of 25.0 mL of methanol. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

- If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

- Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be done in consultation with the client.

9.4.2. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the

purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.4.3. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B," and appropriate comments may be made in a narrative to provide further documentation.

9.4.4. Refer to the STL QC Program document (QA-003) for further details of the corrective actions.

9.5. Laboratory Control Samples (LCS)

9.5.1. For each batch of samples, analyze a LCS. The LCS is analyzed after the calibration standard, and normally before any samples. The LCS contains a representative subset of the analytes of interest (See Table 9), and must contain the same analytes as the matrix spike. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (Examples of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS.)

- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5.2. Refer to the STL QC Program document (QA-003) for further details of the corrective action.

9.5.3. If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be expected statistically. These requirements should be negotiated with the client. Refer to Section 17.5 for Ohio VAP specific analytes.

9.6. Matrix Spikes

9.6.1. For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 9. Compare the percent recovery and relative percent difference

(RPD) to that in the laboratory specific historically generated limits. See Section 17.5 for Ohio VAP specific analytes.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

- If the recovery for any component is outside QC limits for both the matrix spike/ spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include reanalysis of the batch.

- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.

- The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7. Nonconformance and Corrective Action

9.7.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.8. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.9. STL QC Program

Further details of QC and corrective action guidelines are presented in the STL QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. Prior to the analysis of samples and blanks, each GC/MS system must be tuned and calibrated. Hardware tuning is checked through the analysis of the 4-Bromofluorobenzene (BFB) to establish that a given GC/MS system meets the standard mass spectral abundance criteria. The

GC/MS system must be calibrated initially at a minimum of five concentrations (analyzed under the same BFB tune), to determine the linearity of the response utilizing target calibration standards. Once the system has been calibrated, the calibration must be verified each twelve hour time period for each GC/MS system.

10.2.1. General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 AMU
Scan Time:	to give at least 5 scans/peak, but not to exceed 2 second/scan
Injector Temperature:	200–250°C
Source Temperature:	According to manufacturer's specifications
Transfer Line	Temperature: 250–300°C
Purge Flow:	40 mL/minute
Carrier Gas	Flow: 15 mL/minute
Make-up Gas Flow:	25–30 mL/minute

10.2.2. Gas chromatograph suggested temperature program

10.2.2.1. BFB Analysis

Isothermal:	170°C

10.2.2.2. Sample Analysis

Initial Temperature:	40°C
Initial Hold Time:	4 minutes
Temperature Program:	8°C/minute
Final Temperature:	184°C
Second Temperature	Program: 40°C/minute
Final Temperature:	240°C
Final Hold Time:	2.6 minutes

10.3. Instrument Tuning

10.3.1. Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 10 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB.

10.4. Initial Calibration

10.4.1. A series of five initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Six standards must be used for a quadratic least squares calibration. Suggested calibration levels for a 5 mL purge are: 5, 20, 50, 100, and 200 µg/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Suggested calibration levels for a 25 mL purge are 1, 5, 10, 20, and 40 µg/L. Again, some analytes are prepared at higher levels. Tables 2 and 4 list the calibration levels for each analyte. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument. (For example, adequate sensitivity can be obtained on the Agilent 5973 instruments to use a 5 mL purge volume to reach the same reporting limits that once required a 25 mL purge. The calibration levels will still be the same 1, 5, 10, 20, 40µg/L.) However, the same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

10.4.2. It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for same tests. For example, the Appendix IX list requires the Primary standard (Table 5) and the Appendix IX standard (Table 6). If acceptable analytical performance can be obtained the primary and appendix IX standards may be analyzed together.

10.4.3. Internal standard calibration is used. The internal standards are listed in Table 7. Target compounds should reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See equation 1, Section 12, for calculation of response factor.

10.4.4. The % RSD of the calibration check compounds (CCC) must be less than 30%. Refer to Table 12 for the CCCs.

10.4.4.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.4.5. The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 11 for the SPCC compounds and required minimum response factors.

10.4.6. If the average of all the %RSDs in the calibration is $\leq$ 15%, then all analytes may use average response factor for calibration.

10.4.6.1. If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve then the appropriate curve should be used for quantitation. If Relative Standard Error (RSE) is used to evaluate the curve it must be better than 15%. Otherwise the correlation coefficient (coefficient of determination for non-linear curves) must be $\geq$ 0.990.

10.4.6.2. If the average of all the %RSDs in the calibration is > 15% then calibration on a curve must be used for all analytes with %RSD > 15%. Linear or quadratic curve fits may be used. The analyst should consider instrument maintenance to improve the linearity of response. If Relative Standard Error (RSE) is used to evaluate the curve it must be better than 15%. If the % RSD is > 15%, the analyst may drop the low or high in the ICAL , as long as a minimum of 5 points are maintained and the quantitation range is adjusted accordingly. Otherwise the correlation coefficient, (coefficient of determination, r^2 for non-linear curves) must be $\geq$ 0.990. If the correlation coefficient is < 0.990, then any hit for these compounds must be flagged as estimated.

10.4.6.3. Refer to Section 17.5 for specific Ohio VAP criteria.

10.4.7. Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called 1/X^2 weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4.8. If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.9. The calibration standards for the initial 5-point calibration for low level soils that are not preserved in sodium bisulfate (i.e. are preserved by freezing, or not preserved) must be heated to 40°C for purging. Using this calibration curve for water samples is acceptable as long as all calibration, QC, and samples are also heated to 40°C. A separate five point calibration must be prepared for analysis of low level soils that are preserved with sodium bisulfate. Low level soils analysis requires the use of a closed vial autosampler such as the Varian Archon, O.I. 4552 or Tekmar Precept. Each standard for analysis of sodium

bisulfate preserved samples is prepared by spiking the methanolic standard solution through the septum of a VOA vial containing 5 mL of water and 1 g sodium bisulfate. The standards are heated to 40°C for purging. All low-level soil samples, standards, and blanks must also be heated to 40°C for purging. Medium soil extracts should be analyzed using the water (unheated or optionally heated) calibration curve as long as all calibration standards, samples, and QC samples are purged at the same temperature.

10.4.10. Non-standard analytes are sometimes requested. For these analytes, it is acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. If the analyte is detected in any of the samples, a five point initial calibration must be generated and the sample(s) reanalyzed for quantitation. However, if the analyte is not detected, the non-detect may be reported and no further action is necessary.

Note: This procedure is may not be used for Ohio VAP samples.

10.5. Continuing Calibration: The initial calibration must be verified every twelve hours.

10.5.1. Continuing calibration begins with analysis of BFB as described in Section 10.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. The level 3 calibration standard is used as the continuing calibration.

10.5.2. The RF data from the standards are compared with the average RF from the initial five-point calibration to determine the percent drift of the CCC compounds. The calculation is given in equation 4, Section 12.3.4.

10.5.3. The % drift of the CCCs must be $\leq$ 20% for the continuing calibration to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 11. In addition, the % drift of all analytes must be $\leq$ 50% with allowance for up to six target analytes to have % drift > 50%.

10.5.3.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.5.3.2. Cyclohexanone, one of the components of the Appendix IX standard, is unstable in the calibration solution, forming 1,1-dimethoxycyclohexane. No calibration criteria are applied to cyclohexanone and quantitation is tentative. Cyclohexanone is included on the Universal Treatment Standard and FO-39 regulatory lists (but not on Appendix IX).

10.5.3.3. Refer to Table 12 for specific Ohio VAP analytes.

10.5.4. If the CCCs and or the SPCCs do not meet the criteria in Sections 10.5.3 and 10.5.4, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis begins. Extensive corrective action such as a different type of column will require a new initial calibration.

10.5.5. Once the above criteria have been met, sample analysis may begin. **Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs.** Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample *desorbed* less than or equal to 12 hours after the BFB is acceptable.)

11. PROCEDURE

11.1. Procedural Variations

11.1.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation shall be completely documented using a Nonconformance Memo and approved by a Supervisor or group leader and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.1.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.2. Preliminary Evaluation

11.2.1. Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories.

11.2.2. Dilutions should be done just prior to the GC/MS analysis of the sample. Dilutions are made in volumetric flasks or in a Luerlok syringe. Calculate the volume of reagent water required for the dilution. Fill the syringe with reagent water, compress the water to vent any residual air and adjust the water volume to the desired amount. Adjust the plunger to the mark and inject the proper aliquot of sample into the syringe. If the dilution required would use less than 1 µL of sample then serial dilutions must be made in volumetric flasks.

11.2.2.1. The diluted concentration is to be estimated to be in the upper half of the calibration range.

11.3. Sample Analysis Procedure

11.3.1. All analysis conditions for samples must be the same as for the continuing calibration standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.3.2. All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain a MS/MSD, a LCS, and a method blank.

11.3.2.1. If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next tune period. However, if any re-tuning of the instrument is necessary, or if a period of greater than 24 hours from the preceding BFB tune has passed, a new batch must be started. For medium level soils the batch is defined at the sample preparation stage.

11.3.2.2. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

11.3.2.3. It is not necessary to reanalyze batch QC with reanalyses of samples. However, any reruns must be as part of a valid batch.

11.4. Water Samples

11.4.1. All samples and standard solutions must be at ambient temperature before analysis.

11.4.2. Fill a syringe with the sample. If a dilution is necessary it may be made in the syringe if the sample aliquot is $\geq$ 5 µL. Check and document the pH of the remaining sample.

11.4.3. Add 250 ng of each internal and surrogate standard (10 µL of a 25 µg/mL solution, refer to Tables 7, 8 and 16). The internal standards and the surrogate standards may be mixed and added as one spiking solution (this results in a 50 µg/L solution for a 5 mL sample, and a 10 µg/L solution for a 25 mL sample). Inject the sample into the purging chamber.

11.4.3.1. For TCLP samples use 0.5 mL of TCLP leachate with 4.5 mL reagent water and spike with 10 µL of the 25 µg/mL TCLP spiking solution. (Note that TCLP reporting limits will be 10 times higher than the corresponding aqueous limits).

11.4.4. Purge the sample for eleven minutes (the trap must be below 35°C).

11.4.5. After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for approximately 3-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.4.6. Desorb and bake time and temperature are optimized for the type of trap in use. The same conditions must be used for samples and standards.

11.5. Methanol Extract Soils

11.5.1. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe. Refer to Section 17.8 for Michigan project requirements.

11.6. Liquid wastes that are soluble in methanol and insoluble in water.

11.6.1. Pipet 2 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1 gram.

11.6.2. Quickly add 7 mL of methanol, then add 1 mL of surrogate spiking solution to bring the final volume to 10 mL. Cap the vial and shake for 2 minutes to mix thoroughly. For a MS/MSD or LCS, 6 mL of methanol, 1 mL of surrogate solution, and 1 mL of matrix spike solution is used.

11.6.3. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe.

11.7. Aqueous and Low level Soil Sample Analysis (Purge and Trap units that sample directly from the VOA vial)

11.7.1. Units which sample from the VOA vial should be equipped with a module which automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

11.7.2. If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise the internal and surrogate standards must be added to the vial. *Note:* Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to analyzed as soils.

11.7.3. Soil samples must be quantitated against a curve prepared with standards containing about the same amount of sodium bisulfate as the samples (1 g in 5 mL).

11.7.4. Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.7.5. For aqueous samples, check the pH of the sample remaining in the VOA vial after analysis is completed.

11.8. Low-Level Solids Analysis using discrete autosamplers, Method 8260A, 5030A.

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

This method is based on purging a heated soil/sediment sample mixed with reagent water containing the surrogates and internal standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.8.1. Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

11.8.2. Weigh out 5 g (or other appropriate aliquot) of sample into a disposable culture tube or other purge vessel. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 1.0 g. If the sample is contaminated with analytes such that a purge amount less than 1.0 g is appropriate, use the medium level method. For the medium level method, add 4g soil to 10 mL methanol containing the surrogates, mix for two minutes, allow to settle then remove a portion of the methanol and store in a clean Teflon capped vial at 4°C until analysis. Analyze as described in section 11.5.

11.8.3. Connect the purge vessel to the purge and trap device.

11.8.4. Rinse a 5 mL gas-tight syringe with organic free water, and fill. Compress to 5 mL. Add surrogate/internal standard (and matrix spike solutions if required.). Add directly to the sample from 11.5.2.

11.8.5. The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

11.8.6. Add the heater jacket or other heating device and start the purge and trap unit.

11.8.7. Soil samples that have low IS recovery when analyzed (<50%) should be reanalyzed once to confirm matrix effect.

11.9. Medium-Level Soil/Sediment and Waste Samples

11.9.5. Sediments/soils and waste that are insoluble in methanol.

11.9.5.1. Sediments/soils and waste that are insoluble in methanol.

11.9.5.1.1. Gently mix the contents of the sample container with a narrow metal or wood spatula. Weigh 4 g (wet weight) into a tared vial. Use a top-loading balance. Record the weight to 0.1 gram. Do not discard any supernatant liquids.

11.9.5.1.2. Quickly add 9 mL of methanol, and 1 mL of surrogate spiking solution to bring the final volume of methanol to 10 mL. For an LCS or MS/MSD sample add 8 mL of methanol, 1 mL of surrogate spike solution, and 1 mL of matrix spike solution. Cap the vial and vortex to mix thoroughly.

NOTE: Sections 11.9.5.1.1 and 11.9.5.1.2 must be performed rapidly and without interruption to avoid the loss of volatile organics.

11.10. Initial review and corrective actions

11.10.1. If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minutes from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.10.2. If the internal standard response in the continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.10.2.1. Any samples that do not meet the internal standard criteria for the continuing calibration must be evaluated for validity. If the change in sensitivity is a matrix effect confined to an individual sample reanalysis is not necessary. If the change in sensitivity is

due to instrumental problems all affected samples must be reanalyzed after the problem is corrected.

11.10.3. The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis may not be necessary. Out of control surrogate standard response may be a matrix effect. It is only necessary to reanalyze a sample once to demonstrate matrix effect, but reanalysis at a dilution should be considered.

11.11. Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.10.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgement.

11.10.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component

characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)

12.1.1. If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

12.2. Tentatively Identified Compounds (TICs)

12.2.1. If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. Guidelines are:

12.2.1.1. Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

12.2.1.2. The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

12.2.1.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.1.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

12.2.1.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the spectrum because of background contamination or coeluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

12.2.1.6. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst assign a tentative identification.

12.3. Calculations.

12.3.1. Response factor (RF):

Equation 1

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_{is} = Concentration of the specific internal standard, ng

C_x = Concentration of the compound being measured, ng

12.3.2. Standard deviation (SD):

Equation 2

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(Xi - X)^2}{N - 1}}$$

X_i = Value of X at i through N

N = Number of points

X = Average value of X_i

12.3.3. Percent relative standard deviation (%RSD):

Equation 3

$$\%RSD = \frac{\text{Standard Deviation}}{\overline{RF_i}} \times 100$$

$\overline{RF_i}$ = Mean of RF values in the curve

12.3.4. Percent drift between the initial calibration and the continuing calibration:

Equation 4

$$\% \text{ Drift} = \frac{C_{expected} - C_{found}}{C_{expected}} \times 100$$

Where

$C_{expected}$ = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.3.5. Target compound and surrogate concentrations:

Concentrations in the sample may be determined from linear or second order (quadratic) curve fitted to the initial calibration points, or from the average response factor of the initial calibration points. Average response factor may only be used when the % RSD of the response factors in the initial calibration is $\leq$ 15%.

12.3.5.1. Calculation of concentration using Average Response Factors

Equation 5

$$\text{Concentration } \mu g / L = \frac{x}{RF}$$

12.3.5.2. Calculation of concentration using Linear fit

Equation 6

$$\text{Concentration } \mu g / L = A + Bx$$

12.3.5.3. Calculation of concentration using Quadratic fit

Equation 7

$$\text{Concentration } \mu g / L = A + Bx + Cx^2$$

x is defined in equations 8, 9 and 10

A is a constant defined by the intercept

B is the slope of the curve

C is the curvature

12.3.5.4. Calculation of x for Water and water-miscible waste:

Equation 8

$$x = \frac{(A_x)(I_s)(D_f)}{(A_{is})(V_o)}$$

Where:

A_x = Area of characteristic ion for the compound being measured (secondary ion quantitation is allowed only when there are sample interferences with the primary ion)

A_{is} = Area of the characteristic ion for the internal standard

I_s = Amount of internal standard added in ng

$$\text{Dilution Factor} = D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

V_o = Volume of water purged, mL

12.3.5.5. Calculation of x for Medium level soils:

Equation 9

$$x = \frac{(A_x)(I_s)(V_t)(1000)(D_f)}{(A_{is})(V_a)(W_s)(D)}$$

Where:

A_x, I_s, D_f, A_{is}, same as for water.

V_t = Volume of total extract, mL (Typically 25 mL)

V_a = Volume of extract added for purging, µL

W_s = Weight of sample extracted, g

$$D = \frac{100 - \%\text{moisture}}{100}$$

12.3.5.6. Calculation of x for Low level soils:

Equation 10

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$

Where:

A_x, I_s, A_{is}, same as for water.

D is as for medium level soils

W_s = Weight of sample added to the purge vessel, g

12.3.5.7. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x = Area in the total ion chromatogram for the compound being measured

A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

In other words, the concentration is equal to x as defined in equations 8, 9 and 10.

12.3.6. MS/MSD Recovery

Equation 11

$$\textbf{Matrix Spike Recovery, \%} = \frac{SSR - SR}{SA} \times 100$$

SSR = Spike sample result
SR = Sample result
SA = Spike added

12.3.7. Relative % Difference calculation for the MS/MSD

Equation 12

$$RPD = \frac{|MSR - MSDR|}{\frac{1}{2}(MSR + MSDR)} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result

MSDR = Matrix spike duplicate result

13. METHOD PERFORMANCE

13.1. Method Detection Limit

13.2. Generally, each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: QA-005. When non-standard compounds are analyzed at client request, lesser requirements are possible with client agreement. At a minimum, a standard at the reporting limit must be analyzed to demonstrate the capability of the method.

13.3. Initial Demonstration

13.4. Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest. The QC check sample is made up at 20 µg/L. (Some compounds will be at higher levels, refer to the calibration standard levels for guidance.)

13.4.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.4.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. The %RSD should be $\leq$ 15% for each analyte, and the % recovery should be within 80-120%.

13.4.3. If any analyte does not meet the acceptance criteria, check the acceptance limits in the reference methods (Table 6 of method 8240B, paragraph 8.3.5 of method 8260A). If the recovery or precision is outside the limits in the reference methods, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.4.4. Training Qualification

13.4.4.1. The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260B, Update III, December 1996

16.2. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260A, Update II, September 1994.

17. MISCELLANEOUS

17.1. Modifications from the reference method

17.1.1. Ion 119 is used as the quantitation ion for chlorobenzene-d5 for 25 mL purge tests.

17.1.2. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.3. The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.1.4. Method 8260A recommends that the purge vessel is run through an additional purge cycle after 25 mL sample analysis to remove carryover. Instead, purge vessels are oven baked between analyses or disposable vessels are used one time only.

17.1.5. SW-846 recommends that a curve be used for any analytes with %RSD of the response factors > 15%. However, some industry standard data systems and forms generation software cannot report this data with the necessary information for data validation. In

addition most software available does not allow weighting of the curve. Unweighted curves may exhibit serious errors in quantitation at the low end, resulting in possible false positives or false negatives. Therefore, this SOP allows used of average response factors if the average %RSD for all compounds is ≤ 15%.

17.2. Modifications from previous revision

This SOP has been substantially revised to reflect the changes included in Update III to SW-846. Directions for method 524.2 and method 624 have also been added.

17.3. Facility specific SOPs

Each facility shall attach a list of facility-specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4. Flow diagrams

17.4.1. Initial Demonstration and MDL

17.5. The following are protocols that must be followed when analyzing OhioVAP samples:

- Sections 9.5 and 9.6: n-Hexane must be spiked and reported for both the LCS and MS/MSD.
- Sections 10.4.6: All analytes must have a %RSDs ≤ 15%. Corrective action must be completed for any compounds failing the <15% requirement.
- Section 11.1 and 17.1.5 (Method deviations) are not to be performed.
- Section 11.9.2: For OhioVAP projects, the laboratory will reanalyze any sample where the internal standard fails and there is no evidence of matrix interference.

17.6. The following are protocols that must be followed when analyzing BP Oil – Lima Refinery RFI work plan.

- Section 8.1 STL will continue to follow the 14 day holding time specified in the Corporate SOP.
- Delete for this project Section 8.3 At specific client request, unpreserved soil samples may be accepted.

- Delete for this project Section 8.8.1 At specific client request unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the lab. This is the old procedure based on method 5030A. It is no longer included and is likely to generate results that are biased low, possibly by more than an order of magnitude.

- Modify Section 8.5.8 For the purpose of this project, the soil/methanol mixture may be stored for two days prior to analysis.

- Modify Section 8.6.12 For the purpose of this project, the soil/methanol mixture may be stored for two days prior to analysis.

- Modify (per discussion with Region V representative) to Section 10.4.6.2 Compounds with %RSD >15% are to be calibrated using an alternate calibration technique (e.g. linear or quadratic calibration curve). For poor responders, the alternate calibration technique requirements may not be met either. This sentence is added for those cases.If the correlation coefficient is < 0.990, then any hit for these compounds must be flagged as estimated.

- Modify Section 10.4.2 It is necessary to analyze the Appendix IX standard separately from the primary standard due to the presence of xylene solvent in the Appendix IX standard. Alternatively, STL will purchase the Appendix IX standard in a solvent other than xylene.

- Modify Section 10.4.9 For this project, this section will be modified to comply with the requirement of adding methanol to the calibration standards so that those standards contain the same amount of methanol as the diluted soil extracts.

- Modify Table 6

- For the project specific SOP, acetonitrile will be removed from table 6, page 49 and appended onto table 5, page 48. Acetonitrile will be calibrated as part of the STL primary standard, using a separate acetonitrile standard. This will ensure that the calibration curve for acetonitrile will be done free from any interference from allyl chloride.

17.7. The following are protocols that must be followed when analyzing South Carolina Projects only.

- **Delete** from Section 10.4.7 In a linear *or quadratic* calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve.

- **Delete** from Section 12.3.5 Concentrations in the sample may be determined from linear *or second order (quadratic)* curve fitted to the initial calibration points, or from the average response factor of the initial calibration points.

- **Delete** from Section 12.3.5.1

- Calculation of concentration using Quadratic fit

Equation 13

Concentration $\mu g / L = A + Bx + Cx^2$

x is defined in equations 8, 9 and 10

A is a constant defined by the intercept

B is the slope of the curve

C is the curvature

- **Change** Section 9.3 The compounds included in the surrogate spiking solutions are listed in Tables 8 *and 9*.

17.8. The following are protocols that must be followed to achieve the lower reporting limits required when analyzing Michigan projects.

17.8.1. Modify Section 8.5.4 and 8.6.8 (add 5 uL of 2500 ug/mL surrogate solution for a nominal 25 g sample).

17.8.2. Modify Section 8.5.5 and 8.6.9 (add 100 uL of 50 ug/mL spike solution for a nominal 25 g sample).

17.8.3. Modify Section 8.5.6 and 8.6.10 (add 100 uL of 50 ug/mL spike solution for a nominal 25g sample).

17.8.4. Michigan reporting limits for methanol preserved soils are achieved by injecting 100 uL of the methanol extract in a 5 mL purge. The instrument is calibrated using the recommended calibration levels in water of 1 ug/L, 2 ug/L (if a quadratic calibration is to be used), 5 ug/L, 10 ug/L, 20 ug/L and 40 ug/L. Some analytes are prepared at higher concentrations.

17.8.5. Samples for Michigan projects frequently require calibration for 2-Methylnaphthalene. Recommended calibration levels for this compounds are 2 ug/L, 10 ug/L, 20 ug/L, 40 ug/L and 80 ug/L.

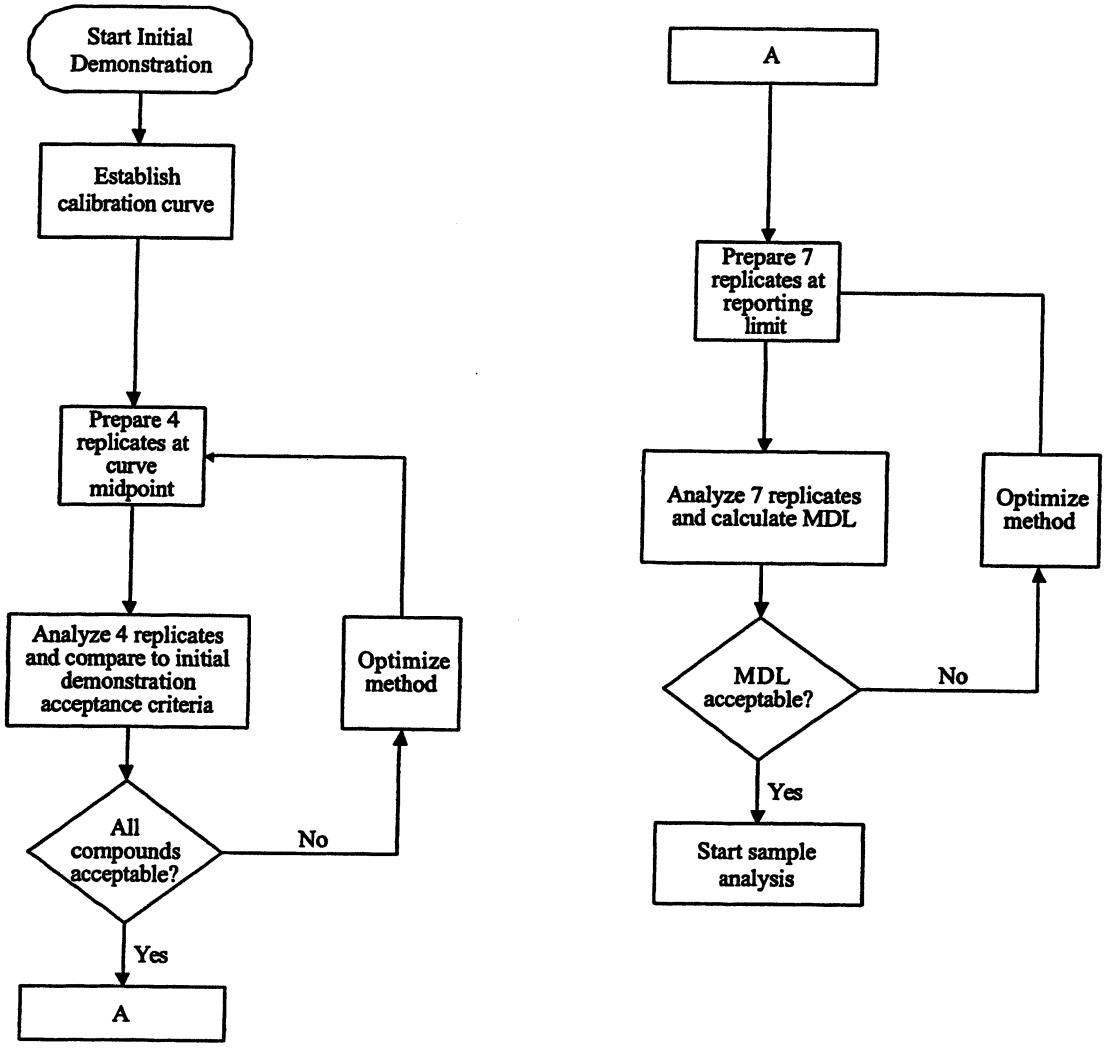

Start Initial Demonstration
Establish calibration curve
Prepare 4 replicates at curve midpoint
Analyze 4 replicates and compare to initial demonstration acceptance criteria
Optimize method
All compounds acceptable?
No
Yes
A
A
Prepare 7 replicates at reporting limit
Analyze 7 replicates and calculate MDL
Optimize method
MDL acceptable?
No
Yes
Start sample analysis

Table 1

STL Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	25 mL[3] water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
Dichlorodifluoromethane	75-71-8	10	2	10	250	1200
Chloromethane	74-87-3	10	2	10	250	1200
Bromomethane	74-83-9	10	2	10	250	1200
Vinyl chloride	75-01-4	10	2	10	250	1200
Chloroethane	75-00-3	10	2	10	250	1200
Trichlorofluoromethane	75-69-4	10	2	10	250	1200
Acrolein	107-02-8	100	20	100	5000	12000
Acetone	67-64-1	20	10	20	1000	2500
Trichlorotrifluoroethane	76-13-1	5	1	5	250	620
Iodomethane	74-88-4	5	1	5	250	620
Carbon disulfide	75-15-0	5	1	5	250	620
Methylene chloride	75-09-2	5	1	5	250	620
tert-Butyl alcohol	75-65-0	200	50	200	10,000	25000
1,1-Dichloroethene	75-35-4	5	1	5	250	620
1,1-Dichloroethane	75-34-3	5	1	5	250	620
trans-1,2-Dichloroethene	156-60-5	5.0	1.0	5.0	250	310
Acrylonitrile	107-13-1	100	20	100	5000	12000
Methyl *tert*-butyl ether (MTBE)	1634-04-4	20	5	20	1000	2500
Hexane	110-54-3	5	1	5	250	620
cis-1,2-Dichloroethene	156-59-2	5.0	1.0	5.0	250	310
1,2-Dichloroethene (Total)	540-59-0	5	1	5	250	620
Tetrahydrofuran	109-99-9	20	5	20	1000	2500
Chloroform	67-66-3	5	1	5	250	620
1,2-Dichloroethane	107-06-2	5	1	5	250	620
Dibromomethane	74-95-3	5	1	5	250	620
2-Butanone	78-93-3	20	5	20	1000	2500
1,4-Dioxane	123-91-1	500	200	500	25000	62000
1,1,1-Trichloroethane	71-55-6	5	1	5	250	620
Carbon tetrachloride	56-23-5	5	1	5	250	620
Bromodichloromethane	75-27-4	5	1	5	250	620
1,2-Dichloropropane	78-87-5	5	1	5	250	620
cis-1,3-Dichloropropene	10061-01-5	5	1	5	250	620

Table 1

STL Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	25 mL[3] water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
Trichloroethene	79-01-6	5	1	5	250	620
Dibromochloromethane	124-48-1	5	1	5	250	620
1,2-Dibromoethane	106-93-4	5	1	5	250	620
1,2,3-Trichloropropane	96-18-4	5	1	5	250	620
1,1,2-Trichloroethane	79-00-5	5	1	5	250	620
Benzene	71-43-2	5	1	5	250	620
Ethylmethacrylate	97-63-2	5	1	5	250	620
trans-1,3-Dichloropropene	10061-02-6	5	1	5	250	620
Bromoform	75-25-2	5	1	5	250	620
4-Methyl-2-pentanone	108-10-1	20	5	20	1000	2500
2-Hexanone	591-78-6	20	5	20	1000	2500
Tetrachloroethene	127-18-4	5	1	5	250	620
Toluene	108-88-3	5	1	5	250	620
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	250	620
2-Chloroethyl vinyl ether	110-75-8	N/A[2]	N/A	50	1000	6200
Vinyl acetate	108-05-4	10	2	10	500	1200
Chlorobenzene	108-90-7	5	1	5	250	620
Ethylbenzene	100-41-4	5	1	5	250	620
Styrene	100-42-5	5	1	5	250	620
t-1,4-Dichloro-2-butene	110-57-6	5	1	5	250	620
m and p Xylenes		5.0	0.5	2.5	125	310
o-xylene	95-47-6	5.0	0.5	2.5	125	310
Total xylenes	1330-20-7	5	1	5	250	620
1,3-Dichlorobenzene	541-73-1	5	1	5	250	620
1,4-Dichlorobenzene	106-46-7	5	1	5	250	620
1,2-Dichlorobenzene	95-50-1	5	1	5	250	620
2,2-Dichloropropane	590-20-7	5	1	5	250	
Bromochloromethane	74-97-5	5	1	5	250	
1,1-Dichloropropene	563-58-6	5	1	5	250	
Bromodichloromethane	75-27-4	5	1	5	250	
1,2-Dichloropropane	78-87-5	5	1	5	250	
1,3-Dichloropropane	142-28-9	5	1	5	250	

Table 1

STL Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	25 mL[3] water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
Isopropylbenzene	98-82-8	5	1	5	250	
Bromobenzene	108-86-1	5	1	5	250	
n-Propylbenzene	103-65-1	5	1	5	250	
2-Chlorotoluene	95-49-8	5	1	5	250	
4-Chlorotoluene	106-43-4	5	1	5	250	
1,3,5-Trimethylbenzene	108-67-8	5	1	5	250	
tert-Butylbenzene	98-06-6	5	1	5	250	
1,2,4-Trimethylbenzene	95-63-6	5	1	5	250	
sec-butylbenzene	135-98-8	5	1	5	250	
4-Isopropyltoluene	99-87-6	5	1	5	250	
n-Butylbenzene	104-51-8	5	1	5	250	
1,2,4-Trichlorobenzene	120-82-1	5	1	5	250	
Napthalene	91-20-3	5	1	5	250	
Hexachlorobutadiene	87-68-3	5	1	5	250	
1,2,3-Trichlorobenzene	87-61-6	5	1	5	250	
Acetonitrile	75-05-8	100	20	100	5000	

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

[2] 2-Chloroethyl vinyl ether cannot be reliably recovered from acid preserved samples

[3] Optionally, 5 mL purge volume if adequate sensitivity is obtained.

Table 2

STL Primary Standard Calibration Levels, 5 mL purge[1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2-Dichloroethane-d4 (Surrogate)	5	20	50	100	200
Toluene-d8 (Surrogate)	5	20	50	100	200
4-Bromofluorobenzene (Surrogate)	5	20	50	100	200
Dichlorodifluoromethane	5	20	50	100	200
Chloromethane	5	20	50	100	200
Bromomethane	5	20	50	100	200
Vinyl chloride	5	20	50	100	200
Chloroethane	5	20	50	100	200
Trichlorofluoromethane	5	20	50	100	200
Acrolein	50	200	500	1000	2000
Acetone	5	20	50	100	200
Trichlorotrifluoroethane	5	20	50	100	200
Iodomethane	5	20	50	100	200
Carbon disulfide	5	20	50	100	200
Methylene chloride	5	20	50	100	200
tert-Butyl alcohol	100	400	1,000	2,000	4,000
1,1-Dichloroethene	5	20	50	100	200
1,1-Dichloroethane	5	20	50	100	200
trans-1,2-Dichloroethene	5	20	50	100	200
Acrylonitrile	50	200	500	1,000	2,000
Methyl *tert*-butyl ether (MTBE)	5	20	50	100	200
Hexane	5	20	50	100	200
cis-1,2-Dichloroethene	5	20	50	100	200
Tetrahydrofuran	5	20	50	100	200
Chloroform	5	20	50	100	200
1,2-Dichloroethane	5	20	50	100	200
Dibromomethane	5	20	50	100	200
2-Butanone	5	20	50	100	200
1,4-Dioxane	250	1000	2,500	5,000	10,000
1,1,1-Trichloroethane	5	20	50	100	200
Carbon tetrachloride	5	20	50	100	200
Bromodichloromethane	5	20	50	100	200
1,2-Dichloropropane	5	20	50	100	200
cis-1,3-Dichloropropene	5	20	50	100	200
Trichloroethene	5	20	50	100	200
Dibromochloromethane	5	20	50	100	200

Table 2

STL Primary Standard Calibration Levels, 5 mL purge[1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2-Dibromoethane	5	20	50	100	200
1,2,3-Trichloropropane	5	20	50	100	200
Acetonitrile	50	200	500	1000	2000
1,1,2-Trichloroethane	5	20	50	100	200
Benzene	5	20	50	100	200
Ethylmethacrylate	5	20	50	100	200
trans-1,3-Dichloropropene	5	20	50	100	200
Bromoform	5	20	50	100	200
4-Methyl-2-pentanone	5	20	50	100	200
2-Hexanone	5	20	50	100	200
Tetrachloroethene	5	20	50	100	200
Toluene	5	20	50	100	200
1,1,2,2-Tetrachloroethane	5	20	50	100	200
2-Chloroethyl vinyl ether	10	40	100	200	400
Vinyl acetate	5	20	50	100	200
Chlorobenzene	5	20	50	100	200
Ethylbenzene	5	20	50	100	200
Styrene	5	20	50	100	200
t-1,4-Dichloro-2-butene	5	20	50	100	200
m and p Xylenes	10	40	100	200	400
o-xylene	5	20	50	100	200
1,3-Dichlorobenzene	5	20	50	100	200
1,4-Dichlorobenzene	5	20	50	100	200
1,2-Dichlorobenzene	5	20	50	100	200
2,2-Dichloropropane	5	20	50	100	200
Bromochloromethane	5	20	50	100	200
1,1-Dichloropropene	5	20	50	100	200
Bromodichloromethane	5	20	50	100	200
1,2-Dichloropropane	5	20	50	100	200
1,3-Dichloropropane	5	20	50	100	200
Isopropylbenzene	5	20	50	100	200
Bromobenzene	5	20	50	100	200
n-Propylbenzene	5	20	50	100	200
2-Chlorotoluene	5	20	50	100	200
4-Chlorotoluene	5	20	50	100	200
1,3,5-Trimethylbenzene	5	20	50	100	200
tert-Butylbenzene	5	20	50	100	200

Table 2

STL Primary Standard Calibration Levels, 5 mL purge[1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2,4-Trimethylbenzene	5	20	50	100	200
sec-butylbenzene	5	20	50	100	200
4-Isopropyltoluene	5	20	50	100	200
n-Butylbenzene	5	20	50	100	200
1,2,4-Trichlorobenzene	5	20	50	100	200
Napthalene	5	20	50	100	200
Hexachlorobutadiene	5	20	50	100	200
1,2,3-Trichlorobenzene	5	20	50	100	200

[1] **Levels for 25 mL purge are 5 times lower in all cases**

Table 2A

STL Primary Standard Calibration Levels, Low Level[1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Dibromofluoromethane (Surrogate)	1	5	10	20	40
1,2-Dichloroethane-d4 (Surrogate)	1	5	10	20	40
Toluene-d8 (Surrogate)	1	5	10	20	40
Bromofluorobenzene (Surrogate)	1	5	10	20	40
Dichlorodifluoromethane	1	5	10	20	40
Chloromethane	1	5	10	20	40
Vinyl Chloride	1	5	10	20	40
Bromomethane	1	5	10	20	40
Chloroethane	1	5	10	20	40
Trichlorofluoromethane	1	5	10	20	40
Acrolein	10	50	100	200	400
Acetone	2	10	20	40	80
1,1-Dichloroethene	1	5	10	20	40
Trichlorotrifluoroethane	1	5	10	20	40
Iodomethane	1	5	10	20	40
Carbon Disulfide	1	5	10	20	40
Methylene Chloride	1	5	10	20	40
Acetonitrile	10	50	100	200	400
Acrylonitrile	10	50	100	200	400
Methyl tert-butyl ether	1	5	10	20	40
trans-1,2-Dichloroethene	1	5	10	20	40
Hexane	1	5	10	20	40
Vinyl acetate	1	5	10	20	40
1,1-Dichloroethane	1	5	10	20	40
tert-Butyl Alcohol	20	100	200	400	800
2-Butanone	2	10	20	40	80
cis-1,2-dichloroethene	1	5	10	20	40
2,2-Dichloropropane	1	5	10	20	40
Bromochloromethane	1	5	10	20	40
Chloroform	1	5	10	20	40
Tetrahydrofuran	1	5	10	20	40
1,1,1-Trichloroethane	1	5	10	20	40
1,1-Dichloropropene	1	5	10	20	40
Carbon Tetrachloride	1	5	10	20	40
1,2-Dichloroethane	1	5	10	20	40
Benzene	1	5	10	20	40
Trichloroethene	1	5	10	20	40
1,2-Dichloropropane	1	5	10	20	40
1,4-Dioxane	50	250	500	1000	2000
Dibromomethane	1	5	10	20	40

Table 2A

STL Primary Standard Calibration Levels, Low Level [1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Bromodichloromethane	1	5	10	20	40
2-Chloroethyl vinyl ether	2	10	20	40	80
cis-1,3-Dichloropropene	1	5	10	20	40
4-Methyl-2-pentanone	2	10	20	40	80
Toluene	1	5	10	20	40
trans-1,3-Dichloropropene	1	5	10	20	40
Ethyl Methacrylate	1	5	10	20	40
1,1,2-Trichloroethane	1	5	10	20	40
1,3-Dichloropropane	1	5	10	20	40
Tetrachloroethene	1	5	10	20	40
2-Hexanone	2	10	20	40	80
Dibromochloromethane	1	5	10	20	40
1,2-Dibromoethane	1	5	10	20	40
Chlorobenzene	1	5	10	20	40
1,1,1,2-Tetrachloroethane	1	5	10	20	40
Ethylbenzene	1	5	10	20	40
m + p-Xylene	2	10	20	40	80
Xylene-o	1	5	10	20	40
Styrene	1	5	10	20	40
Bromoform	1	5	10	20	40
Isopropylbenzene	1	5	10	20	40
1,1,2,2-Tetrachloroethane	1	5	10	20	40
1,4-Dichloro-2-butene	1	5	10	20	40
1,2,3-Trichloropropane	1	5	10	20	40
Bromobenzene	1	5	10	20	40
n-Propylbenzene	1	5	10	20	40
2-Chlorotoluene	1	5	10	20	40
1,3,5-Trimethylbenzene	1	5	10	20	40
4-Chlorotoluene	1	5	10	20	40
tert-Butylbenzene	1	5	10	20	40
1,2,4-Trimethylbenzene	1	5	10	20	40
sec-Butylbenzene	1	5	10	20	40
4-Isopropyltoluene	1	5	10	20	40
1,3-Dichlorobenzene	1	5	10	20	40
1,4-Dichlorobenzene	1	5	10	20	40
n-Butylbenzene	1	5	10	20	40
1,2-Dichlorobenzene	1	5	10	20	40
1,2-Dibromo-3-chloropropane	1	5	10	20	40

Table 2A

STL Primary Standard Calibration Levels, Low Level [1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2,4-Trichlorobenzene	1	5	10	20	40
Hexachlorobutadiene	1	5	10	20	40
Naphthalene	1	5	10	20	40
1,2,3-Trichlorobenzene	1	5	10	20	40
Cyclohexane	1	5	10	20	40
Methyl Acetate	2	10	20	40	80
Methylcyclohexane	1	5	10	20	40
1,3,5-Trichlorobenzene	1	5	10	20	40

[1] 25 mL purge samples analyzed at 5 mL purge on more sensitive equipment.

Table 3

STL Appendix IX Standard and Reporting Limits, 5 mL purge

Compound	CAS Number	Reporting Limits			
		5 mL Water µg/L	25mL[2] water µg/L	Low Soil µg/kg	Medium Soil µg/mL
Allyl Chloride	107-05-1	10	2	10	500
Acetonitrile	75-05-8	100	20	100	5000
Dichlorofluoromethane		10	2	10	500
Isopropyl ether	108-20-3	10	2	10	500
Chloroprene	126-99-8	5	2	5	250
n-Butanol	71-36-3	200	50	200	10,000
Propionitrile	107-12-0	20	4	20	1000
Methacrylonitrile	126-98-7	5	2	5	250
Isobutanol	78-83-1	200	50	200	10,000
Methyl methacrylate	80-62-6	5	2	5	250
1,1,1,2-Tetrachloroethane	630-20-6	5	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	10	2	10	500
Ethyl ether	60-29-7	10	2	10	500
Ethyl Acetate	141-78-6	20	4	20	1,000
2-Nitropropane	79-46-9	10	4	10	500
Cyclohexanone	108-94-1	N/A[1]	N/A[1]	N/A[1]	N/A[1]
Isopropylbenzene	98-82-8	5	1	5	250
2-Methylnaphthalene (Michigan only)	91-57-6	NA	5	NA	330

[1] Cyclohexanone decomposes to 1,1-dimethoxycyclohexane in methanolic solution. Reporting limits cannot be accurately determined.

[2] Optionally, 5 mL purge volume if adequate sensitivity is obtained.

Table 4

Recommended/STL Appendix IX Standard Calibration Levels, µg/L

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Allyl Chloride	5	20	50	100	200
Acetonitrile	50	200	500	1,000	2,000
Dichlorofluoromethane	5	20	50	100	200
Isopropyl ether	5	20	50	100	200
Chloroprene	5	20	50	100	200
n-Butanol	100	400	1,000	2,000	4,000
Propionitrile	10	40	100	200	400
Methacrylonitrile	5	20	50	100	200
Isobutanol	100	400	1,000	2,000	4,000
Methyl methacrylate	5	20	50	100	200
1,1,1,2-Tetrachloroethane	5	20	50	100	200
1,2-Dibromo-3-chloropropane	10	40	100	200	400
Ethyl ether	5	20	50	100	200
Ethyl Acetate	10	40	100	200	400
2-Nitropropane	10	40	100	200	400
Cyclohexanone	50	200	500	1,000	2,000
2-Methylnaphthalene (Michigan only)	2	10	20	40	80

Table 5

Reportable Analytes for STL Standard Tests, Primary Standard

Compound	CAS Number	STL Standard List	TCLP	TCL	Appendix IX	UTS
Dichlorodifluoromethane	75-71-8				X	X
Chloromethane	74-87-3	X		X	X	X
Bromomethane	74-83-9	X		X	X	X
Vinyl chloride	75-01-4	X	X	X	X	X
Chloroethane	75-00-3	X		X	X	X
Trichlorofluoromethane	75-69-4				X	X
Acrolein	107-02-8				X	X
Acetone	67-64-1	X		X	X	X
Trichlorotrifluoroethane	76-13-1					X
Ethanol	64-17-5					
Iodomethane	74-88-4				X	X
Carbon disulfide	75-15-0	X		X	X	X
Methylene chloride	75-09-2	X		X	X	X
tert-Butly alcohol	75-65-0					
1,1-Dichloroethene	75-35-4	X	X	X	X	X
1,1-Dichloroethane	75-34-3	X		X	X	X
trans-1,2-Dichloroethene	156-60-5	X		X	X	X
Total dichloroethene		X		X	X	X
Acrylonitrile	107-13-1				X	X
Methyl *tert*-butyl ether (MTBE)	1634-04-4					
Hexane	110-54-3					
cis-1,2-Dichloroethene	156-59-2	X		X		
Tetrahydrofuran	109-99-9					
Chloroform	67-66-3	X	X	X	X	X
1,2-Dichloroethane	107-06-2	X	X	X	X	X
Dibromomethane	74-95-3				X	X
2-Butanone	78-93-3	X	X	X	X	X
1,4-Dioxane	123-91-1				X	X
1,1,1-Trichloroethane	71-55-6	X		X	X	X
Carbon tetrachloride	56-23-5	X	X	X	X	X
Bromodichloromethane	75-27-4	X		X	X	X
1,2-Dichloropropane	78-87-5	X		X	X	X
cis-1,3-Dichloropropene	10061-01-5	X		X	X	X
Trichloroethene	79-01-6	X	X	X	X	X

Table 5

Reportable Analytes for STL Standard Tests, Primary Standard

Compound	CAS Number	STL Standard List	TCLP	TCL	Appendix IX	UTS
Dibromochloromethane	124-48-1	X		X	X	X
1,2-Dibromoethane	106-93-4				X	X
1,2,3-Trichloropropane	96-18-4				X	X
1,1,2-Trichloroethane	79-00-5	X		X	X	X
Benzene	71-43-2	X	X	X	X	X
Ethylmethacrylate	97-63-2				X	X
trans-1,3-Dichloropropene	10061-02-6	X		X	X	X
Bromoform	75-25-2	X		X	X	X
4-Methyl-2-pentanone	108-10-1	X		X	X	X
2-Hexanone	591-78-6	X		X	X	
Tetrachloroethene	127-18-4	X	X	X	X	X
Toluene	108-88-3	X		X	X	X
1,1,2,2-Tetrachloroethane	79-34-5	X		X	X	X
2-Chloroethyl vinyl ether	110-75-8					
Vinyl acetate	108-05-4				X	
Chlorobenzene	108-90-7	X	X	X	X	X
Ethylbenzene	100-41-4	X		X	X	X
Styrene	100-42-5	X		X	X	
t-1,4-Dichloro-2-butene	110-57-6				X	
m and p Xylenes		X		X	X	X
o-xylene	95-47-6	X		X	X	X
Total xylenes	1330-20-7	X		X	X	X
1,3-Dichlorobenzene	541-73-1					
1,4-Dichlorobenzene	106-46-7					
1,2-Dichlorobenzene	95-50-1					

Table 6

Reportable Analytes for STL Standard Tests, Appendix IX standard

Compound	Number	STL Standard List	TCLP	TCL	Appendix IX	UTS
Allyl Chloride	107-05-1				X	
Acetonitrile	75-05-8				X	X
Dichlorofluoromethane	75-43-4					
Isopropyl ether	108-20-3					
Chloroprene	126-99-8				X	
n-Butanol	71-36-3					
Propionitrile	107-12-0				X	
Methacrylonitrile	126-98-7				X	X
Isobutanol	78-83-1				X	X
Methyl methacrylate	80-62-6				X	X
1,1,1,2-Tetrachloroethane	630-20-6				X	X
1,2-Dibromo-3-chloropropane	96-12-8				X	X
Ethyl ether	60-29-7					X
Ethyl Acetate	141-78-6					X
2-Nitropropane	79-46-9					
Cyclohexanone	108-94-1					X
Isopropylbenzene	98-82-8					

Table 7

Internal Standards

	Standard Concentration µg/mL	Quantitation ion (5 mL purge)	Quantitation ion (25 mL purge)
Fluorobenzene	50	96	96
Chlorobenzene-d5	50	117	119
1,4-Dichlorobenzene-d4	50	152	152

Notes:

1) 5 µL of the internal standard is added to the sample. This results in a concentration of each internal in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 8

Surrogate Standards

Surrogate Compounds	Standard Concentration µg/mL
1,2-Dichloroethane-d_4	50
Dibromofluoromethane	50
Toluene-d_8	50
4-Bromofluorobenzene	50

Notes:

1) 5 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

3) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 9

Matrix Spike / LCS Control Compounds

Compound	Standard Concentration µg /mL
1,1-Dichloroethene	50
Trichloroethene	50
Toluene	50
Benzene	50
Chlorobenzene	50
n-Hexane (Ohio VAP only)	50

Notes:

1) 5 µL of the standard is added to the LCS or matrix spiked sample. This results in a concentration of each spike analyte in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Recovery and precision limits for LCS and MS/MSD are generated fromhistorical data and are maintained by the QA dedartment.

3) Full analyte spikes may also be used at the laboratories option or at client request.

Table 10

BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

Table 11

SPCC Compounds and Minimum Response Factors

Compound	8260B, 8260A Min. RF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	>0.100
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

Table 12

CCC compounds

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	<30.0	<20.0
1,1-Dichloroethene	<30.0	<20.0
Chloroform	<30.0	<20.0
1,2-Dichloropropane	<30.0	<20.0
Toluene	<30.0	<20.0
Ethylbenzene	<30.0	<20.0
n-Hexane (Ohio VAP only)	<30.0	<20.0

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d_4 (Surrogate)	65	102	
Dichlorodifluoromethane	85	87	50, 101,103
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58
Iodomethane	142	127	141
Carbon disulfide	76	78	
Trichlorotrifluoroethane	151	101	153
Ethanol	45	46	
Acetone	43	58	
Methylene chloride	84	49	51, 86
tert-Butyl alcohol	59	74	
trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl *tert* butyl ether	73		
Hexane	57	43	
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	
Tetrahydrofuran	42	71	
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	
Vinyl acetate	43	86	
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121
Benzene	78	52	77
Trichloroethene	130	95	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Toluene-d_8 (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65
4-Methyl-2-pentanone	43	58	57, 100

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77
Ethylbenzene	106	91	
Xylenes	106	91	
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133
Allyl Chloride	76	41	78
Acetonitrile	40	41	
Dichlorofluoromethane	67	69	
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74
Methyl methacrylate	41	69	100
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	

* The primary ion should be used for quantitation unless interferences are present, in which case a secondary ion may be used.

** m/z 43 may be used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

1. REQUIREMENTS FOR EPA 624

1.1. Method 624 is required for demonstration of compliance with NPDES wastewater discharge permits. This method can be applied only to aqueous matrices. The standard analyte list and reporting limits are listed in Table B-1.

1.2. The tune period for this method is defined as 24 hours.

1.3. The initial calibration curve for this method requires at least three points.

1.4. Sample concentrations are calculated using the average RRF from the initial calibration curve.

1.5. Each target analyte is assigned to the closest eluting internal standard.

1.6. Initial demonstration of Proficiency

1.6.1. The spiking level for the four replicate initial demonstration of proficiency is 20 µg/L. The acceptance criteria are listed in Table B-2

1.7. Initial calibration curve requirements:

1.7.1. Target compounds must have RSD ≤ 35%.

1.7.2. If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds. There is no correlation coefficient requirement for the regression curve.

1.8. Continuing calibration verification requirements:

1.8.1. The continuing calibration standard is from a different source than the initial calibration standard. The acceptance criteria are listed in Table B-2.

1.9. Matrix Spike and LCS requirements

1.9.1. The matrix spike and LCS are spiked at 20 µg/L. A matrix spike duplicate is not necessary for this method. The recovery limits for matrix spike and LCS recovery are listed in Table C-2.

1.10. Method clarifications, modifications and additions

1.10.1. Section 5.2.2 of the source method describes the trap packing materials as Tenax GC, Methyl silicone, silica gel and coconut charcoal. STL routinely employs the Supelco VOCARB 3000, which consists of Carbopack B and Carboxen 1000 and 1001.

1.10.2. Section 5.3.2 of the source method describes a packed analytical column. STL routinely employs capillary columns when performing this method.

1.10.3. The source method provides a suggested list of compounds for internal and surrogate standards. STL uses the following two compounds which are not on the table: Chlorobenzene-d_5 (internal standard) and 1,2-Difluorobenzene-d_4 (surrogate).

Table A-1.

Method 624 Analytes and Reporting Limits

Analytes	µg/L
Benzene	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	5
Carbon tetrachloride	5
Chlorobenzene	5
Chloroethane	5
2-Chloroethyl vinyl ether	5
Chloroform	5
Chloromethane	5
Dibromochloromethane	5
1,2-Dichlorobenzene	5
1,3-Dichlorobenzene	5
1,4-Dichlorobenzene	5
1,1-Dichloroethane	5
1,2-Dichloroethane	5
1,1-Dichloroethene	5
trans-1,2-Dichloroethene	5
1,2-Dichloropropane	5
cis-1,3-Dichloropropene	5
trans-1,3-Dichloropropene	5
Ethylbenzene	5
Methylene chloride	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	5
Vinyl chloride	5

Table A-2.

Method 624 QC Acceptance Criteria

Analytes	Daily QC check acceptance criteria (20µg/L spike)	Mean recovery, 4 replicate initial demonstration acceptance criteria (20µg/L spike)	Standard deviation, 4 replicate initial demonstration acceptance criteria (20µg/L spike)	Matrix spike and LCS acceptance criteria (% recovery)
Benzene	12.8-27.2	15.2-26.0	6.9	37-151
Bromodichloromethane	13.1-26.9	10.1-28.0	6.4	35-155
Bromoform	14.2-25.8	11.4-31.1	5.4	45-169
Bromomethane	2.8-37.2	D-41.2	17.9	D-242
Carbon tetrachloride	14.6-25.4	17.2-23.5	5.2	70-140
Chlorobenzene	13.2-26.8	16.4-27.4	6.3	37-160
Chloroethane	7.6-32.4	8.4-40.4	11.4	14-230
2-Chloroethyl vinyl ether	D-44.8	D-50.4	25.9	D-305
Chloroform	13.5-26.5	13.7-24.2	6.1	51-138
Chloromethane	D-40.8	D-45.9	19.8	D-273
Dibromochloromethane	13.5-26.5	13.8-26.6	6.1	53-149
1,2-Dichlorobenzene	12.6-27.4	11.8-34.7	7.1	18-190
1,3-Dichlorobenzene	14.6-25.4	17.0-28.8	5.5	59-156
1,4-Dichlorobenzene	12.6-27.4	11.8-34.7	7.1	18-190
1,1-Dichloroethane	14.5-25.5	14.2-28.5	5.1	59-155
1,2-Dichloroethane	13.6-26.4	14.3-27.4	6.0	49-155
1,1-Dichloroethene	10.1-29.9	3.7-42.3	9.1	D-234
trans-1,2-Dichloroethene	13.9-26.1	13.6-28.5	5.7	54-156
1,2-Dichloropropane	6.8-33.2	3.8-36.2	13.8	D-210
cis-1,3-Dichloropropene	4.8-35.2	1.0-39.0	15.8	D-227
trans-1,3-Dichloropropene	10.0-30.0	7.6-32.4	10.4	17-183
Ethylbenzene	11.8-28.2	17.4-26.7	7.5	37-162
Methylene chloride	12.1-27.9	D-41.0	7.4	D-221
1,1,2,2-Tetrachloroethane	12.1-27.9	13.5-27.2	7.4	46-157
Tetrachloroethene	14.7-25.3	17.0-26.6	5.0	64-148
Toluene	14.9-25.1	16.6-26.7	4.8	47-150
1,1,1-Trichloroethane	15.0-25.0	13.7-30.1	4.6	52-162
1,1,2-Trichloroethane	14.2-25.8	14.3-27.1	5.5	52-150
Trichloroethene	13.3-26.7	18.6-27.6	6.6	71-157
Trichlorofluoromethane	9.6-30.4	8.9-31.5	10.0	17-181
Vinyl chloride	0.8-39.2	D-43.5	20.0	D-251

STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: **EXTRACTION AND CLEANUP OF ORGANIC COMPOUNDS FROM WATERS AND SOILS, BASED ON SW-846 3500 SERIES, 3600 SERIES, 8151A AND 600 SERIES METHODS.**

(SUPERSEDES: Revision 3.7 (Dated 03/20/01))

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1. SCOPE AND APPLICATION

This SOP describes procedures for preparation (extraction and cleanup) of semivolatile organic analytes in aqueous, TCLP leachate, and soil matrices for analysis by Gas Chromatography (GC) and Gas Chromatography / Mass Spectrometry (GC/MS). The procedures are based on SW-846 and 600 series methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.

1.1. Extraction procedures for the following determinative methods are covered:

8081A, 8082, 8141A, 8151B, 8270C, 8310, 8015B, 8100, 608, 610, and 625

1.2. The extraction procedures here may be appropriate for other determinative methods when appropriate spiking mixtures are used.

1.3. Other extraction procedures (PFE, SPE, accelerated Soxhlet, etc.) may be used but are not currently covered in this SOP.

1.4. The applicable LIMS method codes are: P2 (8141A), QJ (8081A), QS (8151A), QL (8270C), SG (8310), DM (608), VT (610), DP (625), A1 (8100), HS (8015B)

2. SUMMARY OF METHOD

2.1. Separatory Funnel Extraction

A measured volume of sample, typically 1 liter, is adjusted, if necessary, to a specified pH and serially extracted with methylene chloride using a separatory funnel.

2.2. Continuous Liquid/Liquid Extraction

A measured volume of sample, typically 1 liter, is placed into a continuous liquid/liquid extractor, adjusted, if necessary, to a specific pH and extracted with methylene chloride for 18-24 hours.

2.3. Sonication Extraction

A measured weight of sample, typically 30 g, is mixed with anhydrous sodium sulfate to form a free flowing powder. This is solvent extracted three times using an ultrasonic horn.

2.4. Soxhlet Extraction

A 30 g sample is mixed with anhydrous sodium sulfate to form a free flowing powder. This is extracted with refluxing solvent.

2.5. Cleanup and Concentration

Procedures are presented for removing interferents from sample extracts, and for drying and concentration of the extract to final volume for analysis.

2.6. Phenoxy acid herbicide extractions

Procedures for the extraction and cleanup of phenoxy acid herbicides are presented in Appendix A.

3. DEFINITIONS

Definitions of terms used in this SOP may be found in the glossary of the STL North Canton Laboratory Quality Manual (LQM), current version .

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL North Canton associates. The following requirements must be met:

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded, other gloves will be cleaned immediately. Viton gloves may be worn when halogenated solvents are used for extractions or sample preparation. Nitrile gloves may be used when other solvents are handled. [Note: VITON is readily degraded by acetone]. When good manual dexterity is needed, for example, when handling small quantities/containers, disposable gloves (such as latex or N-DEX®) shall be used. While these gloves protect against splashes, they give little or no protection against contact with large quantities of solvent, and no protection against spills or immersion.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

Chemicals that have been classified as **carcinogens** or **potential carcinogens** under OSHA include methylene chloride, PCBs, certain pesticides.

The following materials are known to be **corrosive**: Concentrated sulfuric acid, sodium hydroxide, 1N nitric acid.

Sulfuric acid cleanup must not be performed on any matrix that may have water present as a violent reaction between the acid and water may result in acid exploding out of the vessel.

Chemicals known to be **flammable** are: Diethyl ether, acetone, hexane, 2-propanol.
Mercury is a highly toxic compound that must be handled with care. The analyst must be aware of the appropriate handling and clean-up techniques before handling this material.

5.4. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operations will permit. Use of methylene chloride for glassware cleaning should be avoided as far as possible. If more than 500 mL of Methylene chloride is spilled, evacuate the area until the area has been cleaned by a Hazardous Material Team.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Glassware should be cleaned with soap and water, rinsed with water and dried in an oven at 400°C for at least 2 hours. Alternatively the glassware can be solvent rinsed with acetone or methanol followed by methylene chloride after the water rinse.

6.2. Equipment and supplies for extraction procedures

EQUIPMENT AND SUPPLIES	Sep fun.	CLLE	Soni	Sox	Conc
Separatory Funnel: 2 L	√				
Separatory Funnel Rack	√				
Balance: >1400 g capacity, accurate ±1 g	√	√			
pH indicator paper, wide-range: covers extraction pH	√	√			
Graduated cylinder: 1 liter. (other sizes may be used)	√	√			
Erlenmeyer Flask or Fleaker: 125 & 300 mL (other sizes optional)	√		√		
Centrifuge	√				
Auto-Shaker	√				
Methylene Chloride Collection Tank	√	√			

EQUIPMENT AND SUPPLIES	Sep fun.	CLLE	Soni	Sox	Conc
Solvent Dispenser Pump or 100 mL Graduated Cylinder	√		√		
Continuous Liquid/Liquid Extractor		√			
Round or flat Bottom: 250, 500 mL or 1 L		√			
Boiling Chips: Contaminant free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent).		√		√	√
Cooling Condensers		√		√	
Heating Mantle: Rheostat controlled		√		√	
Auto-timer for heating mantle		√		√	
Beakers: 250 & 400 mL, graduated			√		
Balance: >100 g capacity, accurate ±0.1 g			√	√	
Soxhlet Extractor				√	
Sonicator (at least 300 watts)			√		
Sonicator horn, 3/4 inch			√		
Kuderna-Danish (K-D) Apparatus: 500 mL					√
Concentrator Tube: 10 mL, attached to K-D with clips					√
Supply Manifold: Heated, capable of temperature control (± 5°C) up to 95°C.					√
MegaCT: 150 ml					√
Snyder Column: Three-ball macro					√
Water Bath: Heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.					√
Vials: Glass, 2 mL, 4 mL, and 10 mL capacity with Teflon®-lined screw-cap					√
Nitrogen Blowdown Apparatus					√
Nitrogen: reagent grade.					√
Culture tubes: 10 mL, 16 mmx100 mm					√
Syringe: 1 mL	√	√	√	√	
Phase Separation Paper	√	√	√	√	
Glass Wool	√	√	√	√	
Glass Funnel: 75 X 75 mm	√	√	√	√	√
Disposable Pipets	√	√	√	√	√
Aluminum foil	√	√	√	√	√
Paper Towels	√	√	√	√	√
Wrist Shaker					

6.3. Equipment and Supplies for Cleanup Procedures

EQUIPMENT AND SUPPLIES	GPC	Florisil	Sulfur	Acid	A/B	FC
Gel permeation chromatography system (GPC Autoprep Model 1002A or 1002B Analytical Biochemical Laboratories, Inc. or Zymark	√					

EQUIPMENT AND SUPPLIES	GPC	Florisil	Sulfur	Acid	A/B	FC
Benchmate or equivalent).						
Bio Beads: (S-X3) -200-400 mesh, 70 gm (Bio-Rad Laboratories, Richmond, CA, Catalog 152-2750 or equivalent).	√					
Chromatographic column: 700 mm x 25 mm ID glass column. Flow is upward.	√					
Ultraviolet detector: Fixed wavelength (254 nm) and a semi-prep flow-through cell.	√					
Strip chart recorder, recording integrator, or laboratory data system.	√					
Syringe: 10 mL with Luerlok fitting.	√					
Syringe filter assembly, with disposable 5 um filter discs, Millipore No. LSWP 01300 or equivalent.	√					
Chromatographic column: 250 mm long x 10 mm ID; with Pyrex glass wool at the bottom and a Teflon stopcock (for silica gel cleanup).	√					
Vacuum system for eluting multiple cleanup cartridges. Vac Elute Manifold - Analytichem International, J.T. Baker, or Supelco (or equivalent). The manifold design must ensure that there is no contact between plastics containing phthalates and sample extracts.		√				
Vacuum trap made from a 500 mL sidearm flask fitted with a one-hole stopper and glass tubing.		√				
Vacuum pressure gauge.		√				
Rack for holding 10 mL volumetric flasks in the manifold.		√				
Mechanical shaker or mixer: Vortex Genie or equivalent.			√	√		
Separatory Funnels with Ground-Glass Stoppers: 250 mL					√	
Erlenmeyer Flasks: 125 mL					√	
Disposable Pipets		√	√	√	√	√
Culture tubes: 10 mL, 16 mmx100 mm	√	√	√	√	√	√

7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

All reagents must be ACS reagent grade or better unless otherwise specified.

REAGENTS	Sep fun.	CLLE	Soni	Sox	Conc
Sodium hydroxide (NaOH), Pellets: Reagent Grade	√	√			
Sodium hydroxide solution, 10 N: Dissolve 40 g of NaOH in reagent water and dilute to 100 mL.	√	√			
Sulfuric acid (H_2SO_4), Concentrated: Reagent Grade	√	√			
Sulfuric acid (1:1): Carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.	√	√			

REAGENTS	Sep fun.	CLLE	Soni	Sox	Conc
Hydrochloric Acid (HCl)					
Organic free reagent water.	√	√			
Sodium sulfate (Na_2SO_4), Granular, Anhydrous: Purify by heating at 400°C a minimum of two hours.	√	√	√	√	√
Extraction/Exchange Solvents: Methylene chloride, hexane, acetonitrile, acetone, pesticide quality or equivalent	√	√	√	√	√
Acetone: Used for cleaning	√	√	√	√	√

7.2. Reagents for Cleanup Procedures

REAGENTS	GPC	Florisil	Sulfur	Acid	A/B	*FC*
Florisil: 500 mg or 1 g cartridges with stainless steel or Teflon frits (calalog 694-313, Analytichem, 24201 Frampton Ave., Harbor City, CA, or equivalent.)		√				
Mercury: triple distilled			√			
Tetrabutylammonium hydrogen sulfate			√			
Sodium sulfite			√			
Tetrabutylammonium (TBA) sulfite reagent: Prepare reagent by dissolving 3.39 g of Tetrabutylammonium hydrogen sulfate in 100 mL organic-free reagent water. Extract this solution 3 times with 20 mL portions of hexane. Discard the hexane extracts. Add 25 g sodium sulfite to the water solution.			√			
2-Propanol			√			
Nitric acid: 1N			√			
Copper powder: remove oxides (if powder is dark) by treating with 1N nitric acid, rinse with organic-free reagent water to remove all traces of acid, rinse with acetone, and dry under a stream of nitrogen.			√			
Sulfuric acid, Concentrated				√		
Sodium hydroxide, Pellets					√	
Sodium hydroxide, 10N: Dissolve 40 g of NaOH in 100 mL of reagent water					√	
Sulfuric acid (H_2SO_4), Concentrated: Reagent Grade					√	
Sulfuric acid (1:1): Carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.					√	
Fluorocarbon: PF-5080, 3M						√

7.3. Standards

7.3.1. Stock Standards

Stock standards are purchased as certified solutions or prepared from neats. Semivolatile stock standards are stored at ≤ 6°C. All stock standards must be protected from light. Stock standard solutions must be replaced after one year (from the time of

preparation, if prepared in house, or from the time the ampoule is opened if purchased.) Standards must be allowed to come to room temperature before use.

7.3.2. Surrogate Spiking Standards

Prepare or purchase surrogate spiking standards at the concentrations listed in Table 5. Surrogate spiking standards are purchased or prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.3.3. Matrix Spiking and Laboratory Control Spiking Standards.

The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Prepare MS/LCS spiking standards at the concentrations listed in Table 6. Spiking standards are purchased or prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.3.4. GPC calibration solution - prepare or purchase a solution in methylene chloride that contains the following analytes in the concentrations listed below:

Analyte	mg/mL
Corn Oil	25.0
Bis (2-ethylhexyl) phthalate	1.0
Methoxychlor	0.2
Perylene	0.02
Sulfur	0.08

NOTE: Sulfur is not very soluble in methylene chloride, however, it is soluble in warm corn oil. Therefore, one approach is to weigh out the corn oil, warm it, and transfer the weighed amount of sulfur into the warm corn oil. Mix it and then transfer into a volumetric flask with methylene chloride, along with the other calibration compounds. This standard has a lifetime of 6 months.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1 Extraction is initiated within 7 days of the sampling date for aqueous samples, 14 days for solid and waste samples.

8.3.2 For TCLP leachates, extraction is initiated within seven days from when the TCLP Leach tumbling has been completed, excluding the filtration step. If the filtration step requires extended times, this time counts as part of the seven day holding time.

8.3.3. Analysis of the extracts is completed within forty days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS and a matrix spike / matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS / MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD. See policy QA-003 for further definition of the batch.

9.2. Definition of matrix

The possible matrix types are aqueous, soil, waste and TCLP leachate.

9.3. Insufficient Sample

If insufficient sample is available to process a MS/MSD, then a second LCS must be processed. The LCS pair is then evaluated according to the MS/MSD criteria. Use of a LCS pair in place of a MS/MSD must be documented.

9.4. Sample count

Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count. Field samples are included.

9.5. Method Blank

A method blank consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at the same level as the samples. The method blank is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

9.5.1. Aqueous Method Blanks use 1000 mL of reagent water spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.5.2. Solid method blanks use 30 g of sodium sulfate spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.5.3. TCLP method blanks use 250 mL of leachate fluid (100 mL for herbicides) spiked with the surrogates. The leachate may optionally be diluted to 1000 mL with reagent water.

The method blank goes through the entire analytical procedure, including any cleanup steps.

9.6. Laboratory Control Sample (LCS)

Laboratory Control Samples are well-characterized, laboratory generated samples used to monitor the laboratory's day to day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure, including any cleanup steps.

9.6.1. The LCS is made up in the same way as the method blank (See sections 9.5.1 - 9.5.3) but spiked with the LCS standard and the surrogates.

9.7. Surrogates

9.7.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.7.2. Each applicable sample, blank, LCS and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

9.8. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second spiked aliquot of the same sample which is prepared and analyzed along with the sample and matrix spike.

9.9. Initial Demonstration of Capability

The initial demonstration and method detection limit studies described in section 13 must be acceptable before analysis of samples may begin.

9.10. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

9.11. STL North Canton QC Program

Further details of QC and corrective action guidelines are presented in the STL North Canton QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION AND STANDARDIZATION

10.1. On a weekly basis, measure 1.0 mL of solvent into an autovial using a gastight syringe that is manufactured to a certified volume delivery tolerance of ±0.01 mL (1mL total volume). The "standard" autovial is sealed and the top and bottom of the meniscus are marked. The autovials containing the sample extracts are then compared against the "standard" vial to ensure that the final volume is consistently 1.0 ± 0.01mL. If a new box of autovials are used, then the steps are repeated to further ensure that variations due to vial size and shape are minimized. A log is kept of the lot number of the vials and the day the vials were prepared.

10.2. Refer to section 11.8.1.2. for calibration of the GPC. Otherwise this section is not applicable.

11. PROCEDURE

Procedures for separatory funnel liquid/liquid extraction (11.2), continuous liquid/liquid extraction (11.3), sonication extraction (11.4), soxhlet extraction (11.5), waste dilution (11.6), extract concentration (11.7), and extract cleanup (11.8) are presented in this section.

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file. Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Separatory Funnel Liquid/Liquid Extraction of Water Samples.

A flow chart for this procedure is included in Section 17.

11.2.1. Measure the initial sample pH with wide-range pH paper and record on the extraction benchsheet. If sample is a leachate (e.g. TCLP), compare the current pH against leachate log, note on the benchsheet if there is any discrepancy.

11.2.2. The normal sample volume is 1 liter. Other sample volumes may be used to obtain specific reporting limits, and reduced sample volumes, diluted to 1 liter with reagent water, may be used for very dirty samples.

11.2.3. Weigh the sample container on a balance (± 1 g), taring the sample and container. Transfer the sample to the separatory funnel. . Add the appropriate volume of surrogate spiking solution (see Table 3). Also add appropriate volume of matrix spiking solution to any matrix spike / matrix spike duplicate samples (see.Table 4) Mix well.

If the sample is a 600 series analysis rinse the sample bottle with 60 mL methylene chloride and transfer to the separatory funnel. Reweigh the container. Assume a density of 1 g/mL and record the difference as the sample volume on the benchsheet to the nearest milliliter.

Note: Aqueous samples must be determined volumetrically for Ohio VAP samples.

Warning: Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the sample container has been sealed and inverted. Vent into hood away from analysts and other samples.

11.2.3.1. If the entire sample bottle will not be used, transfer the aliquot to the separatory funnel, then add the spiking solutions to the sample in the separatory funnel.

Note: Alternative methods of measurement of sample volume include a) transferring the sample to a measuring cylinder and b) marking a meniscus on the sample bottle and then measuring the volume of water required to fill the bottle to the meniscus after the sample is transferred. The former method is not recommended because of the risk of cross contamination while the latter is not recommended because of poor accuracy. However, either method may be necessary for specific client programs.

11.2.4. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP. Use 1 L of reagent water for method blanks and LCS. The LCS is spiked with the surrogate and matrix spike solutions, the method blank only with the surrogates.

11.2.5. Use 250 mL of leachate for TCLP pesticides and TCLP semivolatiles, measured in a graduated cylinder. An alternative method would be to tare 400ml beaker on the balance and then add 250g to the beaker. Assume a density of 1g/mL and record the volume on the benchsheet. The leachate may be made up to 1 L in volume with reagent water.

11.2.6. For a TCLP method blank,LCS and LCS Dup measure 250 mL of the buffer solution used in the leaching procedure and transfer to the separatory funnel. Add 60 mL of methylene chloride to the separatory funnel. The TCLP leachate may be diluted to approximately 1 liter before extraction if desired.

11.2.7. Adjust sample pH as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH necessary. Recheck the sample with pH paper by dipping a disposable pipette into the sample and wetting the pH paper. Record adjusted pH, spiking volumes and standard numbers on the benchsheet. Return spiking solutions to the refrigerator as soon as possible.

11.2.8. Seal and shake or rotate the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. An autoshaker may be used to shake and rotate the separatory funnel.

Warning: Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.

11.2.9. Allow the organic layer to separate from the water phase until complete visible separation has been achieved (approximately 10 minutes). If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. If the emulsion cannot be broken (recovery of <80% of the methylene chloride*), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in continuous liquid-liquid extraction (Section 11.2.). If this is done, the sample must be extracted as part of a valid CLLE batch.

*Note: 15 - 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 35 mL from the first shake and still be acceptable. Subsequent shakes should recover at least 50 mL of solvent.

11.2.10. Fill a funnel with 10-20 g of anhydrous sodium sulfate. The funnel can be plugged with glass wool or filter paper may be used to hold the sodium sulfate. Drain the solvent extract from the separatory funnel through the prepared filtration funnel into a clean glass container. The extract may be drained directly into the KD flask. Close the stopcock just before the water level begins draining out of the separatory funnel. If the sodium sulfate becomes saturated with water add more to the funnel or replace the existing sodium sulfate with fresh drying agent.

11.2.11. Repeat the extraction process two more times using fresh 60 mL portions of solvent, combining the three solvent extracts in the collection container.

11.2.12. If extraction at a secondary pH is required, adjust the pH of the sample in the separatory funnel to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure with pH paper and record the adjusted pH on the benchsheet. Serially extract with three 60 mL portions of methylene chloride, as outlined in Steps 11.1.7 to 11.1.9. Collect these three extracts in the same container used for the previous fraction.

11.2.13. Rinse the extract residue from the sodium sulfate by pouring 20-30 mL of clean methylene chloride through the funnel and into the collection container.

11.2.14. Dispose of solvent and water remaining in the extractor into the appropriate waste container.

11.2.15. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.7 for concentration and Section 11.8 for cleanup.

11.3. Continuous Liquid/Liquid Extraction from Water Samples.

A flow chart for this procedure is included in Section 17.

11.3.1. Assemble the apparatus. Add 250-400mL of methylene chloride to the extractor body. Add 3 to 5 boiling chips to the round-bottom distilling flask.

11.3.2. Measure the initial sample pH with wide-range pH paper and record on the extraction benchsheet. If sample is a leachate (e.g. TCLP), compare the current pH against leachate log, note on the benchsheet if there is any discrepancy.

11.3.3. Weigh the sample container on a balance (± 1 g), taring the sample and container. Transfer the sample to the continuous liquid/liquid extractor . Add the appropriate volume of surrogate spiking solution (see Table 3). Also add appropriate volume of matrix spiking solution to any matrix spike / matrix spike duplicate samples (see.Table 4) Mix well. If the sample is a 600 series analysis rinse the sample bottle with 60 mL methylene chloride and transfer to the continuous liquid/liquid extractor. Reweigh the container. Assume a density of 1 g/mL and record the difference as the sample volume on the benchsheet to the nearest milliliter.

Note: Aqueous samples must be determined volumetrically for Ohio VAP samples.

Warning: Dichloromethane creates excessive pressure very rapidly! Therefore, during the rinse for 600 series methodology venting should be done immediately after the sample container has been sealed and inverted. Vent into hood away from analysts and other samples.

11.3.3.1. If the entire sample bottle will not be used, transfer the aliquot to the extractor, then add the spiking solutions to the sample in the extractor.

Note: Alternative methods of measurement of sample volume include: a.) transferring the sample to a measuring cylinder and b.) marking a meniscus on the sample bottle and then measuring the volume of water required to fill the bottle to the meniscus after the sample is transferred. The former method is not recommended because of the risk of cross contamination while the latter is not recommended because of poor accuracy. However, either method may be necessary for specific client programs.

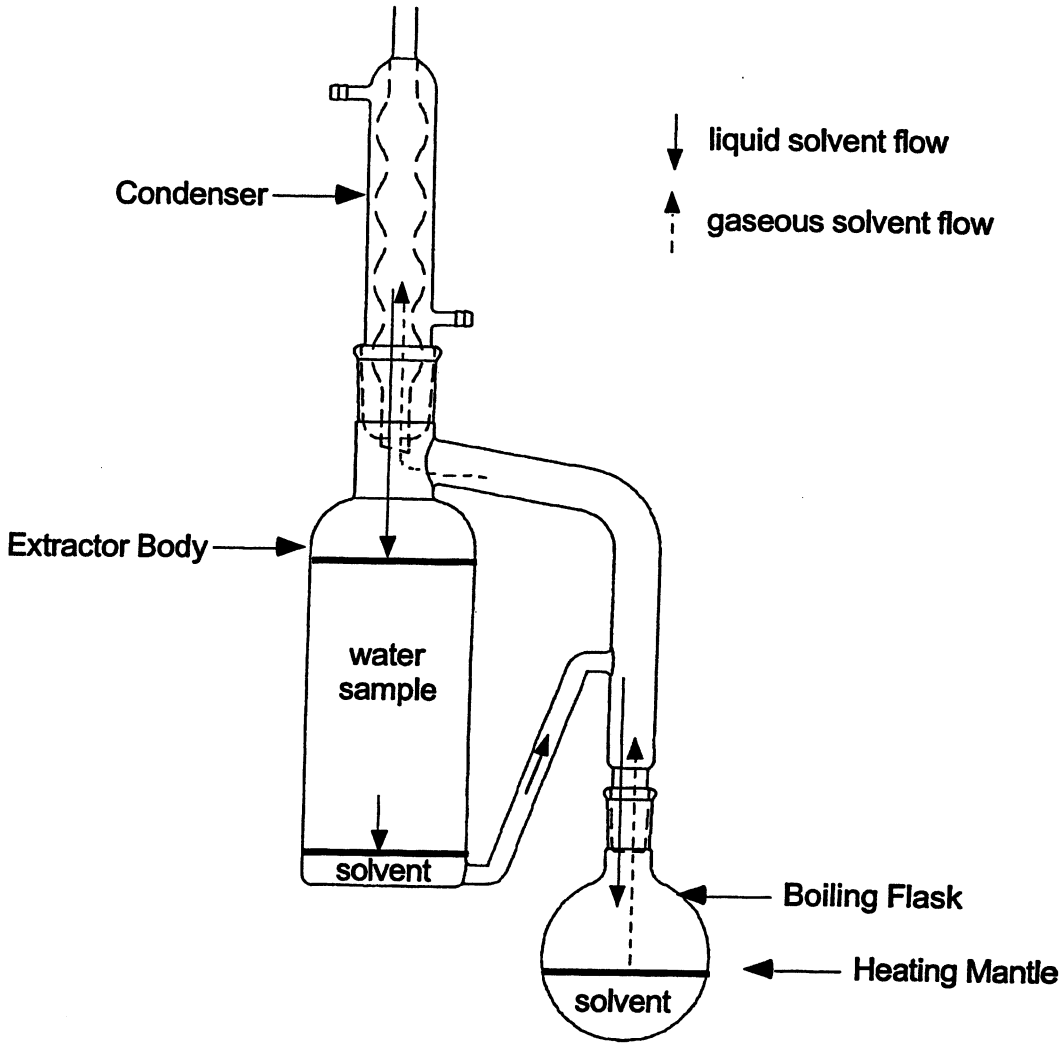
11.3.3.2. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP. Use 1 L of reagent water for method blanks and LCS. The method blank is spiked with the surrogates, the LCS and matrix spikes with the surrogates and matrix spiking solutions..

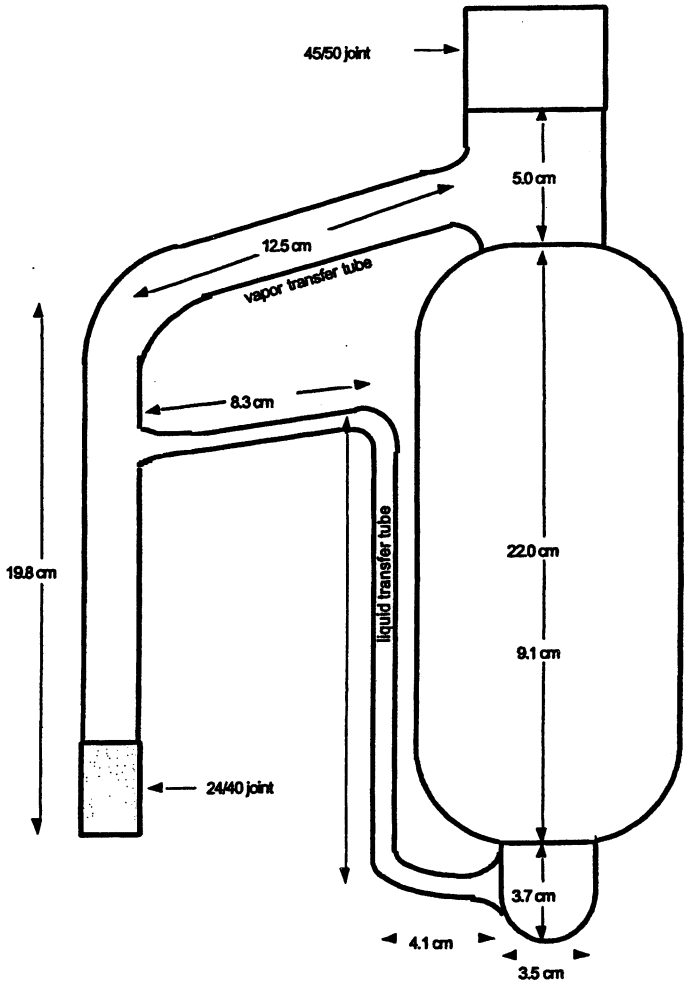
11.3.3.3. Use 250mL of leachate for TCLP for TCLP semivolatiles and TCLP pesticides measured in a graduated cylinder. An alternative method would be to tare 400ml beaker on the balance

and then add 250g to the beaker. Assume a density of 1g/mL and record the volume on the benchsheet. Dilute to about 1 liter with reagent water.

11.3.3.4. For a TCLP method blank, LCS and LCS Dup measure 250 mL of the buffer solution used in the leaching procedure and transfer to the separatory funnel. Dilute to about 1 liter with reagent water.

11.3.3.5. Less than one liter of sample may be used, for highly contaminated samples, or if the reporting limit can be achieved with less than one liter of sample. In this event dilute the sample to about 1 liter with reagent water.




45/50 joint
5.0 cm
12.5 cm
vapor transfer tube
8.3 cm
19.8 cm
liquid transfer tube
22.0 cm
9.1 cm
24/40 joint
3.7 cm
4.1 cm
3.5 cm

11.3.4. Adjust sample pH as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH necessary. Recheck the sample with pH paper. Record adjusted pH, spiking volumes and standard numbers on the benchsheet. Return spiking solutions to the refrigerator as soon as possible.

11.3.5. Add reagent water to the extractor body until approximately 250 mL of methylene chloride is pushed over into the round-bottomed flask to ensure proper operation and solvent cycling. Attach cold condenser (about 10°C). Turn on heating mantle. Inspect joints for leaks once solvent has begun cycling. Extract for 18-24 hours. (24 hours required for 600 series)

11.3.6. If extraction at a secondary pH is required, (see Table 1) turn off the heating mantle and allow the extractor to cool. Detach the condenser and adjust the pH of the sample in the extractor body to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure with pH paper and record the adjusted pH on the benchsheet. Reattach the condenser and turn on heating mantle. Extract for 18-24 hours.

11.3.7. Turn off the heating mantle and allow the extractor to cool.

11.3.8. Place a funnel containing 10-20 g of anhydrous sodium sulfate on the Kuderna-Danish (K-D) apparatus or other glass container. The funnel can be plugged with glass wool enabling it to hold the granular anhydrous sodium sulfate or phase separation filter paper may be used.

11.3.9. Dry the extract in the round bottom flask by filtering it through the sodium sulfate filled funnel. Note that it is not necessary or advisable to attempt to add the solvent remaining in the continuous extractor body to the extract.

11.3.10. Collect the dried extract in a K-D or other glass container. Rinse the flask which contained the solvent extract with 20-30 mL of methylene chloride and add it to the funnel to complete the quantitative transfer. Dispose of solvent and water remaining in the extractor in the appropriate waste container.

Note: Some types of CLLE apparatus have built in drying columns. If this type of apparatus is used then a drying step subsequent to the extraction may not be necessary.

11.3.11. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.7 for concentration and Section 11.8 for cleanup.

11.4. Sonication

A flow chart for this procedure is included in Section 17.

11.4.1. Determination of percent moisture (Optional - if a different group performs this test, refer to the facility SOP.)

In some cases, sample results are needed on a dry weight basis. If this is the case, weigh 5-10 g of sample into a suitable tared container (typically an aluminum weigh pan. Determine the % moisture by drying overnight (at least 12 hours) at 105°C. Allow to cool in a desiccator before weighing.

$$\% \text{ Moisture} = \frac{(\text{weight of wet sample} - \text{weight of dry sample})}{\text{weight of wet sample}} \times 100$$

11.4.2. Determination of pH (Optional - if a different group performs this test, refer to the facility SOP.)

If pH determination is required, transfer 10 g of soil to a beaker. Add 10 mL of water. Stir for five minutes then let stand for 1 hour. Determine the pH of the sample with a glass electrode and pH meter.

11.4.3. Decant and discard any water layer on a sediment/soil sample. Record and document if a water layer was discarded on the benchsheet. Homogenize the sample by mixing it thoroughly in the container. If this is not possible place the sample in clean beaker and homogenize. Upon completion of homogenization in beaker return sample to original container. Discard foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by client. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator).

11.4.4. 11.4.4. Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

11.4.5. Weigh 30 g of sample ± 0.2g into a 250 or 400 mL beaker. Record the weight to the nearest 0.01 g in the appropriate column on the benchsheet. Use 30 g of sodium sulfate for the method blank and 30 g of sodium sulfate with 30 g of reagent sand for the LCS.

11.4.6. Mix weighed sample with a spatula adding enough anhydrous sodium sulfate (approximately 30 g) to be free flowing. (If the sample is not free flowing extraction efficiency may be reduced)

11.4.7. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP.

11.4.8. Add the appropriate volume of surrogate spiking solution (see Table 3). Also add appropriate volume of matrix spiking solution to any matrix spike / matrix spike

duplicate samples (see.Table 4) Add 1 mL of the surrogate spiking solution to each sample, method blank, Laboratory Control Sample (LCS), and matrix spikes. Refer to Table 6 for details of the surrogate spiking solutions. Add 1 mL of the appropriate matrix spiking solution to each Matrix Spike/Matrix Spike Duplicate (MS/MSD) and LCS. Refer to Tables 3 and 5 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

Note: The same volume of surrogate and matrix spiking solution is used if GPC is indicated since the final volume would be reduced to compensate for loss of extract during the GPC procedure.

11.4.9. Immediately add a minimum of 100 mL of solvent to the beaker.

Solvents:

Semivolatile GC/MS Organochlorine pesticides and PCBs	1:1 Methylene Chloride / Acetone
Hydrocarbons (DRO, PAH, etc.) and OPPs	Methylene Chloride

Note: Steps 11.4.5 - 11.4.9 should be performed rapidly to avoid loss of the more volatile extractables.

11.4.10. Place the bottom surface of the appropriate disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.4.11. Sonicate for 3 minutes, making sure the entire sample is agitated. If the W-380 or W-385 sonicator is used the output should be set at 6 for the 3/4 inch high gain (Q) horn or 10 for the 3/4 inch standard horn. with mode switch on pulse, and percent-duty cycle knob set at 50%.

Note: Do ***not*** use ***Microtip*** probe.

11.4.12. Loosely plug the stem of a 75 mm x 75 mm glass funnel with glass wool and/or line the funnel with filter paper. Add 10-20 g of anhydrous sodium sulfate to the funnel cup.

11.4.13. Place the prepared funnel on a collection apparatus (beaker or K-D Apparatus.

11.4.14. Decant and filter extracts through the prepared funnel into a clean beaker or K-D Apparatus.

11.4.15. Repeat the extraction two more times with additional 100 mL minimum portions of solvent each time. Decant off extraction solvent after each sonication. On the final sonication pour the entire sample (sediment and solvent) into the funnel and rinse with an additional 10 mL-20 mL of the methylene chloride/acetone appropriate solvent (Refer to Table in 11.4.9).

Note: Alternatively, the three extracts may be collected together and then filtered through the sodium sulfate.

11.4.16. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.7 for concentration and Section 11.8 for cleanup.

11.4.17. Sonicator Tuning.

11.4.17.1. Tune the sonicator according to manufacturer's instructions. The sonicator must be tuned at least every time a new horn is installed.

11.5. Soxhlet

11.5.1. Determination of % moisture

In some cases, sample results are needed on a dry weight basis. If this is the case, weigh 5-10 g of sample into a suitable tared container (typically an aluminum weigh pan. Determine the % moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing.

$$\% \text{ Moisture} = \frac{(\text{weight of wet sample} - \text{weight of dry sample})}{\text{weight of wet sample}} \times 100$$

11.5.2. Determination of pH

If pH determination is required, transfer 10 g of soil to a beaker. Add 10 mL of water. Stir for 1 hour. Determine the pH of the sample with a glass electrode and pH meter.

11.5.3. Decant and discard any water layer on a sediment/soil sample. Record and document if a water layer was discarded on the benchsheet. Homogenize the sample by mixing it thoroughly in the container. If this is not possible place the sample in clean beaker and homogenize. Upon completion of homogenization in beaker return sample to original container. Discard foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by client. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator).

11.5.4. Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

11.5.5. Weigh 30 g of sample ± 0.2g into a beaker, recording the weight to the nearest 0.01 g on the benchsheet.. Use 30 g of sodium sulfate for the method blank and LCS. Add 30 g of anhydrous sodium sulfate and mix well. The mixture should have a free flowing texture. If not, add more sodium sulfate. Add the sample/sodium sulfate mixture to a soxhlet thimble, but do not pack the thimble tightly. The extraction thimble must drain freely for the duration of the extraction

period. A glass wool plug above and below the sample in the soxhlet extractor is an acceptable alternative for the thimble.

11.5.6. Add the appropriate amount of surrogate and matrix spiking solution as indicated in Tables 3, 4, 5, and 6.

Sample weights less than 30 g but over 5 g may be used if the appropriate reporting limits can be met.

11.5.7. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP, using sodium sulfate as the matrix. The weight of sodium sulfate used should be approximately the weight of soil used in each sample.

11.5.8. Place approximately 200mL of solvent into a 250 mL flat bottom flask containing one or two clean boiling chips. Attach the flask to the extractor and extract the sample for 16-24 hours at 4-6 cycles per hour. Check the system for leaks at the ground glass joints after it has warmed up.

NOTE: If a reduced quantity of sample is extracted, it is usually necessary to increase the amount of sodium sulfate added or increase the solvent boiling rate to properly set the cycling rate.

Solvents:

Semivolatile GC/MS Organochlorine pesticides and PCBs	1:1 Methylene Chloride / Acetone
Hydrocarbons (DRO, PAH, etc.) and OPPs	Methylene Chloride

11.5.9. Allow the extract to cool after the extraction is complete, then disassemble by gently twisting the soxhlet from the flask. Dry the extract in the flask by filtering it through a sodium sulfate filled funnel.

11.5.10. Collect the dried extract in a K-D or other glass container. Rinse the flask which contained the solvent extract with 20-30 mL of methylene chloride and add it to the funnel to complete the quantitative transfer.

11.5.11. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.7 for concentration and Section 11.8 for cleanup.

11.6. Waste Dilution

11.6.1. This method is used for materials that are soluble in an organic solvent.

11.6.2. Transfer 10 mL of the solvent to be used for dilution into a Teflon capped vial. Mark the meniscus on the vial, then discard the solvent.

11.6.3. Tare the vial, then transfer approximately 1g of sample to the vial. Record the weight to the nearest 0.01 g.

11.6.4. Add 1 mL appropriate volume of surrogate solution (Table 3). 11.6.5. Dilute to 10 mL with the appropriate solvent. (Methylene Chloride for GC/MS and Hexane for pesticides pesticide and PCB analysis.)

11.6.6. Add 2 g + 0.1 g sodium sulfate to the sample. Cap and shake for 2 minutes.

11.6.7. Add 4-5 g sodium sulfate to a small funnel. The funnel can be plugged with glass wool or phase separation filter paper may be used to hold the sodium sulfate.

11.6.8. Pour the sample through the funnel, collecting as much as possible in a clean vial. Do NOT rinse the funnel with additional solvent, and do NOT concentrate the sample. The final volume is defined as 10 mL.

11.6.9. Label the sample, which is now ready for cleanup or analysis.

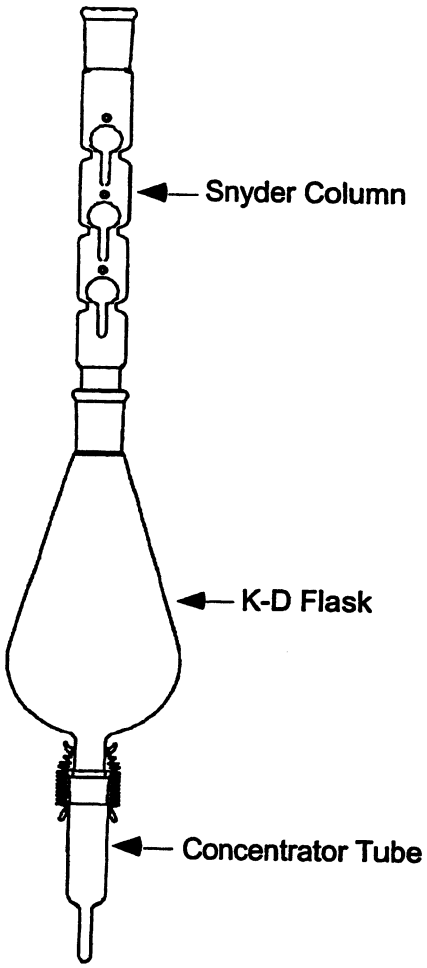
11.7. Concentration

According to the type of sample and any cleanup procedures needed, different final solvents and volumes will be required. Refer to Table 2 for the appropriate final volumes and concentrations.

11.7.1. Kuderna-Danish (KD) Method:

11.7.1.1. Assemble a Kuderna-Danish concentrator by attaching a 10 mL concentrator tube to the 500 mL KD flask. Transfer the sample to the K-D flask.

11.7.1.2. Add one or two clean boiling chips and the extract to be concentrated to the KD flask and attach a three ball Snyder Column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column (this is important to ensure that the balls are not stuck and that the column will work properly).



11.7.1.3. Place the KD apparatus on a water bath (90-98°C) so that the tip of the concentrator tube is submerged. The water level should not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter but the chambers should not flood.

11.7.1.4. Concentrate to 5-15 mL. If the determinative method requires a solvent exchange add the appropriate exchange solvent (50 mL hexane, 20 ml hexane/acetone, or 4 mL acetonitrile,4mL toluene (Herbicide TCLP), or 10 mL toluene) to the top of the Snyder Column, and then continue the water bath concentration back down to 1-4 mL. Refer to

Table 2 for details of exchange solvents and final volumes. The Snyder column may be insulated if necessary to maintain the correct rate of distillation.

Note: Add an additional boiling chip with the addition of exchange solvent. An alternative technique for solvent exchange is to replace the macro Snyder column and KD flask with a micro Snyder column, concentrate to approximately 1 mL, add the appropriate solvent, and concentrate back down to 1 mL. The extract must be cool before the macro Snyder assembly is removed.
Note: It is very important not to concentrate to dryness as analytes will be lost.

11.7.1.5. Remove the KD apparatus from the water bath and allow to cool for a minimum of 10 minutes. If the level of the extract is above the level of the concentrator tube joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes.

11.7.1.6. If the final volume is 5 or 10 mL the extract may be made up to volume in the graduated KD tube or transferred to a 12 mL vial previously marked at the appropriate volume level. Document the final volume. Otherwise proceed to section 11.7.2

11.7.2. Nitrogen Evaporation to Final Concentration

11.7.2.1. Transfer the entire extract to a calibrated evaporation tube. Rinse the concentrator tube with 1-2 mL of the appropriate solvent and transfer the solvent rinsate to the evaporation tube.

11.7.2.2. Place the tube in a warm water bath that is at least 5°C below the boiling temperature of the solvent being evaporated and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but should not create splattering of the extract.

Boiling points of commonly used solvents are:

Methylene chloride	40°C
Acetone	56°C
Hexane	69°C
Acetonitrile	82°C
Toluene	111°C

11.7.2.3. During the course of the evaporation rinse the sides of the evaporation tube twice with approximately 1 mL of clean solvent. The first rinse should be about half way through the process, with the second rinse when the solvent volume gets close to 1 mL. Concentrate the solvent accurately to the calibrated volume line and transfer the extract to the appropriate storage vial.

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.7.2.4. An alternative technique is to follow the previous steps concentrating the solvent to slightly below the required final volume and then drawing the extract into a syringe. Rinse the evaporation tube with a small amount of solvent and draw additional solvent into the syringe to make up the accurate final volume.

Note: It is very important not to concentrate to dryness as analytes will be lost.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

Alternative QES Concentration Method:

This concentration method uses a hot water jacketed concentrator tube (CT) instead of the hot water bath and concentrator tube used in Section 11.7.1.1. The construction of the jacketed concentrator tube reduces the tendency of the extract to evaporate to dryness. Thus, low boiling analytes are retained in the extract better with less analyst monitoring of the concentration process.

Assemble the jacketed concentrator tube, KD body and hot water hoses. Add 1 mL of exchange solvent (if needed) and one large, clean boiling chip.

NOTE: The boiling chips used in the jacketed concentrator tube must be large enough to prevent them falling down into the tip of the CT. If the boiling chip is not in the proper position, the extract may superheat and bump vigorously if the extract solvent warms up slowly.

Pour the extract to be concentrated into the KD flask, and attach a three-ball Snyder column.

Turn on the hot (95°C) water flow to the jacketed concentrator tube.

Concentrate to 1 mL. If the determinative method requires a solvent exchange, add the appropriate exchange solvent (5-10 mL hexane, 1 mL acetonitrile, 4 mL acetone/hexane) to the top of the Snyder column. Continue to concentrate until the Snyder column balls stop chattering.

Cool the jacketed concentrator tube until it is cold to the touch.

Quantitatively transfer the extract and dilute to final volume, or continue concentration with nitrogen evaporation (section 11.7.2).

11.7.3. Turbovap Method

11.7.3.1. Turn on the Turbovap and adjust the water temperature to 5-10°C less than the boiling point of the solvent to be evaporated.

11.7.3.2. Switch all endpoint sensors to the "NO" position.

11.7.3.3. Adjust the water bath level

11.7.3.4. Adjust the nitrogen gas pressure to approximately 12 psi.

11.7.3.5. Transfer the extract into the Turbovap tube and load into the Turbovap. Do not fill the Turbovap tubes over approximately 3/4 full.

11.7.3.6. Reset the sensor and close the lid.

Note: If the extract splashes when the nitrogen flow starts, reduce the nitrogen flow or transfer a portion of the extract back into the original extract container.

11.7.3.7. As the extract concentrates, transfer the remainder of the extract into the appropriate Turbovap tube. After all of the extract has been transferred, rinse the flask with a few mL of methylene chloride and add to the Turbovap tube.

11.7.3.8. During the concentration rinse the Turbovap tube walls with a few mL of solvent 1 or 2 times.

11.7.3.9. Concentrate the extract to slightly less than the required final volume.

11.7.3.10. If solvent exchange is required, concentrate to 1-4 mL and add 50 mL of the exchange solvent. Concentrate back down to the appropriate volume. Refer to table 2 for details of exchange solvents and final volumes.

11.7.3.11. Transfer the concentrated extract to volumetric glassware for adjustment of final volume, using a small amount of solvent to rinse the tube and complete the transfer.

Note: Water contamination from condensation during concentration is not acceptable. If water is present, remove the Turbovap tube and filter the extract through sodium sulfate. Transfer to a clean Turbovap tube and continue the concentration.

Note 2: Dark, opaque or turbid samples may not concentrate. If this occurs, set the concentrator sensor to dryness and supervise the entire concentration procedure.

11.8. Cleanup Techniques

The following techniques may be used to remove interfering peaks, and /or to remove materials that may cause column deterioration and/ or loss of detector sensitivity.

Gel Permeation Chromatography (Section 11.8.1) is a generally applicable technique which can be used to prepare extracts for Semivolatiles (8270), and pesticides (8080) analysis. It is capable of separating high molecular weight material from the sample analytes, and so is particularly useful if tissue or vegetable matter is part of the sample, and for many soil samples.
Note: GPC used only for CLP Projects

Florisil column cleanup (Section 11.8.2) is particularly useful for cleanup of pesticides for analysis by method 8080 and should normally be applied to these samples unless the matrix is clean. It separates compounds with a different polarity from the target analytes.
Note: GPC used only for CLP Projects
Gel Permeation Chromatography and Florisil column cleanup may both be applied to samples for analysis by method 8080. In this case the GPC should be performed first.

Sulfur cleanup (Section 11.8.3) is generally applied to samples for analysis by method 8080, since the Electron Capture Detector responds strongly to sulfur. It is performed after GPC and Florisil cleanup.

Sulfuric acid cleanup (Section 11.8.4) is applied to samples requiring analysis for Polychlorinated Biphenyls (PCBs) only. Most organic matter is destroyed by the sulfuric acid.
WARNING: Sulfuric acid cleanup must not be performed on any matrix that may have water present as a violent reaction between the acid and water may result in acid exploding out of the vessel.

Acid Base Partition Cleanup (Section 11.8.5) is useful for separating organic acids and phenols from basic and neutral organics.

Fluorocarbon cleanup (Section 11.8.7) is used to remove hydrocarbons from water samples to be analyzed for water soluble alcohols.

11.8.1. Gel Permeation Chromatography (GPC)

Note: GPC systems include the GPC Autoprep Model 1002A or 1002B Analytical Biochemical Laboratories, Inc., Zymark Benchmate, or equivalent.

11.8.1.1. GPC Column Preparation

11.8.1.1.1. Weigh out 70 g of Bio Beads (SX-3) into a 400-mL beaker.

11.8.1.1.2. Add approximately 300 mL of methylene chloride and stir gently.

11.8.1.1.3. Cover with aluminum foil and allow the beads to swell for a minimum of two hours. Maintain enough solvent to sufficiently cover the beads at all times.

11.8.1.1.4. Position and tighten the outlet bed support (top) plunger assembly in the tube by inserting the plunger and turning it clockwise until snug. Install the plunger near the column end but no closer than 5 cm (measured from the gel packing to the collar).

11.8.1.1.5. Turn the column upside down from its normal position with the open end up. Place the tubing from the top plunger assembly into a waste beaker below the column.

11.8.1.1.6. Swirl the bead/solvent slurry to get a homogeneous mixture and pour the mixture into the open end of the column. Transfer as much as possible with one continuous pour trying to minimize bubble formation. Pour enough to fill the column. Wait for the excess solvent to drain out before pouring in the rest. Add additional methylene chloride to transfer the remaining beads and to rinse the beaker and the sides of the column. If the top of the gel begins to look dry, add more methylene chloride to rewet the beads.

11.8.1.1.7. Wipe any remaining beads and solvent from the inner walls of the column with a laboratory tissue. Loosen the seal slightly on the other plunger assembly (long plunger) and insert it into the column. Make the seal just tight enough so that any beads on the glass surface will be pushed forward, but loose enough so that the plunger can be pushed forward.

CAUTION: Do not tighten the seal if beads are between the seal and the glass surface because this can damage the seal and cause leakage.

11.8.1.1.8. Push the plunger until it meets the gel, then compress the column bed about 4 cm.

11.8.1.1.9. Connect the column inlet to the solvent reservoir and place the column outlet tube in a waste container. Pump methylene chloride through the column at a rate of 5 mL/min. for one hour.

11.8.1.1.10. After washing the column for at least one hour, connect the column outlet tube to the inlet side of the UV detector. Connect the system outlet to the outlet side of the UV detector. Placing a restrictor (made from a piece of capillary tubing of 1/16"OD x 10/1000"ID x 2") in the outlet tube from the UV detector will prevent bubble formation which causes a noisy UV baseline. The restrictor will not effect the flow rate. After pumping methylene chloride through the column for an additional 1-2 hours, adjust the inlet bed support plunger until approximately 6-10 psi back-pressure is achieved. Push the plunger in to increase pressure or slowly pull outward to reduce pressure.

11.8.1.1.11. When the GPC column is not to be used for several days, connect the column inlet and outlet lines to prevent column drying and/or

channeling. If channeling occurs, the gel must be removed from the column, re-swelled, and re-poured as described above. If drying occurs, pump methylene chloride through the column until the observed column pressure is constant and the column appears wet. Always recalibrate after column drying has occurred to verify that retention volumes have not changed.

11.8.1.2. Initial Calibration of the GPC Column

11.8.1.2.1. Before use, the GPC must be calibrated based on monitoring the elution of standards with a UV detector connected to the GPC column.

11.8.1.2.2. Pump solvent through the GPC column for 2 hours. Verify that the flow rate is 4.5-5.5 mL/min. Corrective action must be taken if the flow rate is outside this range. Record the column pressure (should be 6-10 psi) and room temperature (22°C is ideal).

Note: Changes in pressure, solvent flow rate, and temperature conditions can affect analyte retention times and must be monitored. If the flow rate and/or column pressure do not fall within the above ranges, a new column should be prepared.

11.8.1.2.3. Inject the calibration solution and retain a UV trace that meets the following requirements (See resolution calculation in section 11.8.1.6):

- Peaks must be observed and should be symmetrical for all compounds in the calibration solution.
- Corn oil and phthalate peaks must exhibit >85% resolution.
- Phthalate and methoxychlor peaks must exhibit >85% resolution.
- Methoxychlor and perylene peaks must exhibit >85% resolution.
- Perylene and sulfur peaks must not be saturated and must exhibit >90% baseline resolution.

11.8.1.2.4. A UV trace that does not meet the criteria in paragraph 11.8.1.2.3 indicates the need for system maintenance and/or the need for a new column.

11.8.1.2.5. Determine appropriate collect and dump cycles.

11.8.1.2.6. The calibrated GPC program for pesticides/PCB should dump >85% of the phthalate and should collect >95% of the methoxychlor and perylene. Use a wash time of 10 minutes.

11.8.1.2.7. For semivolatile extracts, initiate a column eluate collection just before the elution of bis (2-ethylhexyl) phthalate and after the elution of the corn oil. Stop eluate collection shortly after the elution of perylene. Stop collection

before sulfur elutes. Use a wash time of 10 minutes after the elution of sulfur.

11.8.1.2.8. Reinject the calibration solution after appropriate dump and collect cycles have been set.

11.8.1.2.9. Measure and record the volume of collected GPC eluate in a graduated cylinder.

11.8.1.2.10. The retention times for both bis(2-ethylhexyl) phthalate and perylene must not vary more than +/- 5% between calibrations.

11.8.1.3. GPC calibration check

Check the calibration of the GPC immediately after the initial calibration and at least every 7 days thereafter, while the column is in use.

11.8.1.3.1. Inject the calibration solution, and obtain a UV trace. If the retention times of bis(2-ethylhexyl)phthalate or perylene have changed by more than ± 5% use this run as the start of a new initial calibration. Otherwise, proceed with the recovery check. Excessive retention time shifts may be caused by poor laboratory temperature control or system leaks, an unstabilized column, or high laboratory temperature causing outgassing of methylene chloride. Pump methylene chloride through the system and check the retention times each day until stabilized.

11.8.1.4. GPC Recovery Check for Pesticides/ PCBs

11.8.1.4.1. The recovery from the GPC must be verified immediately after the initial calibration and at least every 7 days, when the instrument is in use. Two recovery check solutions are used. The first mixture is prepared by diluting 1.0 2.0 mL of the pesticide matrix spiking solution (Table 6) to 10 mL in methylene chloride. The second mixture is prepared by diluting 12.0 mL of the PCB only matrix spiking solution (Table 6) to 10 mL with methylene chloride.

11.8.1.4.2. Load the pesticide matrix spike mixture, the PCB mixture, and a methylene chloride blank onto the GPC using the GC dump and collect values.

Note: If the analysis is for PCBs only, then the pesticide recovery check is not necessary.

11.8.1.4.3. After collecting the GPC calibration check fraction, concentrate, solvent exchanging to hexane. Adjust the final volume to 5.0 mL, and analyze by GC/EC. Refer to concentration, section 11.7.

11.8.1.4.4. The methylene chloride blank may not exceed more than one half the reporting limit of any analyte. And if the recovery of each of the single component analytes is 80-10120% and if the Aroclor pattern is the same as previously run standards, then the analyst may use the column. If the above criteria are not met, there may be a need for system maintenance.

11.8.1.5. GPC Recovery Check for Semivolatiles

11.8.1.5.1. The recovery from the GPC must be verified immediately after the initial calibration and at least every 7 days, when the instrument is in use. Dilute 1.0 mL of the semivolatiles matrix spiking solution (Table 6) to 10 mL in methylene chloride.

11.8.1.5.2. Load the matrix spike mixture and a methylene chloride blank onto the GPC using the semivolatiles dump and collect values.

11.8.1.5.3. After collecting the GPC recovery check fraction, concentrate to 0.5 mL, and analyze by GC/MS. Refer to the concentration section 11.7.

Recovery of the matrix spike analytes should be at least 60%. The blank should not contain any analytes at or above the reporting limit. If these conditions are met the column may be used for sample analysis. Otherwise correct the contamination problem, or extend the collect time to improve recovery of target analytes. THIS IS NOT SPECIFIED OR REQUIRED BY THE SOW.

11.8.1.6. Sample Extract Cleanup

11.8.1.6.1. Reduce the sample extract volume to 1-2 mL, then adjust to 10 mL with methylene chloride prior to cleanup. This reduces the amount of acetone in the extract. Refer to section 11.7.

11.8.1.6.2. Start the pump and let the flow stabilize for 2 hours. The solvent flow rate should be 4.5-5.5 mL/min. The ideal laboratory temperature to prevent outgassing of the methylene chloride is 22°C. The normal backpressure is 6-10 psi.

11.8.1.6.3. In order to prevent overloading of the GPC column, highly viscous sample extracts must be diluted prior to cleanup. Any sample extract with a viscosity greater than that of a 1:1 glycerol:water solution (by visual comparison) must be diluted and loaded into several loops.

11.8.1.6.4. Samples being loaded onto the GPC should be filtered with a 5 micron (or less) filter disk. Attach a filter to a 10 mL Luerlok syringe and filter the 10 mL sample extract into the sample tube.

Note: The Zymark Benchmate automatically filters the sample extracts, but manual prefiltration of very dirty extracts may be necessary.

11.8.1.6.5. Load the filtered (or unfiltered if using the Benchmate) samples into the proper sample tubes and place on the GPC.

Note: For the GPC Autoprep Model 1002A, wash the loading port with methylene chloride after loading each sample loop in order to minimize cross contamination. This step is automated on the GPC Autoprep 1002B and on the Benchmate.

11.8.1.6.6. Set the collect, dump, and wash times determined by the calibration procedure.

11.8.1.6.7. Switch to the run mode and start the automated sequence. Process each sample using the collect and dump cycle times established by the calibration procedure.

11.8.1.6.8. Collect each sample in a suitable glass container. Monitor sample volumes collected.

11.8.1.6.9. Any samples that were loaded into 2 or more positions must be recombined.

11.8.1.6.10. Concentrate semivolatile sample extracts to 0.5 mL in methylene chloride. Refer to the concentration section 11.7.

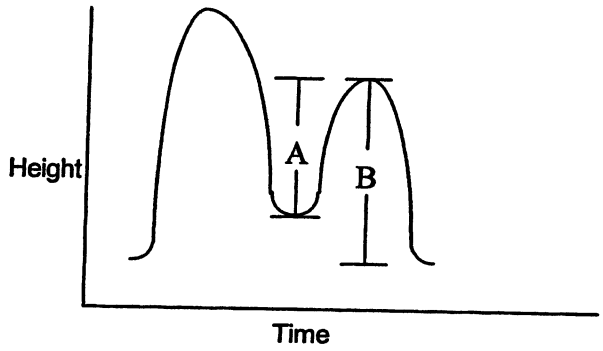
11.8.1.6.11. Solvent exchange pesticide/PCB sample extracts into hexane and concentrate to 5.0 mL. Refer to the concentration section 11.7.

11.8.1.7. Calculations

11.8.1.7.1. Resolution

To calculate the resolution between two peaks on a chromatograph, divide the depth of the valley between the peaks by the peak height of the smaller peak being resolved and multiply by 100.

Resolution Calculation



$$\% \text{ Resolution} = \frac{A}{B} \times 100$$

Where: A = depth of valley to height of smaller peak
B = peak height of smaller peak

11.8.1.7.2. Dump Time

Mark on the chromatograph the point where collection is to begin. Measure the distance from the injection point. Divide the distance by the chart speed. Alternatively the collect and dump times may be measured by means of an integrator or data system.

$$\text{Dump time (min)} = \frac{\text{Distance (cm) from injection to collection start}}{\text{Chart speed (cm / min)}}$$

11.8.1.7.3. Collection Time

$$\text{Collection time (min)} = \frac{\text{Distance (cm) between collection start and stop}}{\text{Chart speed (cm / min)}}$$

11.8.2. Florisil Cartridge Cleanup

Florisil cleanup is generally used for organochlorine pesticides, although it may be applied to other analytes. Sections 11.8.2.1 through 11.8.2.8 outline the procedure for organochlorine pesticides, while section 11.8.2.9 outlines modifications required for other analytes.

Note 1: Systems for eluting multiple cleanup cartridges include the Supelco, Inc. Solid Phase Extraction (SPE) assemblyor equivalent.

Note 2: Follow the lab specific procedure when using ABC Model 1002B.

11.8.2.1. Before Florisil cleanup sample volume must be reduced to 10 mL (5 mL if GPC cleanup was used) and the solvent must be hexane. Refer to Section 11.7 for details of concentration.

11.8.2.2. Attach a vacuum manifold to a vacuum pump or water aspirator with a trap installed between the manifold and the vacuum. Adjust the vacuum in the manifold to 5-10 psi.

11.8.2.3. Place one Florisil cartridge into the vacuum manifold for each sample extract. Prior to cleanup of samples, pre-elute each cartridge with 5 mL of hexane/acetone (9:1). Adjust the vacuum applied to each cartridge so that the flow through each cartridge is approximately 2 mL/min. Do not allow the cartridges to go dry.

11.8.2.4. Just before the cartridges go dry, release the vacuum to the manifold and remove the manifold top.

11.8.2.5. Place a rack of clean labeled 12 mL concentrator tubes into the manifold and replace the manifold top. Make sure that the solvent line from each cartridge is placed inside the appropriate tube.

11.8.2.6. After the clean tubes are in place, vacuum to the manifold is restored and 2.0 mL of the extract is added to the appropriate Florisil cartridge.

11.8.2.7. The pesticides/aroclors in the extract concentrates are then eluted through the column with 8 mL of hexane/acetone (90:10) and are collected into the 10 mL culture tube or concentrator tube held in the rack inside the vacuum manifold.

11.8.2.8. Transfer the extract to a graduated concentrator tube and concentrate the extract to 2 mL. Refer to the concentration section. (11.7)

Note 1: A cartridge performance standard must be run with each lot of Florisil cartridges.

Note 2: Florisil cartridge performance check--every lot number of Florisil must be tested before use. Add 0.5 ug/mL of 2,4,5-trichlorophenol solution and 0.5 mL of GC Standard Mix A (midpoint concentration) to 4 mL hexane. Reduce volume to 0.5 mL. Add the concentrate to a pre-washed Florisil cartridge and elute with 9 mL hexane/acetone [(90:10)(v/v)]. Rinse cartridge with 1.0 mL hexane two additional times. Concentrate eluate to 1.0 mL final volume and transfer to vial. Analyze the solution by GC/EC. The test sample must show 80 to 120% recovery

of all pesticide analytes with <5% trichlorophenol recovery, and no peaks interfering with target compounds can be detected. This standard has a lifetime of six months. Alternatively, this standard may be purchased as a stock solution.

11.8.2.9. Modifications for other analyte classes

11.8.2.9.1. PCBs

Pre-elute the cartridge with 4 mL hexane. Add 2 mL of the sample extract and elute with 3 mL hexane. The eluant will contain the PCBs together with any heptachlor, aldrin, 4,4'DDE and part of any 4,4'DDT. Any BHC isomers, heptachlor epoxide, chlordane, endosulfan I and II, endrin aldehyde and endrin sulfate and methoxychlor will be retained on the column and can be eluted in a separate fraction with 8 mL 90:10 hexane:acetone if required.

11.8.3. Sulfur Removal

11.8.3.1. Sulfur can be removed by one of three methods: mercury, copper, or tetrabutylammonium sulfite (TBA) according to laboratory preference. If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract to settle the crystals, and carefully draw off the sample extract with a disposable pipet, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean concentrator tube before proceeding with further sulfur cleanup.

11.8.3.2. Sulfur Removal with Elemental Mercury

Note: Use Mercury sparingly in order to minimize exposure and disposal costs.

11.8.3.2.1. Transfer 2 mL of sample extract into a clean concentrator tube or Teflon sealed vial.

11.8.3.2.2. Add one to three drops of mercury to the extract vial and seal.

11.8.3.2.3. Shake well for 15-30 seconds. If prolonged shaking is required, use a mechanical shaker.

11.8.3.2.4. Remove the extract from the mercury using a disposable pipette and transfer to a clean vial.

11.8.3.2.5. If black precipitate forms, sulfur was present. Shake again, then centrifuge. After centrifugation, transfer the supernate to a clean test tube and repeat. Do this until relatively little precipitate remains, or the screens indicate that cleanup is complete.

11.8.3.2.6. Properly dispose of the mercury waste.

11.8.3.3. Sulfur Removal with Copper

11.8.3.3.1. Transfer 1.0 mL of sample extract into a centrifuge or concentrator tube.

11.8.3.3.2. Add approximately 2 g of cleaned copper powder (see 7.2 for copper cleaning procedure) to the sample extract tube.

11.8.3.3.3. Mix for one minute on a mechanical shaker.

11.8.3.3.4. If the copper changes color, sulfur was present. Repeat the sulfur removal procedure until the copper remains shiny.

11.8.3.3.5. Transfer the supernate to a clean vial.

11.8.3.4. Sulfur Removal with Tetrabutylammonium (TBA) Sulfite Reagent

11.8.3.4.1. Transfer 1.0 mL of sample extract into a culture tube.

11.8.3.4.2. Add 1.0 mL TBA sulfite reagent and 2 mL 2-propanol to the sample extract. Cap and shake for 1 minute. If clear crystals (precipitated sodium sulfite) form, sufficient sodium sulfite is present.

11.8.3.4.3. If a precipitate does not form, add sodium sulfite in approximately 0.1 g portions until a solid residue remains after repeated shaking.

11.8.3.4.4. Add 5 mL organic free reagent water and shake for 1 minute. Allow sample to stand for 5-10 minutes. (Centrifuge if necessary to separate the layers). Transfer the sample extract (top layer) to a vial. The final volume is defined as 1.0 mL in section 11.8.3.4.1.

11.8.4. Sulfuric Acid Cleanup

11.8.4.1. Add approximately 2-5 mL of concentrated sulfuric acid to 2 mL of sample extract in a Teflon capped vial.

Caution: There must be no water present in the extract or the reaction may shatter the sample container.

11.8.4.2. Shake or vortex for about thirty seconds and allow to settle. (Centrifuge if necessary)

11.8.4.3. Remove the sample extract (top layer) from the acid using a Pasteur pipet and transfer to a clean vial. ***CAUTION:*** It is not necessary to remove all the extract since the final volume is already determined. Transfer of small amounts of sulfuric acid along with the extract will result in extremely rapid degradation of the chromatographic column.

11.8.4.4. If the sulfuric acid layer becomes highly colored after shaking with the sample extract, transfer the hexane extract to a clean vial and repeat the cleanup procedure until color is no longer being removed by the acid, or a maximum of 5 acid cleanups.

11.8.4.5. Properly dispose of the acid waste.

11.8.5. Acid/Base Partition Cleanup

11.8.5.1. Place 10 mL of the solvent extract from a prior extraction procedure into a 125 mL separatory funnel.

11.8.5.2. Add 20 mL of methylene chloride to the separatory funnel.

11.8.5.3. Slowly add 20 mL of DI water which has been previously adjusted to a pH of 12 to 13 with 10 N sodium hydroxide.

11.8.5.4. Seal and shake the separatory funnel for at least two minutes with periodic venting to release excess pressure.

CAUTION: Methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the separatory funnel has been sealed.

11.8.5.5. Allow the organic layer to separate from the aqueous phase for a minimum of ten minutes.

11.8.5.6. If an emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods.

11.8.5.7. Separate the aqueous phase and transfer it to a 125 mL Erlenmeyer flask. Repeat the extraction two more times using fresh 20 mL aliquots of dilute sodium hydroxide (pH 12-13). Combine the aqueous extracts.

11.8.5.8. Water-soluble organic acids and phenols will be primarily in the aqueous phase. Base/neutral analytes will be in the methylene chloride. If the analytes of interest are only in the aqueous phase discard the methylene chloride and proceed to Section 11.8.5.9. If the analytes of interest are only in the methylene chloride, discard the aqueous phase and proceed to Section 11.8.5.11.

11.8.5.9. Externally cool the flask with ice while adjusting the aqueous phase to a pH of 1-2 with sulfuric acid (1:1). Transfer the cool aqueous phase to a clean 125 mL separatory funnel.

11.8.5.10. Add 20 mL of methylene chloride to the separatory funnel and shake for at least two minutes. Allow the methylene chloride to separate from the aqueous phase and collect the

methylene chloride in an Erlenmeyer flask. Repeat the extraction two more times using fresh methylene chloride and extracting at pH 1-2. Combine the three extracts.

11.8.5.11. Dry the extract by passing through a funnel containing 10-20 g anhydrous sodium sulfate. Rinse the funnel with an additional 20-30 mL of clean methylene chloride

11.8.5.12. Cover with aluminum foil if the extract is not concentrated immediately. Refer to section 11.7 for concentration.

11.8.5.13. Dispose of solvent and water remaining in the separatory funnel into the appropriate waste container.

11.8.7. Fluorocarbon Cleanup

This procedure is appropriate for the removal hydrocarbons from water samples prior to analysis for water soluble alcohols

11.8.7.1. Transfer 1-2 mL of water sample to a 10 mL culture tube.

11.8.7.2. Add 1-2 mL of fluorocarbon solvent (PF-5080) to the culture and cap.

11.8.7.3. Shake for 30-60 seconds.

11.8.7.4. After the two phases have separated, pipette about 1 mL of water sample (top layer) into an autosampler vial for analysis. If necessary, centrifuge to enhance the phase separation.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Method detection limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The procedure for the determination of the method detection limit is given in STL North Canton QA Policy #: QA-005

13.2. Initial demonstration

Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC Check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which should contain all the analytes of interest. The spiking level should be equivalent to a mid-level calibration. (For certain tests more than one set of QC check samples may be necessary in order to demonstrate capability for the full analyte list.)

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.2.2. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs. (CORP-GC-0001, CORP-MS-0001, 0002)

13.3. Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

Within the constraints of following the methodology in this SOP, use of organic solvents should be minimized.

15. WASTE MANAGEMENT

Waste generated in this procedure will be segregated, and disposed according to the facility hazardous waste procedures. The Laboratory Environmental, Health and Safety Coordinator should be contacted if additional information is required.

15.1. Solvent waste must be disposed of in the clearly labeled waste cans.

15.2. Acid waste must be collected in the clearly labeled acid waste containers.

15.3. Solid materials (soil, gloves, soiled paper products) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.4. Methylene chloride saturated water and remaining sample after the extraction is dumped into Methylene chloride waste cans.

16. REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III (December 1996). Sections 3500B, 3510C, 3520C, 3540C, 3550B, 3600C, 3610B, 3620B, 3640A, 3650B, 3660B, AND 3665A.

17. MISCELLANEOUS

17.1. Modifications from Reference method

17.1.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.1.2. Aqueous sample volumes may be determined by weight.

17.1.3. Spiking levels for method 608 have been reduced by a factor of ten to bring the levels within the normal calibration range of the instrument.

17.1.4. 10 g of soil is used for pH determination, rather than the 50 g suggested in the reference method. The volume of water is also adjusted to maintain the sample / water ratio specified in the method.

17.2. Modifications from previous revision

17.2.1. SOP change forms are on record in the Quality Assurance Department.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4. Tables

Table 1
Liquid /Liquid Extraction Conditions

Determinative Method	Initial Ext. pH	Secondary Ext. pH
BNA: 8270C[1]	1-2	11-12
625	11-12	1-2
Pest/PCB: 8081A, 8082 & 608	5-9	None
OPP: 8141A	as received	None
Hydrocarbons: 8015B	as received	None
PAH: 8310, & 610	as received	None

[1] If the laboratory has validated acid only 8270 extraction for the target compound list required then the base extraction step may be omitted. The required validation consists of a 4 replicate

initial demonstration of capability and a method detection limit study. (See section 13). Additionally, either of the base or acid fractions of Method 8270 can be run first.

Table 2
Exchange Solvents and Final Volumes

Final Volumes and Exchange Solvents if no cleanup is used		
Type	Exchange Solvent for Analysis	Final Volume for Analysis in mL
Semivolatiles	N/A	1.0 or 5.0* 2.0 for H2O**
PCB	Hexane	10.0 for solids 5.0 for H2O 2.0 for H2O**
Pesticides	Hexane	10.0 for solids 5.0 for H2O
PAH by HPLC	Acetonitrile	1.0 mL
TPH	N/A	1.0
OPP	Hexane/Acetone	2.0

*Requires high sensitivity mass spec tune (refer to NC-MS-0015 PAH by SIM)
** Michigan work requires a final volume of 2 mL.

Final Volumes and exchange solvents if GPC cleanup is used CLP ONLY			
Type	Exchange Solvent for GPC	Final Volume for GPC	Final Volume and solvent for Analysis
Semivolatiles Semivolatiles	CH_2Cl_2 CH_2Cl_2	10 mL[1] 10 mL[1]	0.5 mL CH_2Cl_2 -OLM03.1 2.5 mL CH_2Cl_2 -OLM04.2
Pesticides	CH_2Cl_2	10 mL[1]	5 mL, hexane

[1] Final volume for GPC may be 4 mL if a 2 mL sample loop is used

Final volumes and exchange solvents if Florisil cleanup is used CLP ONLY			
Type	Exchange Solvent for Florisil	Final Volume for Florisil	Final Volume and solvent for Analysis
Pesticides	Hexane	10 mL (2 mL aliquot used)	2 mL, hexane

Final volumes and exchange solvents if both GPC and Florisil cleanup are used CLP ONLY					
Type	Exchange Solvent for GPC	Final Volume for GPC	Exchange Solvent for Florisil	Final Volume for Florisil	Final volume for analysis
Pesticides	Methylene Chloride	10 mL	Hexane	5 mL (2 mL aliquot used)	2 mL, hexane

Note: Different final volumes may be necessary to meet special client reporting limit requirements.

Table 3		
Surrogate Spiking Solutions		
Analyte Group	Surrogate Spike Solution ID	Volume (mL)
BNA	100/150 ppm BNA	0.5
BNA-SIM	100/150 ppm BNA	0.05
BNA-Michigan H2O	100/150 ppm BNA	0.2
Pest	0.2 ppm DCB/TCX	1.0
TPH	40ng C9	1.0
PCB	0.2 ppm DCB/TCX	1.0
PAH	1.0 ug/mL p-Terphenyl-d14 5.0 ug/mL Benzo(e)pyrene	1.0
OPP	10 ug/mL Triphenyl Phosphate	1.0

Table 4		
Matrix Spike and LCS Solutions		
Analyte Group	Matrix Spike Solution ID	Volume (mL)
BNA, BNA TCLP, NPDES	100 ppm BNA All-Analyte Spike	0.5
BNA- Michigan H2O	100 ppm BNA All-Analyte Spike	0.2
BNA NPDES	100 ppm BNA All-Analyte Spike	1.0
Pest	Pest NPDES Spike	1.0
Pest TCLP	Pest TCLP Spike	1.0
Pest NPDES	Pest NPDES Spike	1.0
PCB	10 ppm PCB Spike	1.0

Table 5
Surrogate Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
BNA	2-Fluorobiphenyl	Methanol	100
	Nitrobenzene-d5		100
	p-Terphenyl-d14		100
	2-Fluorophenol		150
	Phenol-d6		150
	2,4,6-Tribromophenol		150
	1,2-Dichlorobenzene-d4		100
	2-Chlorophenol-d4		150
Pest/PCB	Decachlorobiphenyl	MethanolAcetone	0.2
	Tetrachloro-m-xylene		0.2
TPH	Nonane (C9)	Methanol	40.0
PAH	p-Terphenyl-d-14	CH3CN	1.0
	Benzo(e)pyrene		5.0
OPP	Triphenylphosphate	Acetone	10.0

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
TCL BNA	Acenaphthene	Methanol	100
	4-Chloro-3-Methylphenol		150
	2-Chlorophenol		150
	1,4-Dichlorobenzene		100
	2,4-Dinitrotoluene		100
	4-Nitrophenol		150
	N-Nitroso-Di-n-Propylamine		100
	Pentachlorophenol		150
	Phenol		150
	Pyrene		100
	1,2,4-Trichlorobenzene		100
BNA TCLP	1,4-Dichlorobenzene	Methanol	100
	2,4-Dinitrotoluene		100
	Hexachlorobenzene		100
	Hexachlorobutadiene		100
	Hexachloroethane		100
	2-Methylphenol		100
	3-Methylphenol		100
	4-Methylphenol		100
	Nitrobenzene		100
	Pentachlorophenol		100
	Pyridine		100
	2,4,5-Trichlorophenol		100
	2,4,6-Trichlorophenol		100

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
BNA NPDES		Methanol	
	Acenaphthene		100
	Acenaphthylene		100
	Anthracene		100
	Benzo(a)anthracene		100
	Benzo(b)fluoranthene	Methanol	100
	Benzo(k)fluoranthene		100
	Benzo(a)pyrene		100
	Benzo(ghi)perylene		100
	Benzyl butyl phthalate		100
	Bis(2-chloroethyl)ether		100
	Bis(2-chloroethoxy)methane		100
	Bis(2-ethylhexyl)phthalate		100
	Bis(2-chloroisopropyl)ether		100
	4-Bromophenyl phenyl ether		100
	2-Chloronaphthalene		100
	4-Chlorophenyl phenyl ether		100
	Chrysene		100
	Dibenzo(a,h)anthracene		100
	Di-n-butylphthalate		100
	1,3-Dichlorobenzene		100
	1,2-Dichlorobenzene		100
	1,4-Dichlorobenzene		100
	3,3'-Dichlorobenzidine		100

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	Diethyl phthalate		100
	Dimethyl phthalate		100
	2,4-Dinitrotoluene		100
	2,6-Dinitrotoluene		100
	Di-n-octylphthalate		100
	Fluoranthene		100
	Fluorene		100
	Hexachlorobenzene		100
	Hexachlorobutadiene		100
	Hexachloroethane		100
	Indeno(1,2,3-cd)pyrene		100
	Isophorone		100
	Naphthalene		100
	Nitrobenzene		100
	N-Nitrosodi-n-propylamine		100
	Phenanthrene		100
	Pyrene		100
	1,2,4-Trichlorobenzene		100
	4-Chloro-3-methylphenol		100
	2-Chlorophenol		100
	2,4-Dichlorophenol		100
	2,4-Dimethylphenol		100
	2,4-Dinitrophenol		100
	2-Methyl-4,6-dinitrophenol		100

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	2-Nitrophenol		100
	4-Nitrophenol		100
	Pentachlorophenol		100
	Phenol		100
	2,4,6-Trichlorophenol		100
Pest TCLP	Heptachlor	MethanolAcetone	0.5
	Heptachlor epoxide		0.5
	Lindane		0.5
	Endrin		0.5
	Methoxychlor		1.0
Pest NPDES /Pest	Aldrin	MethanolAcetone	1.0
	alpha-BHC		1.0
	beta-BHC		1.0
	delta-BHC		1.0
	gamma-BHC (Lindane)		1.0
	4,4'-DDD		1.0
	4,4'-DDE		1.0
	4,4'-DDT		1.0
	Dieldrin		1.0
	alpha-Endosulfan		1.0
	beta-Endosulfan		1.0

Table 6			
Matrix Spike Components			
Type	Compounds	Solvent	Conc. (µg/mL)
	Endosulfan Sulfate		1.0
	Endrin		1.0
	Heptachlor		1.0
	Heptachlor Epoxide		1.0

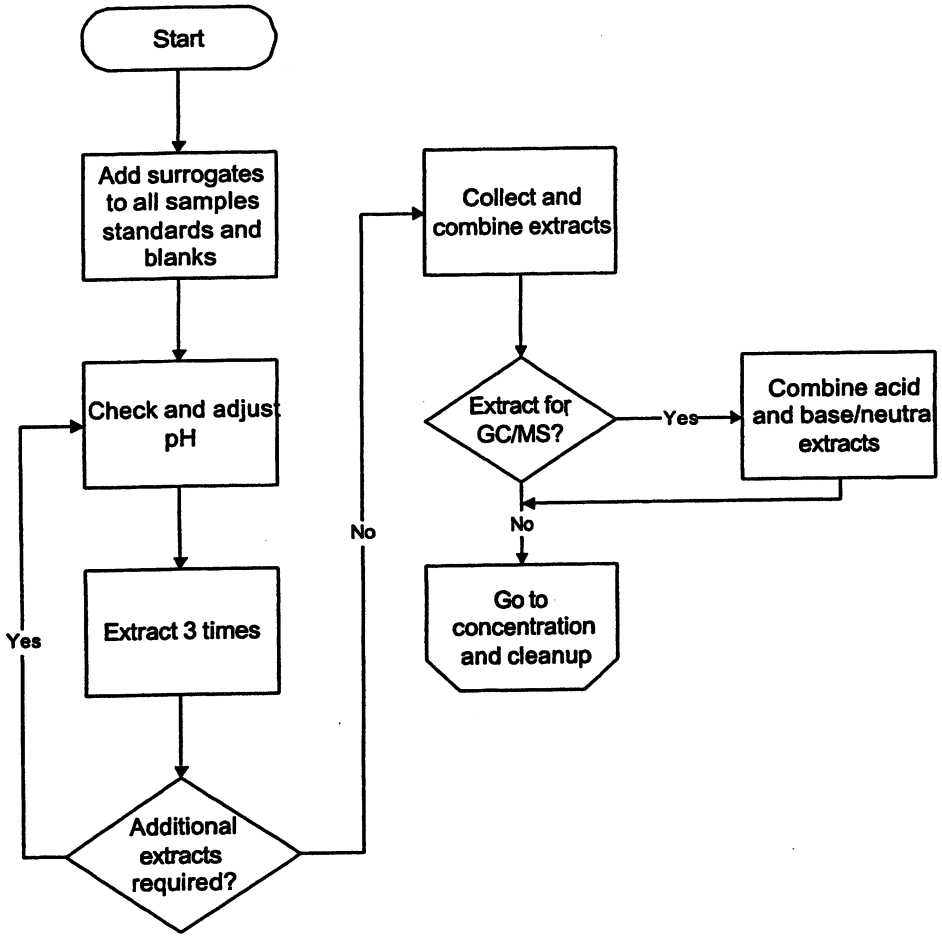
Diesel Range Organics (8015B) Spike	
Compound	**Final Concentration**
n-decane	50 µg/ml
n-dodecane	50 µg/ml
n-tetradecane	50 µg/ml
n-hexadecane	50 µg/ml
n-octadecane	50 µg/ml
n-eicosane	50 µg/ml
n-docosane	50 µg/ml
n-tetracosane	50 µg/ml
n-hexacosane	50 µg/ml
n-octacosane	50 µg/ml

Organophorphorous Pesticides (8141A)	
Compound	**Final Concentration**
dimethoate	20 µg/mL
disulfoton	20 µg/mL
famphur	20 µg/mL
methyl parathion	20 µg/mL
parathion (ethyl)	20 µg/mL
phorate	20 µg/mL
sulfotepp	20 µg/mL
thionazin	20 µg/mL
o,o,o-triethyl phosphorothioate	20 µg/mL
triphenylphosphate (surrogate)	20 µg/mL

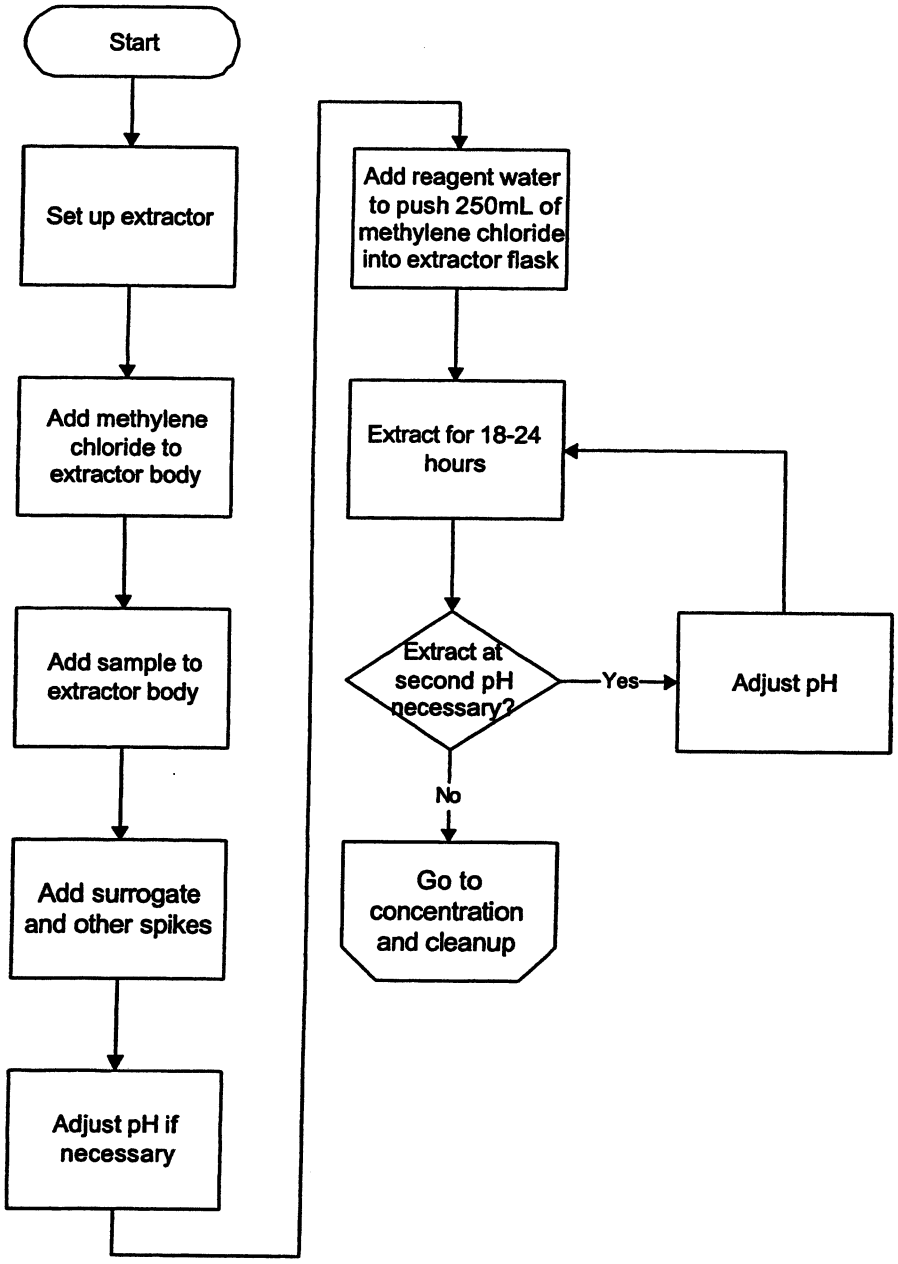
Polynuclear Aromatic Hydrocarbons (8310)	
Compound	**Final Concentration**
Acenaphthylene	10 µg/mL
Carbazole	10 µg/mL
Naphthalene	10 µg/mL
1-Methylnaphthalene	10 µg/mL
2-Methylnaphthalene	10 µg/mL
Acenaphthene	10 µg/mL
Fluorene	2 µg/mL
Phenanthrene	2 µg/mL
Anthracene	2 µg/mL
Fluoranthene	2 µg/mL
Pyrene	2 µg/mL
Benzo(a)anthracene	2 µg/mL
Chrysene	2 µg/mL
Benzo(a)pyrene	2 µg/mL
Benzo(k)fluoranthene	2 µg/mL
Benzo(a)pyrenek)fluoranthene	2 µg/mL
Dibenzo(a,h)anthracene	2 µg/mL
Benzo(g,h,I)perylene	2 µg/mL
Indeno(1,2,3-cd)pyrene	2 µg/mL

17.5. Flow diagrams

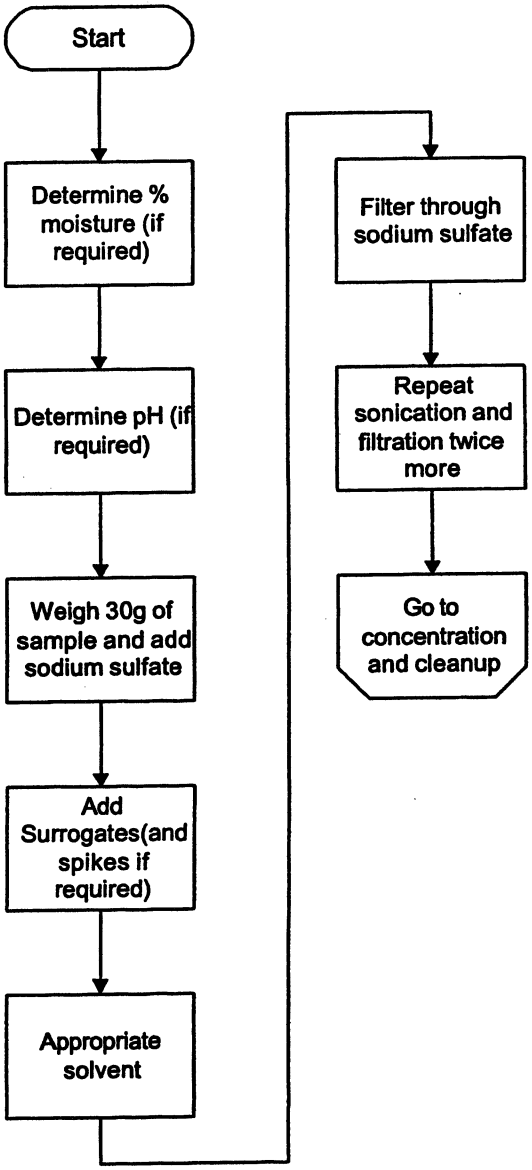
17.5.1. Separatory funnel extraction



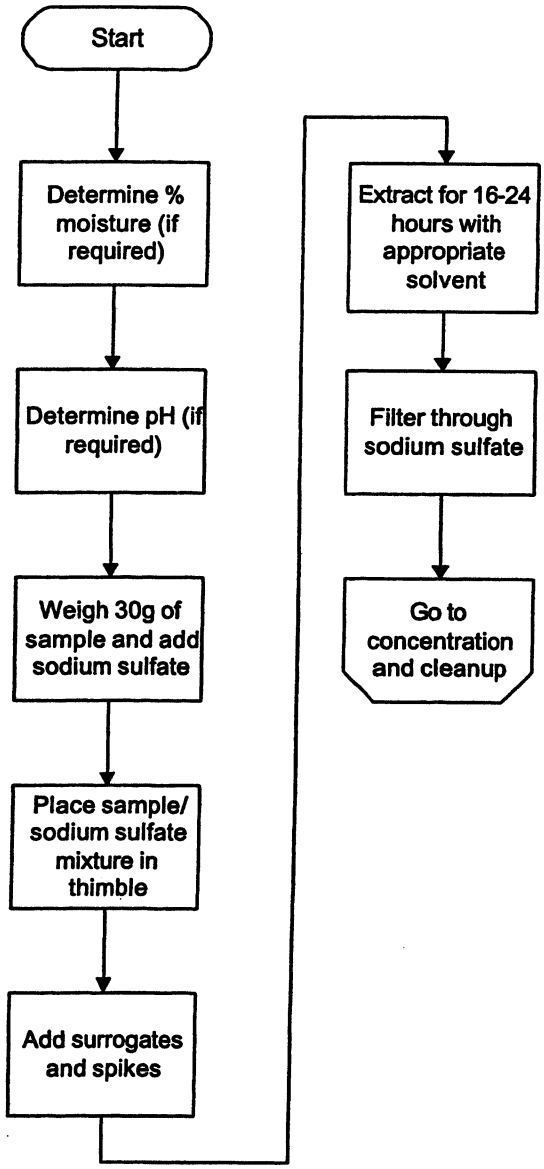
17.5.2. Continuous liquid/liquid extraction


Start
Set up extractor
Add methylene chloride to extractor body
Add sample to extractor body
Add surrogate and other spikes
Adjust pH if necessary
Add reagent water to push 250mL of methylene chloride into extractor flask
Extract for 18-24 hours
Extract at second pH necessary?
Yes
Adjust pH
No
Go to concentration and cleanup

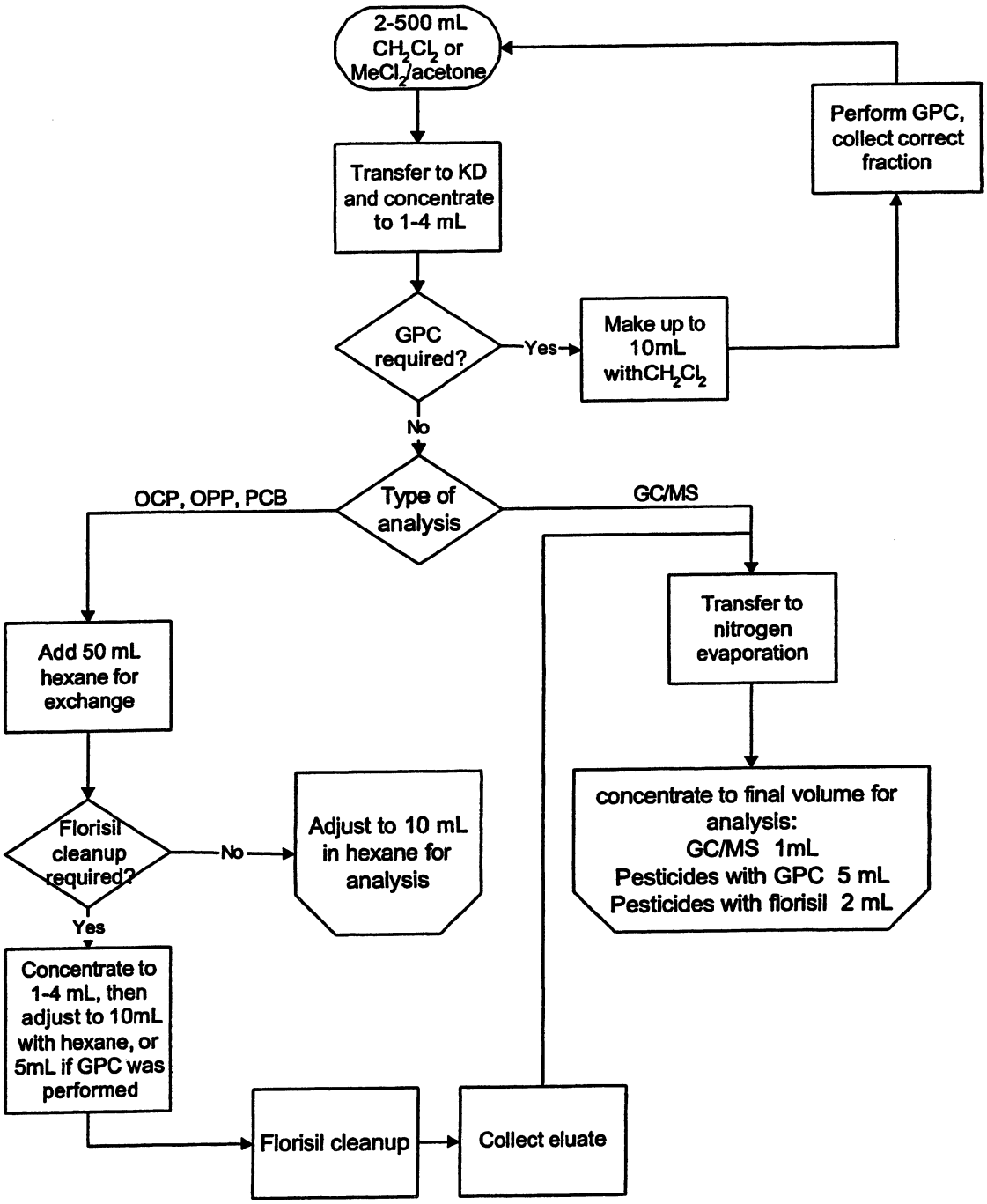
17.5.3. Sonication Extraction



17.5.4. Soxhlet extraction



17.5.5. Concentration and cleanup



18. SCOPE AND APPLICATION

This method is applicable to the extraction of chlorinated herbicides in waters, solids, oils, and TCLP extracts. Appropriate compounds for extraction by this method are listed in CORP-GC-0001, Appendix D, Gas Chromatography of Phenoxy Acid Herbicides based on Method 8151A.

19. SUMMARY OF METHOD

This method is based on SW846 method 8151A. Aqueous samples are hydrolyzed if esters and acids are to be determined, then washed with methylene chloride by a separatory funnel extraction. After acidifying the sample the free acids are extracted into diethyl ether. Solids are extracted into methylene chloride/ acetone by sonication. If esters and acids are to be determined, the extract is hydrolyzed and extracted into diethyl ether. For both soils and aqueous samples, the free acid herbicides in the ether extract are esterified. The final volume is adjusted to prepare the extract for gas chromatography.

20. DEFINITIONS

Refer to section 3 of the main body of this SOP.

21. INTERFERENCES

Refer to section 4 of the main body of this SOP.

22. SAFETY

22.1. Refer to section 5 of the main body this SOP for basic safety information.

22.2. DIAZOMETHANE is an extremely toxic gas with an explosion potential. Since the explosion potential is catalyzed by imperfections in glass, generation of diazomethane must be carried out in glassware free of scratches, cracks, chips and which does not have ground glass joints. Solutions of diazomethane will be kept at temperatures below 90°C. Diazomethane must be generated and handled in a fume hood.

22.3. Diethyl ether is extremely flammable. It also tends to form peroxides when exposed to air. The peroxides can present an explosion hazard, especially when the ether is concentrated.

22.4. Diethyl ether must be free of peroxides as demonstrated by EM (or equivalent) Quant test strips. This test can be done every time the ether is used or once per week if the bottle is marked with the test date(s).

22.5. Concentrated potassium hydroxide solution is highly caustic.

23. EQUIPMENT AND SUPPLIES

23.1. Refer to Section 6 of the main body of this SOP for basic extraction equipment and supplies. Additional equipment and supplies needed for this procedure are listed below.

23.2. Diazomethane generation apparatus

23.3. EM Peroxide test strips

24. REAGENTS AND STANDARDS

24.1. Reagents are listed in Section 7 of the main body of this SOP. Additional Reagents and standards needed for this procedure are listed below.

24.2. Reagents

24.2.1. Potassium hydroxide solution, 37% aqueous solution, (w/v): Dissolve 37 g of potassium hydroxide pellets in reagent water and dilute to 100 mL. **Caution:** Considerable heat will be generated. Other volumes of solution may be made up as convenient.

24.2.2. Sodium hydroxide solution, 10N. Dissolve 400 g NaOH in reagent water and dilute to 1.0L. **Caution:** Considerable heat will be generated. Other volumes of solution may be made up as convenient.

24.2.4. Sulfuric acid, 1:1 Slowly add 500 mL concentrated sulfuric acid to 500 mL water. **Caution:** Considerable heat will be generated. The acid must be added to the water. Wear protective clothing and safety glasses. Other volumes of solution may be made up as convenient.

24.2.5. Diazald, 99% purity

24.2.7. Sodium sulfate, Na_2SO_4, Anhydrous, granular, acidified: Heat sodium sulfate in a shallow tray at 400°C for a minimum of 4 hours to remove phthalates and other interfering organic substances. In a large beaker, acidify by slurrying 500 g sodium sulfate with just enough diethyl ether to cover. Add 20 mL of concentrated sulfuric acid and mix thoroughly. Place the mixture on a steam bath in a hood to evaporate the ether, or allow the ether to evaporate overnight. Larger or smaller batches of acidified sodium sulfate may be prepared using the reagents in the same proportions.

24.2.8. Sodium Chloride, NaCl

24.2.9. Acidified 5% sodium sulfate solution

Add 50 g of sodium sulfate to one liter of reagent water. Add 10 mL of concentrated H_2SO_4. (This reagent may be prepared in different quantities if the proportions are kept the same).

24.2.10. Diethyl ether, reagent grade.

24.2.11. Methanol, reagent grade.

24.2.12. Silicic gel

24.2.13. 2% methanolic KOH,semi-conductor grade

24.3. Standards

24.3.1. Surrogate Standard
See Table A3.

24.3.2. Matrix Spike and LCS standard
See Table A4.

25. SAMPLE COLLECTION PRESERVATION AND STORAGE

25.1. Sample collection and storage is described in Section 8 of the main body of this SOP.

26. QUALITY CONTROL

26.1. Refer to Section 9 of the main body of this SOP for Quality control procedures.

27. CALIBRATION AND STANDARDIZATION

Not applicable

28. PROCEDURE

28.1. Preparation of Aqueous Samples

28.1.1. Weigh the sample bottle and pour approximately 500 ml(100 mL for TCLP leachates) into a 2 liter separatory funnel. Reweigh the bottle and record the sample volume on the benchsheet, assuming a density of 1.0. Alternatively, measure 500 ml in a graduated cylinder. If less than 1 liter was used, reagent water may be added to make the volume up to 1 liter.

Note: Aqueous samples must be determined volumetrically for Ohio VAP samples.

28.1.2. Spike each sample blank, LCS and MS with 1.0 mL of DCAA surrogate solution. Spike matrix spikes and LCS with 1 mL of herbicide matrix spiking solution. (Refer to tables A1 and A2)

28.1.3. Add 60-80 g of NaCl to the sample and shake to dissolve the salt.

28.1.4. Hydrolysis

Use this step only if herbicide esters in addition to herbicide acids are to be determined. This is normally the case. If the herbicide esters are not to be determined, omit this step and go to 11.1.10.
Add 3 mL of 10N NaOH to the sample, seal and shake. Check the pH of the sample with pH paper. If the pH of the sample is not $\geq$12 adjust to $\geq$12 by adding more NaOH. Let the sample sit at room temperature for 2 hours to complete the hydrolysis.

28.1.6. If the whole contents of the sample bottle were not used, add 60 mL of methylene chloride to the separatory funnel.

28.1.7. Extract the sample by shaking or rotating vigorously for 2 minutes, venting as necessary. (An automatic shaker may be used. Place the samples in tumbler and allow them to tumble for one (1) hour. Allow the organic layer to separate from the aqueous layer. If an emulsion layer greater than one third of the solvent layer forms, use mechanical techniques to complete the phase separation. Suggested techniques are stirring, filtration through glass wool and centrifugation.

28.1.8. Discard the **methylene chloride** phase.

28.1.9. Add a second 60 mL of methylene chloride and repeat the extraction a second time, discarding the methylene chloride. Repeat the extraction a third time.

28.1.10. Add 6 mL of cold (4°C) 1:1 sulfuric acid to the sample. Seal, and shake to mix. Check the pH of the sample with pH paper. If the pH is not $\leq$ 2, and more acid to adjust the pH to $\leq$ 2.

Caution: Addition of acid may cause heat and / or pressure build up.

28.1.11. Add 100 mL diethyl ether to the sample and extract by shaking or rotating vigorously for 2 minutes, venting as necessary. (An automatic shaker may be used. Place samples in tumbler and tumble for twenty (20) minutes for each extraction). Allow the organic layer to separate from the aqueous layer. If a emulsion layer greater than one third of the solvent layer forms, use mechanical

techniques to complete the phase separation. Suggested techniques are stirring, filtration through glass wool and centrifugation.

28.1.12. Drain the aqueous layer into a clean flask or beaker. Collect the ether phase in a clean flask or bottle containing approximately 10g of acidified anhydrous sodium sulfate.

28.1.13. Return the aqueous phase to the separatory funnel, add 100 mL diethyl ether and repeat the extraction procedure a second time., combining the ether extracts. Repeat the extraction a third time with 100 mL diethyl ether. Discard the aqueous phase after the third extraction.

28.1.14. Allow the extract to remain in contact with the sodium sulfate for at least 2 hours, shaking periodically. (May be left overnight). The drying step is critical: if the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate. The amount of sodium sulfate is sufficient if some free flowing crystals are visible when the flask or bottle is swirled or shaken.

28.1.15. Proceed to section 11.5, concentration.

28.2. Extraction of soil and sediment samples

28.2.1. Decant and discard any water layer on a sediment/soil sample. Record and document if a water layer was discarded on the benchsheet. Homogenize the sample by mixing it thoroughly in the container. If this is not possible place the sample in clean beaker and homogenize. Upon completion of homogenization in beaker return sample to original container. Discard foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by client. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator). Decant and discard any water layer on a sediment/soil sample.

28.2.2. Weigh 50.0 g of moist solid sample into an clean glass jar. Use 50 g of sodium sulfate for the Method Blank and 50g Ottawa sand for the LCS. Acidify the sample with 5 mL of concentrated HCl.

28.2.3. There should be a small amount of liquid phase. If not, add reagent water until there is. Stir well with a spatula. (Note: This is not necessary for the method blank or LCS)

28.2.4. After 15 minutes, stir with a spatula and check the pH of the liquid phase. Add more acid if necessary to bring the pH to <2, repeating the stirring and standing time after each acid addition. (Note: The pH of the method blank and LCS are not determined.)

28.2.5. Add 60 g of acidified sodium sulfate and mix well. The sample should be free flowing. If not, add more sodium sulfate.

28.2.6. Spike each sample blank, LCS and MS with 1.0 mL of DCAA surrogate solution. Spike matrix spikes and LCS with 1 mL of herbicide matrix spiking solution. (Refer to tables A1 and A2)

28.2.7. Add a minimum of 100 mL of 1:1 methylene chloride:acetone to the beaker or 100 mL of methylene chloride for long list (dinoseb).

28.2.8. Place the bottom surface of the appropriate disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

28.2.9. Sonicate for 3 minutes, making sure the entire sample is agitated. If the W-380 or W-385 sonicator is used the output should be set at 6 for the 3/4 inch high gain (Q) horn or 10 for the 3/4 inch standard horn with mode switch on pulse, and percent-duty cycle knob set at 50%.

28.2.10. Loosely plug the stem of a 75 mm x 75 mm glass funnel with glass wool and/or line the funnel with filter paper. Add 10-20 g of anhydrous sodium sulfate to the funnel cup.

28.2.11. Place the prepared funnel on a collection apparatus. If the herbicide esters are *not* to be determined, the collection apparatus is a bottle or flask containing approximately 10g of anhydrous acidified sodium sulfate. If the herbicide esters *are* to be determined, (normally the case) the collection apparatus is glassware suitable for the hydrolysis step, typically a KD flask or Turbovap tube.

28.2.12. Decant and filter extracts through the prepared funnel into the collection apparatus.

28.2.13. Repeat the extraction two more times with additional 100 mL minimum portions of the appropriate solvent each time. Decant off extraction solvent after each sonication. On the final sonication pour the entire sample (sediment and solvent) into the funnel and rinse with an additional 10 mL-20 mL of the methylene chloride/acetone.

Note: Alternatively, the three extracts may be collected together and then filtered through the sodium sulfate.

28.2.14. If the herbicide esters are not to be determined, dry the extract as described in 11.4.2 or go to cleanup, section 11.3. If the herbicide esters are to be determined (normally the case) proceed to section 11.2.15

28.2.15. Add 5 mL of 37% aqueous potassium hydroxide and 30 mL of water to the extract. Check the pH with pH paper. If the pH is not $\geq$12, adjust with additional KOH.

28.2.16. Heat on a water bath at 60-65°C for 2 hours. Allow to cool. Higher temperatures, up to 90°C, may be used if needed to remove the ether layer within 2 hours.

28.2.17. Transfer the solution to a separatory funnel and extract three times with 100 mL portions of methylene chloride. **Discard the methylene chloride phase.** The aqueous solution contains the herbicides.

28.2.18. Adjust the pH of the solution to $\leq$2 with 1:1 sulfuric acid.

28.2.19. Extract three times with 40 mL diethyl ether.

28.2.20. Proceed to section 11.3, Cleanup, if required, or Section 11.4, Extract drying.

28.3. Cleanup

This cleanup step may be necessary if the procedure for determining the herbicide acids only is being followed. (See section 11.2.14) It is not normally required if the acids and esters are being determined. (The usual case.) If cleanup is not required, proceed to section 11.4, Extract drying.

28.3.1. Prepare 45 mL of basic extraction fluid by mixing 30 mL of reagent water with 15 mL of 37% KOH. Use three 15 mL portions of this fluid to partition the extract from section 11.2.12 or 11.2.20, using a small separatory funnel. **Discard the organic phase.**

28.3.2. Adjust the pH of the solution to $\leq$2 with cold (4°C) sulfuric acid. (1:1). Extract once with 40 mL diethyl ether and twice with 20 mL diethyl ether.

Caution: Addition of acid may cause heat and / or pressure build up.

28.4. Extract drying

28.4.1. Pour the extracts through a funnel containing acidified sodium sulfate into a flask or bottle containing approximately 10 g acidified sodium sulfate. Rinse the funnel with a little extra diethyl ether.

28.4.2. Allow the extract to remain in contact with the sodium sulfate for at least 2 hours, shaking periodically. (May be left overnight). The drying step is critical: if the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate. The amount of sodium sulfate is sufficient if some free flowing

crystals are visible when the flask or bottle is swirled or shaken. Proceed to section 11.5, concentration.

28.5. Concentration

28.5.1. Transfer the ether extract into a Turbovap concentrator tube or a 500 mL K-D flask equipped with a 10 mL concentrator tube. Use a stirring rod to crush the caked sodium sulfate during transfer. Rinse the flask or bottle with 20-30 mL ether to complete transfer.

28.5.2. Attach a three ball Snyder column to the K-D apparatus, prewet the column with a few mL of ether from the top, and place the apparatus on a water bath at approximately 60°C. At the proper rate of distillation, the balls of the column will chatter, but the chambers will not flood. When the apparent volume reaches 2 mL, remove from the water bath and allow to completely cool.

28.5.3. Carefully disassemble the concentrator tube and rinse the lower glass joint with a small amount of diethyl ether.

28.5.5. The extract is now ready for esterification by either the diazomethane solution method (11.6) or the bubbler method (11.7)

28.7. Esterification (Bubbler Method)

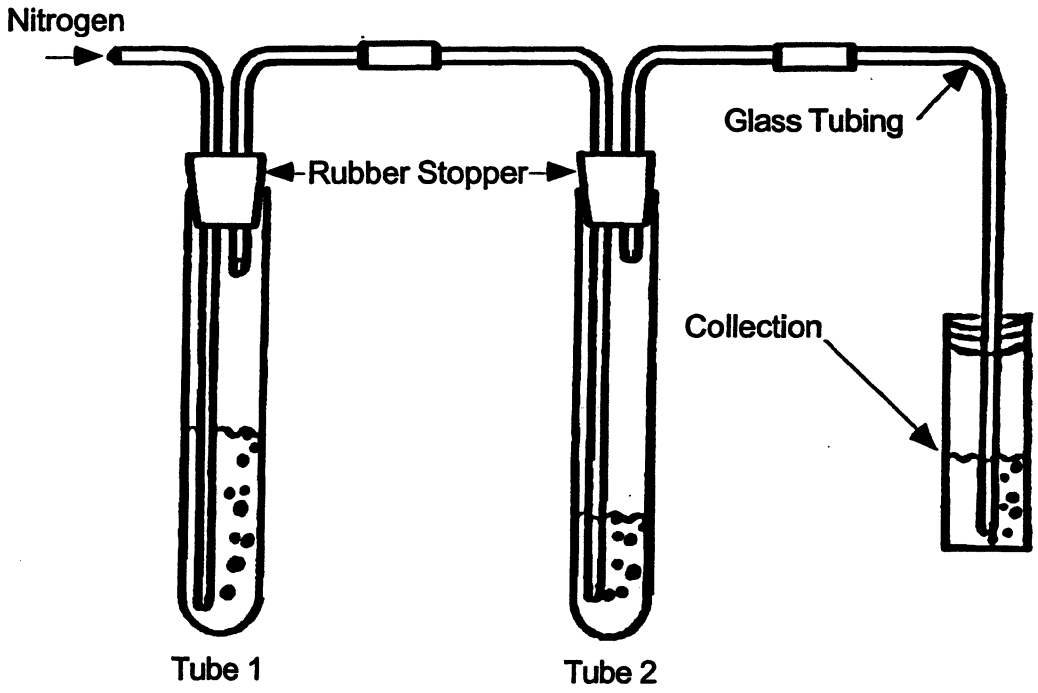
28.7.1. Assemble the diazomethane apparatus (see figure below) in a hood. Add 10 mL of diethyl ether to tube 1. Add 5 mL of 2% methanolic KOH, 3 mL of ether and 0.5-1 g of diazald to tube 2.

28.7.2. Place the tip of the disposable pipette into the vial containing the first sample extract. Apply nitrogen flow (approx. 10 mL/min) to bubble diazomethane through the sample extract for about 1 minute, or until the yellow color persists. Replace the disposable pipette and place the tip into the vial containing the second extract. Continue until the diazald is consumed. (An additional 0.1-0.5 g diazald may be added to extend the generation of diazomethane).

28.7.3. Allow the extracts to stand for 20 minutes, then add approximately 0.2 g of silicic gel to each extract. Allow to stand for an additional 20 minutes.

28.7.4. Adjust the volume to 10 mL with hexane. The sample is now ready for gas chromatography.

28.7.5 A routine 10X dilution occurs on final extracts for all samples. Due to a QuantIMS limitation, the dilution factor field in QuantIMS cannot be used when a dilution is routine, because the dilution factor is automatically applied to all reference values creating reporting problems. For the herbicide analysis, the extract volume will be 10mL and an aliquot at 10X dilution will be analyzed. The final extract volume recorded on the laboratory bench sheet will be recorded as 100mL to avoid using the dilution factor field in QuantIMS.



29. DATA ANALYSIS AND CALCULATIONS

Not applicable

30. METHOD PERFORMANCE

Refer to CORP-GC-0001 for details of method performance.

31. POLLUTION PREVENTION

Refer to section 14 of the main body of this SOP.

32. WASTE MANAGEMENT

Refer to section 15 of the main body of this SOP.

33. REFERENCES

33.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Chlorinated Herbicides, Method 8151A.

34. MISCELLANEOUS

34.1. Modifications from Reference Method

34.1.1. Directions to add sufficient reagent water to the soil sample so that the pH can be measured have been added (Section 11.1.2)

34.1.2. The bubbler esterification method uses methanolic KOH in place of the aqueous KOH / carbitol mixture recommended in method 8150B. This has been found to provide a more effective and reliable esterification.

34.2. Modifications from previous revisions

References have been updated

34.3. Tables

Table A1

Herbicide Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Volume (mL)
Herbicides	Herbicides Water	1.0
Herbicides	Herbicides Soil	1.0

Table A2

Herbicide Matrix Spike and LCS Solutions

Analyte Group	Matrix Spike Solution ID	Volume (mL)
Herbicides	Herbicides MS-Soil	1.0
Herbicides	Herbicides MS-Water	1.0

Table A3
Herbicide Surrogate Spike Components

Type	Compounds[1]	Solvent	Conc. (ug/mL)
Herbicides WS	2,4-DCAA	Acetone	2
Herbicides SS	2,4-DCAA	Acetone Methanol	20

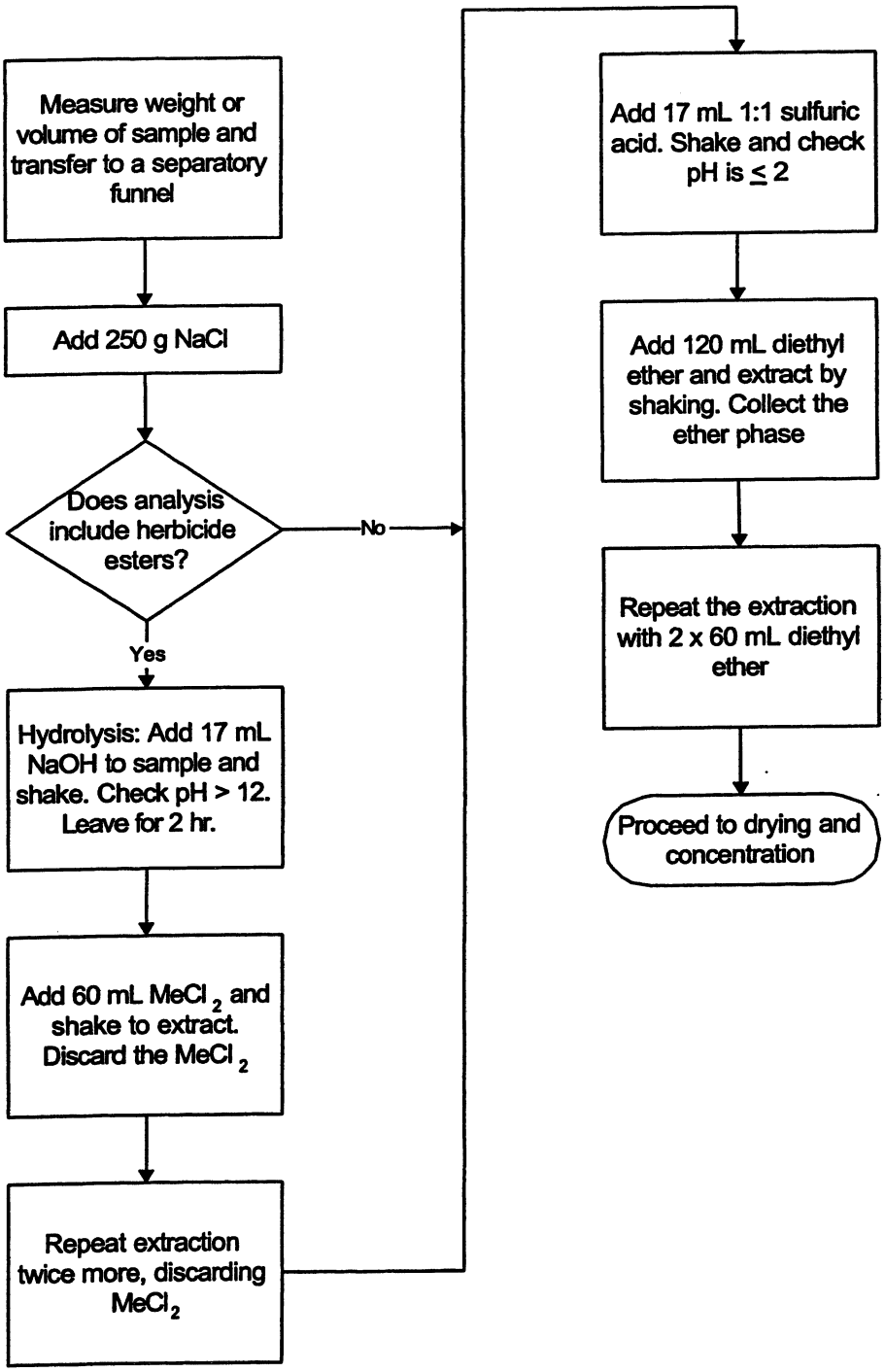
[1]The surrogate is spiked as the free acid

Table A4
Herbicide Matrix Spike Components

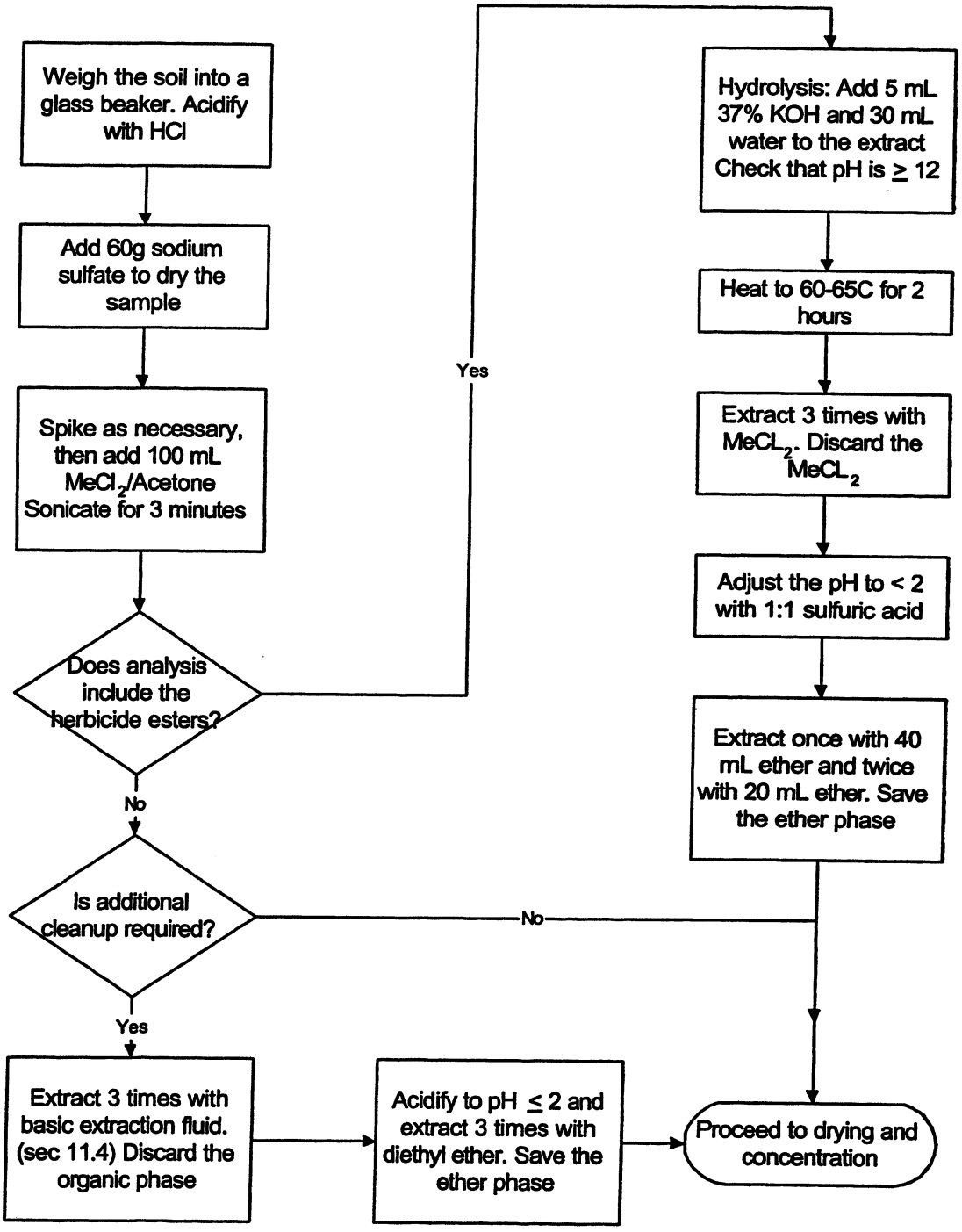
Type	Compounds[1]	Compounds[1] Solvent	Soil Conc. (ug/mL) Solvent	Water Conc. (ug/mL)
Herbicides MS	2,4-D	Acetone	20 Methanol	2
	2,4-DB		20	2
	2,4,5-TP (Silvex)		5	0.5
	Dalapon		10	1
	Dicamba		10	1
	Dichloroprop		10	1
	Dinoseb		3	.3
	2,4,5-T		5	0.5

[1]The herbicide spiking solution contains the herbicides as the free acids.

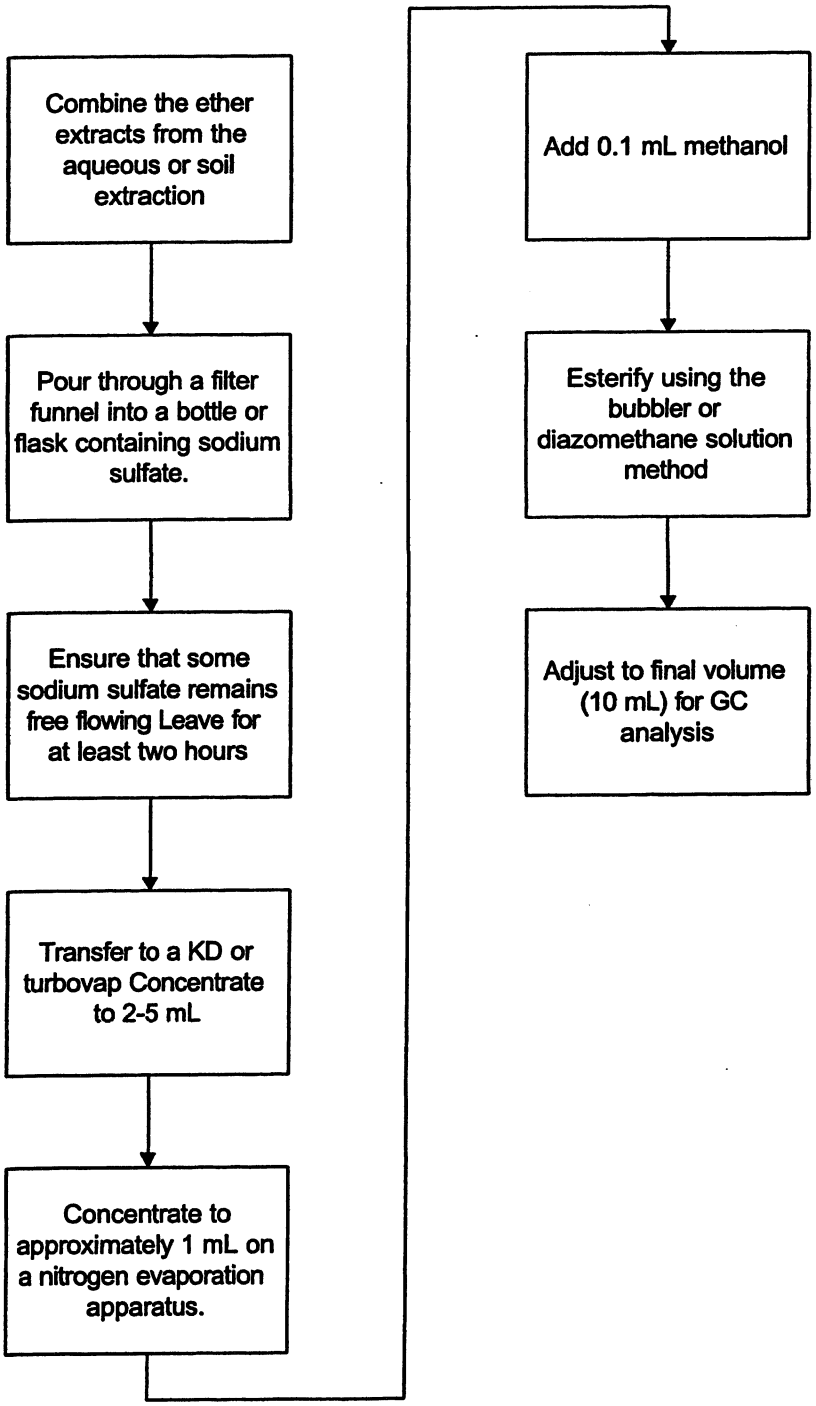
Extraction of Aqueous Samples



Extraction of Soils and Sediments



Drying, Concentration and Esterification



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SOP No: CORP-MS-0001NC
Revision No: 2.4
Revision Date: 05/29/01
Page 1 of 59

STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: GC/MS ANALYSIS BASED ON METHODS 8270C AND 625

(SUPERSEDES: Revision 2.3, 02/15/01)

Prepared by: _____ 5/30/01
Date

Reviewed by: Mark Ulman 5-30-01
Technology Specialist Date

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Environmental Health and Safety — Date

Approved by: _____
Laboratory Director — Date

Approved by: _____ 6/4/01
Coroporate Technology — Date

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TABLE A-1 Method 625 Reporting List and Limits

TABLE-A-2 Method 625 LCS and MS Compounds and Spike Concentrations

1. SCOPE AND APPLICATION

1.1 This method is based upon SW846 8270C, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices. The modifications presented in Attachment A may be followed for analysis of wastewater following method 625. Direct injection of a sample may be used in limited applications. Refer to Tables 1, 2, 3 and 4 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. Additional compounds may be amenable to this method. If non-standard analytes are required, they must be validated by the procedures described in section 13 before sample analysis.

1.2 The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- Hexachlorophene is not amenable to analysis by this method.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method.

1.3 The standard reporting limit (SRL) of this method for determining an individual compound is approximately 0.33 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to Tables 1 and 2 for specific SRLs. Reporting limits will be proportionately higher for sample extracts that require dilution.

1.4 The associated LIMS codes are QL (8270C) and DP (625).

2 SUMMARY OF METHOD

2.1 Aqueous samples are extracted with methylene chloride using a separatory funnel, a continuous extractor or Accelerated One-Step™. Solid samples are extracted with methylene chloride / acetone using sonication, soxhlet, accelerated soxhlet or pressurized fluid extraction. Waste dilution is used for samples that are miscible with the solvent. The extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS. Extraction procedures are detailed in SOP# CORP-OP-0001. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

3 DEFINITIONS

3.1 CCC (Calibration Check Compounds) - A subset of target compounds used to evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCC's is specified for calibration acceptance.

3.2 SPCC (System Performance Check Compounds) - Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance.

3.3 Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the STL North Canton QC Program document (QA-003) for further details of the batch definition.

3.4 Method Blank - An analytical control consisting of all reagents, internal standards and surrogate standards, that is carried through the entire analytical procedure. The method

blank is used to define the level of laboratory background and reagent contamination.

3.5 LCS (Laboratory Control Sample) - A blank spiked with the parameters of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked materials demonstrates that the laboratory techniques for this method are acceptable.

3.6 MS (Matrix Spike)- aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

3.7 MSD (Matrix Spike Duplicate)- a second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method.

4 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.2 The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.3 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.5 Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5 SAFETY PRECAUTIONS

5.1 Procedures shall be carried out in a manner that protects the health and safety of all STL

North Canton associates. The following requirements must be met:

5.1.1 Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.1.2 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

5.1.3 Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include: Benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, and n-nitrosodimethylamine. Primary standards should be purchased in solution. If neat materials must be obtained, they shall be handled in a hood.

5.1.4 Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers should be kept closed unless transfers are being made.

5.1.5 All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported immediately to a laboratory supervisor.

6 EQUIPMENT AND SUPPLIES

6.1 Gas Chromatograph/Mass Spectrometer System: An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

6.2 Column: 30 m x 0.32 mm I.D. (or 0.25 mm I.D.) 0.5-µm film thickness silicon-coated fused-silica capillary column (J & W Scientific DB-5.625 or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.3 Mass Spectrometer: Capable of scanning from 35 to 500 AMU every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 6 when 50 ng of the GC/MS

tuning standard is injected through the GC.

6.4 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.5 Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.

6.6 Syringe: 10 µL Hamilton Laboratory grade syringes or equivalent.

6.7 Carrier gas: Ultra high purity helium.

7 REAGENTS AND STANDARDS

7.1 A minimum five point calibration curve is prepared. The low point should be at or below the reporting limit. Refer to Tables 12 and 13 for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration.

7.2 An Internal Standard solution is prepared. Compounds in the I.S. Mix are: acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.

7.2.1 Internal Standards are added to all standards and extracts to result in 8 ng or 40ng injected onto the column. For example, if the volume of an extract used was 200 µL, 20 µL of a 400 µg/mL internal standard solution would be added for a 1 µL injection.

7.3 Surrogate Standard Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. Surrogate compounds and levels are listed in Table 11.

7.4 GC/MS Tuning Standard: A methylene chloride solution containing 10 µg/mL or 50 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT, should also be included in the Tuning Standard at 10 µg/mL or 50 µg/mL.

7.5 Laboratory Control Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. LCS compounds and levels are listed in Tables 9 and 10.

7.6 Matrix Spike Solution: Prepare as indicated in the preparative methods. See preparation SOP. The matrix spike compounds and levels are the same as the LCS compounds.

7.7 The standards listed in 7.1 to 7.6 should be refrigerated at $\leq$ 6°C when not in use. Refrigeration at -10°C to -20°C may be used if it can be demonstrated that analytes do not fall out of solution at this temperature. The standards must be replaced at least once a year. The continuing calibration standard must be replaced every week and is stored at $\leq$ 6°C.

8 SAMPLE PRESERVATION AND STORAGE

8.1 Reference appropriate facility SOP for sample bottle preservation and storage.

8.2 Samples are stored at 4 $\pm$ 2°C. Samples and extracts should be stored in suitable glass containers with Teflon lined caps. (Extracts will normally be stored for 30 days after invoicing.)

8.3 Water samples are extracted within seven days of sampling and the extracts are analyzed within forty days of extraction. Solids, sludges, and organic liquids are extracted within fourteen days of sampling and the extracts are analyzed within forty days of extraction.

9 QUALITY CONTROL

9.1 Initial Demonstration of Capability

9.1.1 For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in section 13 must be acceptable before analysis of samples may begin. Refer to the flow chart in section 17.4.1.

9.1.2 For non-standard analytes an MDL study should be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2 Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery +/- 3 standard deviations for surrogates, MS and LCS Precision limits for matrix spikes / matrix spike duplicates are mean relative percent difference +/- 3 standard deviations.

9.2.1 These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported unless the dilution is more

than 5X.

9.2.2 All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.3 Refer to the QC program document (QA-003) for further details of control limits.

9.3 Method Blank

A method blank is prepared and analyzed with each batch of samples. The method blank consists of reagent water for aqueous samples, and sodium sulfate for soil samples (Refer to SOP No. CORP-OP-0001 for details). Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

- If the analyte is a common laboratory contaminant (phthalate esters), the data may be reported with qualifiers if the concentration of the analyte is less than five times the RL. Such action must be taken in consultation with the client.

- Reanalysis of any samples with reportable concentrations of analytes found in the method blank is required unless other actions are agreed with the client.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.3.1 The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.3.2 If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.3.3 Refer to the STL North Canton QC Program document (QA-003) for further details of the corrective actions.

9.3.4 Sample results are NOT blank subtracted unless specific requests and arrangements have been made with a client or agency.

9.4 Instrument Blank

9.4.1 Instruments must be evaluated for contamination during each 12 hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of methylene chloride with the internal standards added. It is evaluated in the same way as the method blank.

9.5 Laboratory Control Sample (LCS)

9.5.1 A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. All analytes must be within established control limits. The LCS is spiked with the compounds listed in Tables 9 and 10 unless specified by a client or agency.

9.5.2 If any analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS).

- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5.3 Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples. The MS/MSD is spiked with the same subset of analytes as the LCS

(See Tables 9 and 10). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.

- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.

- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7 Surrogates

9.7.1 Every sample, blank, and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 11.

9.7.2 If any surrogates are outside limits the following corrective actions must take place (except for dilutions):

- Check all calculations for error.

- Ensure that instrument performance is acceptable.

- Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.

- Re-extract and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client.

It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

Note: If all associated QC meets criteria (blank, LCS/LCSD), up to one surrogate per fraction may be outside of acceptance criteria , as long as the recovery is greater than 10%.

9.7.3 If the sample with surrogate recoveries outside the recovery limits was a sample used for an MS/MSD and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample, the MS, and the MSD do not require reanalysis as this phenomenon would indicate a possible matrix problem.

9.7.4 If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only the reanalyzed data should be reported. (Unless the reanalysis was outside holding times, in which case reporting both sets of results may be appropriate.)

9.7.5 If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effect.

9.8 Nonconformance and Corrective Action

9.8.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.9 Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.10 STL North Canton QC Program

Further details of QC and corrective action guidelines are presented in the STL North Canton QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10 CALIBRATION AND STANDARDIZATION

10.1 Summary

10.1.1 The instrument is tuned for DFTPP, calibrated initially with a five-point calibration

curve, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 5.

10.2 All standards and extracts are allowed to warm to room temperature before injecting.

10.3 Instrument Tuning

At the beginning of every twelve hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table 6) is achieved for DFTPP (decafluorotriphenylphosphine).

10.3.1 Inject 10 ng or 50 ng of the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 6 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.3.2 The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. Benzidine and pentachlorophenol should not exhibit excessive tailing. If DDT is an analyte of interest, it must be included in the tuning standard, and its breakdown must be < 20%. Refer to section 12 for the appropriate calculations.

10.4 Initial Calibration

10.4.1 Internal Standard Calibration Procedure: Internal standards are listed in Table 7. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

10.4.2 Compounds should be assigned to the IS with the closest retention time.

10.4.3 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest. Six standards must be used for a quadratic least squares calibration. Quadratic fit may NOT be used for samples analyzed under South Carolina Certification. It may also be useful to analyze six calibration levels and use the lower five for most analytes and the upper five for analytes that have poor response. Add the internal standard mixture to result in 8 ng or 40 ng on column. The concentrations of all analytes are listed in tables 12 and 13.

10.4.4 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Calculate response factors (RF), average response factors, and the percent RSD of the response factors

for each compound using the equations in section 12 and verify that the CCC and SPCC criteria in section 10.4.5 and 10.4.6 are met. **No sample analysis may be performed unless these criteria are met.**

10.4.5 System Performance Check Compounds (SPCCs): The minimum average RF for semivolatile SPCCs is 0.050. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

SPCC Compounds:

N-nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

10.4.6 Calibration Check Compounds (CCCs): The %RSD of the response factors for each CCC in the initial calibration must be less than 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this criterion.

10.4.6.1 If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.4.6.2 CCC Compounds:

Phenol
Acenaphthene
1,4-Dichlorobenzene
N-nitrosodiphenylamine
2-Nitrophenol
Pentachlorophenol
2,4-Dichlorophenol
Fluoranthene
Hexachlorobutadiene
Di-n-octylphthalate
4-Chloro-3-methylphenol
Benzo(a)pyrene
2,4,6-Trichlorophenol

10.4.7 If the average of all %RSDs in the initial calibration is ≤ 15%, then all analytes may use average response factor for calibration.

10.4.7.1 If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve then the appropriate curve should be used for quantitation.

10.4.7.2 If the average of all the %RSDs in the initial calibration is > 15%, then calibration on a curve must be used for those analytes with %RSD > 15%. Linear or quadratic curve fits may be used. Linear curve fits only may be used for South Carolina Certification. The analyst should consider instrument maintenance to improve the linearity of response. Use of 1/Concentration^2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. If Relative Standard Error (RSE) is used to evaluate the curve it must be better than 15%. If the % RSD is >15%, the analyst may drop the low or high points in the ICAL, as long as a minimum of 5 points are maintained and the quantitation range is adjusted accordingly. If the % RSD is still >15%, a quadratic or linear curve may be used. The correlation coefficient (r) must be ≥ 0.990. If the correlation coefficient is < 0.990, then any hits for these compounds must be flagged as estimated.

10.4.8 Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. For South Carolina Certification, only a linear calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called 1/X^2 weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4.9 If time remains in the 12 hour period initiated by the DFTPP injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.10 Quantitation is performed using the calibration curve or average response factor form the initial curve, not the continuing calibration.

10.5 Continuing Calibration

10.5.1 At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. A 10 ng or 50 ng injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 6.

10.5.2 Following a successful DFTPP analysis the continuing calibration standard(s) are analyzed. The standards must contain all semivolatile analytes, including all required surrogates. A mid level calibration standard is used for the continuing calibration.

10.5.3 The following criteria must be met for the continuing calibration to be acceptable:

- The SPCC compounds must have a response factor of ≥ 0.05.
- The percent difference or drift of the CCC compounds from the initial calibration must be $\leq 20\%$. (see section 12 for calculations) In addition, the percent difference or drift of all analytes must be $\leq 50\%$, with allowance being made for up to six target compounds to have percent drift greater than 50%.
- The internal standard response must be within 50-200% of the response in the mid level of the initial calibration.
- The internal standard retention times must be within 30 seconds of the retention times in the mid-level of the initial calibration.
- NOTE: There is no internal standard criteria for samples. Criteria is only for continuing and initial calibrations.
- NOTE: Ohio VAP rules require that any sample with internal standard outliers be reanalyzed.

10.5.3.1 If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.5.4 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the DFTPP have passed. (A sample *injected* less than 12 hours after the DFTPP is acceptable.)

NOTE: Additional compound analysis outside the TCL (Target Compound List) have no calibration criteria.

11 PROCEDURE

11.1 Sample Preparation

Samples are prepared following SOP CORP-OP-0001.

11.2 Sample Analysis Procedure

11.2.1 Calibrate the instrument as described in section 10. Depending on the target compounds required by the client, it may be necessary to use more than one calibration standard.

11.2.2 All samples must be analyzed using the same instrument conditions as the preceeding continuing calibration standard.

11.2.3 Add internal standard to the extract to result in 40 ng injected on column (for example, 8 µL or 50 µL internal standard solution in 0.5 mL of extract for a 1 µL injection). Mix thoroughly before injection into the instrument.

11.2.4 Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.

11.2.5 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in section 12. Quantitation is based on the initial calibration, not the continuing calibration.

11.2.6 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system.

11.2.7 Target compounds identified by the data system are evaluated using the criteria listed in section 12.1.

11.2.8 Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client. They are evaluated using the criteria in section 12.3.

11.3 Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.3.1 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgement. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

11.3.2 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

11.4 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at 4 ± 2°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

11.5 Retention time criteria for samples

If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.5.1 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

11.6 Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to the facility specific SOP for determination of percent moisture.

11.7 Procedural Variations

11.7.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. Any unauthorized deviations from this procedure must also be documented as a non-conformance, with a cause and corrective action described.

11.8 Troubleshooting Guide

11.8.1 Daily Instrument Maintenance

In addition to the checks listed in the instrument maintenance schedule in the STL North Canton Quality Assurance Manual (LQM), current version, the following daily maintenance should be performed.

- Clip Column as necessary.
- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Perform mass calibration as necessary.

11.8.2 Major Maintenance

A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning the ion volume or repeller, cleaning the source, and replacing the multiplier. Refer to the manufacturer's manual for specific guidance.

12 DATA ANALYSIS AND CALCULATIONS

12.1 Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NBS library. Two criteria must be satisfied

to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- The characteristic ions of a compound must maximize in the same scan or within one scan of each other.
- The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

12.1.1 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation.

12.2 Mass chromatogram searches.

Certain compounds are unstable in the calibration standard and cannot be calibrated in the normal way. In particular, the compound hexachlorophene (CAS 70-30-4) falls into this category, and is required for Appendix IX analysis. For this analyte a mass chromatogram search is made.

12.2.1 Hexachlorophene

Display the mass chromatograms for mass 196 and mass 198 for the region of the chromatogram from at least 2 minutes before chrysene-d12 to at least 4 minutes after chrysene-d12. If peaks for both ions coincide then the analyst evaluates the spectrum for the presence of hexachlorophene. No quantitation is possible.

12.3 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines should not use normalization routines that would

misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches shall the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

- Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30%and 70%.)
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or presence of coeluting compounds.
- Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.
- Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

12.4 Anyone evaluating data is trained to know how to handle isomers with identical mass spectra and close elution times. These include:

Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

Extra precautions concerning these compounds are to more closely scrutinize retention time vs. the calibration standard and also to check that all isomers have distinct retention times.

A second category of problem compounds would be the poor responders or compounds that chromatograph poorly. Included in this category would be:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol

Manually checking the integrations would be appropriate for these compounds.

12.5 Calculations

12.5.1 Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{RF} \times 100$$

RF = Mean of RFs from intial caibration for a compound

SD = Standard deviation of RFs from initial calibration for a compound,

$$= \sqrt{\sum_{i=1}^{N} \frac{\left(RFi - \overline{RF}\right)^2}{N-1}}$$

RFi = RF for each of the calibration levels

N = Number of RF values

12.5.2 Continuing calibration percent drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

C_{actual} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.5.3 Concentration in the extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration. For South Carolina Certification the concentration

of each identified analyte and surrogate in the extract is calculated from the linear curve only fitted to the initial calibration points, or from the average RF of the initial calibration.

12.5.3.1 Average response factor

If the average of all the %RSDs of the response factors in the initial calibration is ≤ 15%, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

12.5.3.2 Linear fit (Use only Linear fit for South Carolina Certification)

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

C_{ex}= Concentration in extract, µg/mL

R_x= Response for analyte

R_{is}= Response for internal standard

C_{is}= Concentration of internal standard

A= Intercept

B= Slope

12.5.3.3 Quadratic fit

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)$$

C= Curvature

12.5.4 The concentration in the sample is then calculated.

12.5.4.1 Aqueous Calculation

$$\text{Concentration, } \mu g / L = \frac{C_{ex} V_t}{V_o}$$

Where:

V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 mL extract will mean V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, V_t = 2,000.)

V_o = Volume of water extracted (mL)

12.5.5 Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis:

$$\text{Concentration, } \mu g / kg = \frac{C_{ex} V_t}{W_s D}$$

W_s = Weight of sample extracted or diluted in grams

D = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis

12.6 MS/MSD percent recovery calculation.

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

S_{SR} = Spike sample result

S_R = Sample result

S_A = Spike added

12.7 Relative % Difference calculation for the MS/MSD

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

RPD = Relative percent difference

MS_R = Matrix spike result

MSD_R = Matrix spike duplicate result

12.8 Relative response factor calculation.

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

A_x=Area of the characteristic ion for the compound being measured

A_{is}=Area of the characteristic ion for the specific internal standard

C_x=Concentration of the compound being measured (µg/L)

C_{is} =Concentration of the specific internal standard (µg/L)

12.9 Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x = Area of the total ion chromatogram for the compound being measured

A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

12.10 Percent DDT breakdown

$$\% \text{ DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDarea}}$$

The total ion current areas are used for this calculation

13 METHOD PERFORMANCE

13.1 Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: QA-005.

13.2 Initial Demonstration

Each laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to the level 4 calibration standard.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in table 14.

13.2.3 If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3 Non-standard analytes

For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

13.4 Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.5 Data Quality Objectives (DQO). Refer to project-specific Quality Assurance plans for DQO information.

14 POLLUTION PREVENTION

14.1 This section is not applicable to this procedure.

15 WASTE MANAGEMENT

15.1 Waste generated during aliquotting and from used vials must be disposed of in accordance with the facility hazardous waste procedures. The Health and Safety Director should be contacted if additional information is required.

16 REFERENCES

16.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update II, October 1994, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270C.

16.2 J. W. Eichelberger, L. E. Harris, and W. L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry," Analytical Chemistry, 47, 995 (1975)

17 MISCELLANEOUS

17.1 Modifications from Reference Method

17.1.1 A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.2 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.2 Modifications from Previous Revision

17.2.1 This SOP has been substantially revised to meet the requirements of method 8270C.

17.2.2 Directions for analysis be method 625 have been added as an attachment.

17.2.3 SOP has been revised to include a 5 mL final volume.

17.3 Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4 Tables

Table 1

STL North Canton Primary Standard[1] and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Pyridine	110-86-1	20	660
N-nitrosodimethylamine	62-75-9	10	330
Aniline	62-53-3	10	330
Phenol	108-95-2	10	330
Bis(2-chloroethyl)ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	10	330
1,4-Dichlorobenzene	106-46-7	10	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	10	330
2-Methylphenol	95-48-7	10	330
2,2'-oxybis(1-chloropropane)[2]	108-60-1	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic acid	65-85-0	50	1600
Bis(2-chloroethoxy)methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene	77-47-4	50	1600
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2-Chloronaphthalene	91-58-7	10	330
2-Nitroaniline	88-74-4	50	1600
Dimethyl phthalate	131-11-3	10	330
Acenaphthylene	208-96-8	10	330
3-Nitroaniline	99-09-2	50	1600
Acenaphthene	83-32-9	10	330
2,4-Dinitrophenol	51-28-5	50	1600
4-Nitrophenol	100-02-7	50	1600
Dibenzofuran	132-64-9	10	330

Table 1

STL North Canton Primary Standard[1] and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
2,4-Dinitrotoluene	121-14-2	10	330
2,6-Dinitrotoluene	606-20-2	10	330
Diethylphthalate	84-66-2	10	330
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	10	330
4-Nitroaniline	100-01-6	50	1600
4,6-Dinitro-2-methylphenol	534-52-1	50	1600
N-Nitrosodiphenylamine	86-30-6	10	330
Azobenzene	103-33-3	10	330
4-Bromophenyl phenyl ether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Pentachlorophenol	87-86-5	50	1600
Phenanthrene	85-01-8	10	330
Anthracene	120-12-7	10	330
Carbazole	86-74-8	10	330
Di-n-butyl phthalate	84-74-2	10	330
Fluoranthene	206-44-0	10	330
Benzidine	92-87-5	100	3300
Pyrene	129-00-0	10	330
Butyl benzyl phthalate	85-68-7	10	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
Benzo(a)anthracene	56-55-3	10	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
Chrysene	218-01-9	10	330
Di-n-octylphthalate	117-84-0	10	330
Benzo(b)fluoranthene	205-99-2	10	330
Benzo(k)fluoranthene	207-08-9	10	330
Benzo(a)pyrene	50-32-8	10	330
Indeno(1,2,3-cd)pyrene	193-39-5	10	330
Dibenz(a,h)anthracene	53-70-3	10	330
Benzo(g,h,i)perylene	191-24-2	10	330
Benzaldehyde	100-52-7	10	330
Caprolactam	105-60-2	10	330
1,1-Biphenyl	92-52-4	10	330
Atrazine	1912-24-9	10	330

[1] The STL North Canton primary standard is the standard normally used at F. Additional standards, such as the Appendix IX standard may be necessary to include all target analytes required for some clients.

[2] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

Table 2

STL North Canton Appendix IX[1] Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
2-Picoline	109-06-8	20	660
N-Nitrosomethylethylamine	10595-95-6	10	330
Methyl methanesulfonate	66-27-3	10	330
N-Nitrosodiethylamine	55-18-5	10	330
Ethyl methanesulfonate	62-50-0	10	330
Pentachloroethane	76-01-7	50	1600
Acetophenone	98-86-2	10	330
N-Nitrosopyrrolidine	930-55-2	10	330
N-Nitrosomorpholine	59-89-2	10	330
o-Toluidine	95-53-4	20	660
3-Methylphenol	108-39-4	10	330
N-Nitrosopiperidine	100-75-4	10	330
o,o,o-Triethyl-Phosphorothioate[2]	126-68-1	50	1600
a,a-Dimethyl-phenethylamine	122-09-8	50	1600
2,6-Dichlorophenol	87-65-0	10	330
Hexachloropropene	1888-71-7	100	3300
p-Phenylenediamine	106-50-3	100	3300
n-Nitrosodi-n-butylamine	924-16-3	10	330
Safrole	94-59-7	20	660
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330
Isosafrole	120-58-1	20	660
1,4-Dinitrobenzene	100-25-4	10	330
1,4-Naphthoquinone	130-15-4	50	1600
1,3-Dinitrobenzene	99-65-0	10	330
Pentachlorobenzene	608-93-5	10	330
1-Naphthylamine	134-32-7	10	330
2-Naphthylamine	91-59-8	10	330
2,3,4,6-Tetrachlorophenol	58-90-2	50	1600
5-Nitro-o-toluidine	99-55-8	20	660
Thionazin[2]	297-97-2	50	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp[2]	3689-24-5	50	1600
Phorate[2]	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Diallate[3]	2303-16-4	20	660
Dimethoate[2]	60-51-5	20	660
4-Aminobiphenyl	92-67-1	50	1600
Pentachloronitrobenzene	82-68-8	50	1600
Pronamide	23950-58-5	20	660
Disulfoton[2]	298-04-4	50	1600
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7	20	660
*Methyl Parathion[2]	298-00-0	50	1600

Table 2

STL North Canton Appendix IX[1] Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
4-Nitroquinoline-1-oxide	56-57-5	100	3300
*Parathion[2]	56-38-2	50	1600
Methapyrilene	91-80-5	50	1600
Aramite	140-57-8	20	660
*Isodrin[3]	465-73-6	10	330
*Kepone[2]	143-50-0	100	3300
Famphur[3]	52-85-7	100	3300
p-(Dimethylamino)azobenzene	60-11-7	20	660
p-Chlorobenzilate[3]	510-15-6	10	330
3,3'-Dimethylbenzidine	119-93-7	50	1600
2-Acetylaminofluorene	53-96-3	100	3300
Dibenz(a,j)acridine	224-42-0	20	660
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660
3-Methylcholanthrene	56-49-5	20	660

1 The Appendix IX standard contains additional analytes required for the Appendix IX list. The STL North Canton primary standard must also be analyzed to include all of the Appendix IX list

2 May also be analyzed by method 8140 or 8141, which can achieve lower reporting limits.

3 It is highly recommended that Famphur is analyzed by method 8081. It is a poor responder by 8270C.

* These compounds are analyzed by GC methods at STL North Canton.

Table 2A
STL North Canton Michigan Program[1]

Semivolatile	CAS Number	Michigan Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Acenaphthene	83-32-9	5	330
Acenaphthylene	208-96-8	5	330
Acetophenone	98-86-2	5	330
Anthracene	120-12-7	5	330
Atrzine	1912-24-9	5	330
Benzaldehyde	100-52-7	10	330
Benzo(a)anthracene	56-55-3	4	330
Benzo(a)pyrene	50-32-8	4	330
Benzo(b)fluoranthene	205-99-2	4	330
Benzo(g,h,i)perylene	191-24-2	5	330
Benzo(k)fluoranthene	207-08-9	5	330
1,1'-Biphenyl	92-52-4	10	330
4-Bromophenylphenyl ehter	101-55-3	5	330
Butylbenzylphthalate	85-68-7	5	330
di-n-Butylphthalate	84-74-2	5	330
Caprolactam	105-60-2	10	330
Carbazole	86-74-8	10	330
4-Chloroaniline	106-47-8	20	1700
bis(2-Chloroethoxy)methane	111-91-1	5	330
bis(2-Chloroethyl)ether	111-44-4	4	330
bis(2-Chloroisopropyl)ether	108-60-1	5	330
4-Chloro-3-Methylphenol	59-50-7	5	330
2-Chloronaphthalene	91-58-7	5	330
2-Chlorophenol	95-57-8	5	330
4-Chlorophenyl phenyl ether	7005-72-3	5	330
Chrysene	218-01-9	5	330
Dibenz(a,h)anthracene	53-70-3	4	330
Dibenzofuran	132-64-9	5	330
3,3'-Dichlorobenzidine	91-94-1	4	2000
2,4-Dichlorophenol	120-83-2	10	330
Diethylphthalate	84-66-2	5	330
2-4-Dimethylphenol	105-67-9	5	330
Dimethylphthalate	131-11-3	5	330
4,6-Dinitro-2-methylphenol	534-52-1	20	1700
2,4-Dinitrophenol	51-28-5	20	1700
2,4-Dinitrotoluene	121-14-2	5	330
2,6-Dinitrotoluene	606-20-2	5	330
bis(2-Ethylhexyl)phthalate	117-81-7	5	330
Fluoranthene	206-44-0	5	330
Fluorene	86-73-7	5	330
Hexachlorobenzene	118-74-1	5	330
Hexachlorobutadiene	87-68-3	5	330
Hexachlorocyclopentadiene	77-47-4	5	330

Table 2A
STL North Canton Michigan Program[1]

Semivolatile	CAS Number	Michigan Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Hexachloroethane	67-72-1	5	330
Indeno(1,2,3-cd)pyrene	193-39-5	4	330
Isophorone	78-59-1	5	330
2-Methylnapthalene	91-57-6	5	330
2-Methylphenol	95-48-7	5	330
4-Methylphenol	106-44-5	5	330
Naphthalene	91-20-3	5	330
2-Nitroaniline	88-74-4	20	1700
3-Nitroaniline	99-09-2	20	1700
4-Nitroaniline	100-01-6	20	1700
Nitrobenzene	95-95-3	4	330
2-Nitrophenol	88-75-5	5	330
4-Nitrophenol	100-02-7	20	1700
N-Nitroso-di-n-propylamine	621-64-7	5	330
N-Nitrosodiphenylamine (diphenylamine)	62-75-9	5	330
di-n-Octylphthalate	117-84-0	5	330
Pentachlorophenol	87-86-5	20	800
Phenanthrene	85-01-8	5	330
Phenol	108-95-2	5	330
Pyrene	129-00-0	5	330
2,4,5-Trichlorophenol	95-95-4	5	330
2,4,6-Trichlorophenol	88-06-2	4	330

[1] Reporting Limits are only for samples performed under the Michigan program.

Table 3

Reportable Analytes for STL North Canton Standard Tests, Primary Standard

Analyte	CAS Number	STL North Canton Standard List	TCLP	TCL	Appendix IX
Pyridine	110-86-1		X		X
N-nitrosodimethylamine	62-75-9				X
Aniline	62-53-3				X
Phenol	108-95-2	X		X	X
Bis(2-chloroethyl)ether	111-44-4	X		X	X
2-Chlorophenol	95-57-8	X		X	X
1,3-Dichlorobenzene	541-73-1	X		X	X
1,4-Dichlorobenzene	106-46-7	X	X	X	X
Benzyl alcohol	100-51-6				X
1,2-Dichlorobenzene	95-50-1	X		X	X
2-Methylphenol	95-48-7	X	X	X	X
2,2'-oxybis(1-chloropropane)[1]	180-60-1	X		X	X

Table 3

Reportable Analytes for STL North Canton Standard Tests, Primary Standard

Analyte	CAS Number	STL North Canton Standard List	TCLP	TCL	Appendix IX
4-Methylphenol	106-44-5	X	X	X	X
N-Nitroso-di-n-propylamine	621-64-7	X		X	X
Hexachloroethane	67-72-1	X	X	X	X
Nitrobenzene	98-95-3	X	X	X	X
Isophorone	78-59-1	X		X	X
2-Nitrophenol	88-75-5	X		X	X
2,4-Dimethylphenol	105-67-9	X		X	X
Benzoic acid	65-85-0				
Bis(2-chloroethoxy)methane	111-91-1	X		X	X
2,4-Dichlorophenol	120-83-2	X		X	X
1,2,4-Trichlorobenzene	120-82-1	X		X	X
Naphthalene	91-20-3	X		X	X
4-Chloroaniline	106-47-8	X		X	X
Hexachlorobutadiene	87-68-3	X	X	X	X
4-Chloro-3-methylphenol	59-50-7	X		X	X
2-Methylnaphthalene	91-57-6	X		X	X
Hexachlorocyclopentadiene	77-47-4	X		X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X	X
2,4,5-Trichlorophenol	95-95-4	X	X	X	X
2-Chloronaphthalene	91-58-7	X		X	X
2-Nitroaniline	88-74-4	X		X	X
Dimethyl phthalate	131-11-3	X		X	X
Acenaphthylene	208-96-8	X		X	X
3-Nitroaniline	99-09-2	X		X	X
Acenaphthene	83-32-9	X		X	X
2,4-Dinitrophenol	51-28-5	X		X	X
4-Nitrophenol	100-02-7	X		X	X
Dibenzofuran	132-64-9	X		X	X
2,4-Dinitrotoluene	121-14-2	X	X	X	X
2,6-Dinitrotoluene	606-20-2	X		X	X
Diethylphthalate	84-66-2	X		X	X
4-Chlorophenyl phenyl ether	7005-72-3	X		X	X
Fluorene	86-73-7	X		X	X
4-Nitroaniline	100-01-6	X		X	X
4,6-Dinitro-2-methylphenol	534-52-1	X		X	X
N-Nitrosodiphenylamine	86-30-6	X		X	X
Azobenzene[4]	103-33-3				
4-Bromophenyl phenyl ether	101-55-3	X		X	X
Hexachlorobenzene	118-74-1	X	X	X	X
Pentachlorophenol	87-86-5	X	X	X	X
Phenanthrene	85-01-8	X		X	X
Anthracene	120-12-7	X		X	X
Carbazole	86-74-8	X		X	
Di-n-butyl phthalate	84-74-2	X		X	X

Table 3

Reportable Analytes for STL North Canton Standard Tests, Primary Standard

Analyte	CAS Number	STL North Canton Standard List	TCLP	TCL	Appendix IX
Fluoranthene	206-44-0	X		X	X
Benzidine	92-87-5				
Pyrene	129-00-0	X		X	X
Butyl benzyl phthalate	85-68-7	X		X	X
3,3'-Dichlorobenzidine	91-94-1	X		X	X
Benzo(a)anthracene	56-55-3	X		X	X
Bis(2-ethylhexyl)phthalate	117-81-7	X		X	X
Chrysene	218-01-9	X		X	X
Di-n-octylphthalate	117-84-0	X		X	X
Benzo(b)fluoranthene	205-99-2	X		X	X
Benzo(k)fluoranthene	207-08-9	X		X	X
Benzo(a)pyrene	50-32-8	X		X	X
Indeno(1,2,3-cd)pyrene	193-39-5	X		X	X
Dibenz(a,h)anthracene	53-70-3	X		X	X
Benzo(g,h,i)perylene	191-24-2	X		X	X
Benzaldehyde	100-52-7			X	
Caprolactam	105-60-2			X	
1,1-Biphenyl	92-52-4			X	
Atrazine	1912-24-9			X	

[1] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

[2] Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 4

Reportable analytes for STL North Canton Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	STL North Canton Standard List	TCLP	TCL	Appendix IX
2-Picoline	109-06-8				X
N-Nitrosomethylethylamine	10595-95-6				X
Methyl methanesulfonate	66-27-3				X
N-Nitrosodiethylamine	55-18-5				X
Ethyl methanesulfonate	62-50-0				X
Pentachloroethane	76-01-7				X
Acetophenone	98-86-2			X	X
N-Nitrosopyrrolidine	930-55-2				X
N-Nitrosomorpholine	59-89-2				X
o-Toluidine	95-53-4				X
3-Methylphenol	108-39-4				X
N-Nitrosopiperidine	100-75-4				X
o,o,o-Triethyl-Phosphorothioate[2]	126-68-1				X
a,a-Dimethyl-phenethylamine	122-09-8				X
2,6-Dichlorophenol	87-65-0				X
Hexachloropropene	1888-71-7				X
p-Phenylenediamine	106-50-3				X
n-Nitrosodi-n-butylamine	924-16-3				X
Safrole	94-59-7				X
1,2,4,5-Tetrachlorobenzene	95-94-3				X
Isosafrole	120-58-1				X
1,4-Dinitrobenzene	100-25-4				
1,4-Naphthoquinone	130-15-4				X
1,3-Dinitrobenzene	99-65-0				X
Pentachlorobenzene	608-93-5				X
1-Naphthylamine	134-32-7				X
2-Naphthylamine	91-59-8				X
2,3,4,6-Tetrachlorophenol	58-90-2				X
5-Nitro-o-toluidine	99-55-8				X
Thionazin[2]	297-97-2				X
1,3,5-Trinitrobenzene	99-35-4				X
Sulfotepp[2]	3689-24-5				X
Phorate[2]	298-02-2				X
Phenacetin	62-44-2				X
Diallate	2303-16-4				X
Dimethoate[2]	60-51-5				X
4-Aminobiphenyl	92-67-1				X
Pentachloronitrobenzene	82-68-8				X
Pronamide	23950-58-5				X
Disulfoton[2]	298-04-4				X
2-secbutyl-4,6-dinitrophenol (Dinoseb)[2]	88-85-7				X
*Methyl parathion[2]	298-00-0				X

Table 4

Reportable analytes for STL North Canton Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	STL North Canton Standard List	TCLP	TCL	Appendix IX
4-Nitroquinoline-1-oxide	56-57-5				X
*Parathion[2]	56-38-2				X
*Isodrin[3]	465-73-6				X
*Kepone [2]	143-50-0				X
Famphur[2]	52-85-7				X
Methapyrilene	91-80-5				X
Aramite	140-57-8				X
p-(Dimethylamino)azobenzene	60-11-7				X
p-Chlorobenzilate[3]	510-15-6				X
3,3'-Dimethylbenzidine	119-93-7				X
2-Acetylaminofluorene	53-96-3				X
Dibenz(a,j)acridine	224-42-0				
7,12-Dimethylbenz(a)anthracene	57-97-6				X
3-Methylcholanthrene	56-49-5				X
Hexachlorophene[4]	70-30-4				X
Diphenylamine[5]	122-39-4				X

[2] May also be analyzed by method 8140 or 8141, which can achieve lower reporting limits.

[3] May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits

[4] Hexachlorophene is a required analyte for Appendix IX. This compound is not stable, and therefore not included in the calibration standard. The characteristic ions for hexachlorophene are searched for in the chromatogram. (See section 12.2.1)

[5] Diphenylamine is a required compound for Appendix IX. N-nitrosodiphenylamine decomposes in the injection port to form diphenylamine. Therefore these two compounds cannot be distinguished. Diphenylamine is not included in the calibration standard.

* These compounds are analyzed by GC methods at STL North Canton.

Table 5

Suggested Instrumental Conditions

Mass Range	35-500 amu
Scan Time	≤1 second/scan
Initial Column Temperature/Hold Time	40°C for 2 minutes
Column Temperature Program	40 - 320°C at 11.5°C/min
Final Column Temperature/Hold Time	320°C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature	250 - 300°C
Transfer Line Temperature	250 - 300°C
Source Temperature	According to manufacturer's specifications
Injector	Grob-type, split / splitless
Sample Volume	1 or 2 µl
Carrier Gas	Helium at 30 cm/sec

Table 6

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 - 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17 - 23% of mass 442

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
N-nitrosodimethylamine	74	42	
Pyridine	79	52	
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d5 (Surrogate Standard)	99	42	71
Benzaldehyde	77	105	106
Aniline	93	66	
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	79
2,2'-oxybis(1-chloropropane)[1]	45	77	79
4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d8 (Internal Standard)	136	68	54
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
Caprolactam	113	55	56
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	115
Hexachlorocyclopentadiene	237	235	272
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
1,1'-Biphenyl	154	153	76
2-Fluorobiphenyl (Surrogate Standard)	172	171	170
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
Acenaphthylene	152	151	153
2,6-Dinitrotoluene	165	63	89
Acenaphthene-d10 (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153	152	154
2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	84
4-Nitrophenol	109	139	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	182	77
N-Nitrosodiphenylamine	169	168	167
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141
Azobenzene	77	182	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Atrazine	200	173	215
Pentachlorophenol	266	264	268
Phenanthrene-d10 (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	100
Benzidine	184	92	185
Pyrene	202	101	100
Terphenyl-d14 (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Benzo(a)Anthracene	228	229	226
Chrysene-d12 (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d12 (Internal Standard)	264	260	265
Indeno(1,2,3-cd)pyrene	276	138	277
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
2-Picoline	93	66	92
N-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
N-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Pentachloroethane	117	119	167
Acetophenone	105	77	120
N-Nitrosopyrrolidine	100	41	42
N-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	
3-Methylphenol	108	107	77
N-Nitrosopiperidine	114	42	55
o,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
p-Phenylenediamine	108	80	
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole 1	162	104	131
Isosafrole 2	162	104	131
1,4-Dinitrobenzene	168	75	122
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	75	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	
2-Naphthylamine	143	115	
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	97	96	143
1,3,5-Trinitrobenzene	213	75	120
Sulfotepp	97	322	202
Phorate	75	97	121
Phenacetin	108	179	109
Diallate	86	234	
Dimethoate	87	93	125
4-Aminobiphenyl	169		
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263
4-Nitroquinoline-1-oxide	190	128	160

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
Parathion	109	97	291
Isodrin	193	66	195
Kepone	272	274	237
Famphur	218	125	93
Methapyrilene	97	58	
Aramite 1	185	319	
Aramite 2	185	319	
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	
2-Acetylaminofluorene	181	180	223
Dibenz(a,j)acridine	279	280	
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

Table 9

8270C LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract[1]
1,2,4-Trichlorobenzene	100
Acenaphthene	100
2,4-Dinitrotoluene	100
Pyrene	100
N-Nitroso-di-n-propylamine	100
1,4-Dichlorobenzene	100
Pentachlorophenol	150
Phenol	150
2-Chlorophenol	150
4-Chloro-3-methylphenol	150
4-Nitrophenol	150

[1] Levels are 50 and 75 ng/µL if 2 µL injection is used

Table 10

TCLP LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract[1]
1,4-Dichlorobenzene	100
2,4-Dinitrotoluene	100
Hexachlorobenzene	100
Hexachlorobutadiene	100
Hexachloroethane	100
2-Methylphenol	100
3-Methylphenol	100
4-Methylphenol	100
Nitrobenzene	100
Pentachlorophenol	100
Pyridine	100
2,4,5-Trichlorophenol	100
2,4,6-Trichlorophenol	100

[1] Levels are 50 ng/µL if 2 µL injection is used

Recovery limits for the LCS and for matrix spikes are generated from historical data and are maintained by the QA department.

Table 11

8270C Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract[2]
Nitrobenzene-d5	100
2-Fluorobiphenyl	100
Terphenyl-d14	100
1,2-Dichlorobenzene-d4[1]	100
Phenol-d5	150
2-Fluorophenol	150
2,4,6-Tribromophenol	150
2-Chlorophenol-d4[1]	150

[1] Included in standard mix, but not routinely evaluated for method 8270B

[2] Levels are 50 and 75 ng/µL if 2 µL injection is used

Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 12[3]
Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
Pyridine	10	25	40	60	80
N-nitrosodimethylamine	10	25	40	60	80
Aniline	10	25	40	60	80
Phenol	10	25	40	60	80
Bis(2-chloroethyl)ether	10	25	40	60	80
2-Chlorophenol	10	25	40	60	80
1,3-Dichlorobenzene	10	25	40	60	80
1,4-Dichlorobenzene	10	25	40	60	80
Benzyl alcohol	10	25	40	60	80
1,2-Dichlorobenzene	10	25	40	60	80
2-Methylphenol	10	25	40	60	80
2,2'-oxybis(1-chloropropane)[1]	10	25	40	60	80
4-Methylphenol	10	25	40	60	80
N-Nitroso-di-n-propylamine	10	25	40	60	80
Hexachloroethane	10	25	40	60	80
Nitrobenzene	10	25	40	60	80
Isophorone	10	25	40	60	80
2-Nitrophenol	10	25	40	60	80
2,4-Dimethylphenol	10	25	40	60	80
Benzoic acid	10	25	40	60	80
Bis(2-chloroethoxy)methane	10	25	40	60	80
2,4-Dichlorophenol	10	25	40	60	80
1,2,4-Trichlorobenzene	10	25	40	60	80
Naphthalene	10	25	40	60	80
4-Chloroaniline	10	25	40	60	80
Hexachlorobutadiene	10	25	40	60	80
4-Chloro-3-methylphenol	10	25	40	60	80
2-Methylnaphthalene	10	25	40	60	80
Hexachlorocyclopentadiene	10	25	40	60	80
2,4,6-Trichlorophenol	10	25	40	60	80
2,4,5-Trichlorophenol	10	25	40	60	80
2-Chloronaphthalene	10	25	40	60	80
2-Nitroaniline	10	25	40	60	80
Dimethyl phthalate	10	25	40	60	80
Acenaphthylene	10	25	40	60	80
3-Nitroaniline	10	25	40	60	80
Acenaphthene	10	25	40	60	80
2,4-Dinitrophenol	10	25	40	60	80
4-Nitrophenol	10	25	40	60	80
Dibenzofuran	10	25	40	60	80
2,4-Dinitrotoluene	10	25	40	60	80
2,6-Dinitrotoluene	10	25	40	60	80
Diethylphthalate	10	25	40	60	80
4-Chlorophenyl phenyl ether	10	25	40	60	80
Fluorene	10	25	40	60	80
4-Nitroaniline	10	25	40	60	80

Table 12[3]
Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
4,6-Dinitro-2-methylphenol	10	25	40	60	80
N-Nitrosodiphenylamine	10	25	40	60	80
Azobenzene[2]	10	25	40	60	80
4-Bromophenyl phenyl ether	10	25	40	60	80
Hexachlorobenzene	10	25	40	60	80
Pentachlorophenol	10	25	40	60	80
Phenanthrene	10	25	40	60	80
Anthracene	10	25	40	60	80
Carbazole	10	25	40	60	80
Di-n-butyl phthalate	10	25	40	60	80
Fluoranthene	10	25	40	60	80
Benzidine	10	25	40	60	80
Pyrene	10	25	40	60	80
Butyl benzyl phthalate	10	25	40	60	80
3,3'-Dichlorobenzidine	10	25	40	60	80
Benzo(a)anthracene	10	25	40	60	80
Bis(2-ethylhexyl)phthalate	10	25	40	60	80
Chrysene	10	25	40	60	80
Di-n-octylphthalate	10	25	40	60	80
Benzo(b)fluoranthene	10	25	40	60	80
Benzo(k)fluoranthene	10	25	40	60	80
Benzo(a)pyrene	10	25	40	60	80
Indeno(1,2,3-cd)pyrene	10	25	40	60	80
Dibenz(a,h)anthracene	10	25	40	60	80
Benzo(g,h,i)perylene	10	25	40	60	80
Benzaldehyde	10	25	40	60	80
Caprolactam	10	25	40	60	80
1,1'-Biphenyl	10	25	40	60	80
Atrazine	10	25	40	60	80

[1] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

[2]Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

[3]Optional concentration levels = 1/5 of the listed concentrations for samples with a final volume of 5 mL.

Table 13[1]

Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5
2-Picoline	10	25	40	60	80
N-Nitrosomethylethylamine	10	25	40	60	80
Methyl methanesulfonate	10	25	40	60	80
N-Nitrosodiethylamine	10	25	40	60	80
Ethyl methanesulfonate	10	25	40	60	80
Pentachloroethane	10	25	40	60	80
Acetophenone	10	25	40	60	80
N-Nitrosopyrrolidine	10	25	40	60	80
N-Nitrosomorpholine	10	25	40	60	80
o-Toluidine	10	25	40	60	80
3-Methylphenol	10	25	40	60	80
N-Nitrosopiperidine	10	25	40	60	80
o,o,o-Triethyl-Phosphorothioate	10	25	40	60	80
a,a-Dimethyl-phenethylamine	10	25	40	60	80
2,6-Dichlorophenol	10	25	40	60	80
Hexachloropropene	10	25	40	60	80
p-Phenylenediamine	10	25	40	60	80
n-Nitrosodi-n-butylamine	10	25	40	60	80
Safrole	10	25	40	60	80
1,2,4,5-Tetrachlorobenzene	10	25	40	60	80
Isosafrole 1 + 2	10	25	40	60	80
1,4-Dinitrobenzene	10	25	40	60	80
1,4-Naphthoquinone	10	25	40	60	80
1,3-Dinitrobenzene	10	25	40	60	80
Pentachlorobenzene	10	25	40	60	80
1-Naphthylamine	10	25	40	60	80
2-Naphthylamine	10	25	40	60	80
2,3,4,6-Tetrachlorophenol	10	25	40	60	80
5-Nitro-o-toluidine	10	25	40	60	80
Thionazin	10	25	40	60	80
1,3,5-Trinitrobenzene	10	25	40	60	80
Sulfotepp	10	25	40	60	80
Phorate	10	25	40	60	80
Phenacetin	10	25	40	60	80
Diallate 1 + 2	10	25	40	60	80
Dimethoate	10	25	40	60	80
4-Aminobiphenyl	10	25	40	60	80
Pentachloronitrobenzene	10	25	40	60	80
Pronamide	10	25	40	60	80
Disulfoton	10	25	40	60	80
2-secbutyl-4,6-dinitrophenol (Dinoseb)	10	25	40	60	80
*Methyl parathion	10	25	40	60	80
4-Nitroquinoline-1-oxide	10	25	40	60	80
*Parathion	10	25	40	60	80

Table 13[1]

Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5
*Isodrin	10	25	40	60	80
*Kepone	10	25	40	60	80
Famphur	10	25	40	60	80
Methapyrilene	10	25	40	60	80
Aramite 1 and 2	10	25	40	60	80
p-(Dimethylamino)azobenzene	10	25	40	60	80
p-Chlorobenzilate	10	25	40	60	80
3,3'-Dimethylbenzidine	10	25	40	60	80
2-Acetylaminofluorene	10	25	40	60	80
Dibenz (a,j)acridine	10	25	40	60	80
7,12-Dimethylbenz(a)anthracene	10	25	40	60	80
3-Methylcholanthrene	10	25	40	60	80

* These compounds are not in the STL North Canton Standard Mix,

[2]Optional concentration levels = 1/5 of the listed concentrations for samples with a final volume of 5 mL.

Table 14
Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
Acenaphthene	60	27.6	60.1-132.3
Acenaphthylene	60	40.2	53.5-126.0
Aldrin[1]	60	39.0	7.2-152.2
Anthracene	60	32.0	43.4-118.0
Benz(a)anthracene	60	27.6	41.8-133.0
Benzo(b)fluoranthene	60	38.8	42.0-140.4
Benzo(k)fluoranthene	60	32.3	25.2-145.7
Benzo(a)pyrene	60	39.0	31.7-148.0
Benzo(ghi)perylene	60	58.9	D-195.0
Benzylbutyl phthalate	60	23.4	D-139.9
B-BHC[1]	60	31.5	41.5-130.6
d-BHC[1]	60	21.6	D-100.0
Bis(2-chloroethyl) ether	60	55.0	42.9-126.0
Bis(2-chloroethoxy)methane	60	34.5	49.2-164.7
Bis(2-chloroisopropyl) ether	60	46.3	62.8-138.6
Bis(2-ethylhexyl) phthalate	60	41.1	28.9-136.8
4-Bromophenyl phenyl ether	60	23.0	64.9-114.4
2-Chloronaphthalene	60	13.0	64.5-113.5
4-Chlorophenyl phenyl ether	60	33.4	38.4-144.7
Chrysene	60	48.3	44.1-139.9
4,4'-DDD[1]	60	31.0	D-134.5

Table 14
Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
4,4'-DDE[1]	60	32.0	19.2-119.7
4,4'-DDT[1]	60	61.6	D-170.6
Dibenzo(a,h)anthracene	60	70.0	D-199.7
Di-n-butyl phthalate	60	16.7	8.4-111.0
1,2-Dichlorobenzene	60	30.9	48.6-112.0
1,3-Dichlorobenzene	60	41.7	16.7-153.9
1,4-Dichlorobenzene	60	32.1	37.3-105.7
3,3'-Dichlorobenzidine	60	71.4	8.2-212.5
Dieldrin[1]	60	30.7	44.3-119.3
Diethyl phthalate	60	26.5	D-100.0
Dimethyl phthalate	60	23.2	D-100.0
2,4-Dinitrotoluene	60	21.8	47.5-126.9
2,6-Dinitrotoluene	60	29.6	68.1-136.7
Di-n-octylphthalate	60	31.4	18.6-131.8
Endosulfan sulfate[1]	60	16.7	D-103.5
Endrin aldehyde	60	32.5	D-188.8
Fluoranthene	60	32.8	42.9-121.3
Fluorene	60	20.7	71.6-108.4
Heptachlor[1]	60	37.2	D-172.2
Heptachlor epoxide[1]	60	54.7	70.9-109.4
Hexachlorobenzene	60	24.9	7.8-141.5
Hexachlorobutadiene	60	26.3	37.8-102.2
Hexachloroethane	60	24.5	55.2-100.0
Indeno(1,2,3-cd)pyrene	60	44.6	D-150.9
Isophorone	60	63.3	46.6-180.2
Naphthalene	60	30.1	35.6-119.6
Nitrobenzene	60	39.3	54.3-157.6
N-Nitrosodi-n-propylamine	60	55.4	13.6-197.9
PCB-1260[1]	60	54.2	19.3-121.0
Phenanthrene	60	20.6	65.2-108.7
Pyrene	60	25.2	69.6-100.0
1,2,4-Trichlorobenzene	60	28.1	57.3-129.2
4-Chloro-3-methylphenol	60	37.2	40.8-127.9
2-Chlorophenol	60	28.7	36.2-120.4
2,4-Chlorophenol	60	26.4	52.5-121.7
2,4-Dimethylphenol	60	26.1	41.8-109.0
2,4-Dinitrophenol	60	49.8	D-172.9
2-Methyl-4,6-dinitrophenol	60	93.2	53.0-100.0
2-Nitrophenol	60	35.2	45.0-166.7
4-Nitrophenol	60	47.2	13.0-106.5
Pentachlorophenol	60	48.9	38.1-151.8
Phenol	60	22.6	16.6-100.0

Table 14
Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
2,4,6-Trichlorophenol	60	31.7	52.4-129.2

[1]Since the organochlorine pesticides and PCBs are normally determined by method 8080 at STL North Canton, they will not be included in the initial demonstration of capability for method 8270B.

ATTACHMENT A

MODIFICATIONS REQUIRED FOR ANALYSIS OF WASTEWATER FOLLOWING METHOD 625

18. REQUIREMENTS FOR METHOD 625

18.1 Method 625 is required for demonstration of compliance with NPDES wastewater discharge permits. The standard analyte list and reporting limits are listed in Table A-1.

18.2 This method can be applied only to aqueous matrices.

18.3 The tune period for this method is defined as 24 hours.

18.4 Initial calibration curve requirements:

18.4.1 The initial calibration curve for this method requires at least three points.

18.4.2 Target compounds must have RSD ≤ 35%.

18.4.3 If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds.

18.5 Continuing calibration verification requirements: All target compounds must have %D ≤ 20%.

18.6 Matrix Spike and LCS requirements:

18.6.1 A full analyte spike is required for method 625. The spiking levels are given in Table A-2.

Table A-1. STL North Canton Method 625 standard reporting list and reporting limits.

Analytes	CAS Number	Aqueous µg/L
Phenol	108-95-2	10
Bis(2-chloroethyl)ether	111-44-4	10
2-Chlorophenol	95-57-8	10
1,3-Dichlorobenzene	541-73-1	10
1,4-Dichlorobenzene	106-46-7	10
1,2-Dichlorobenzene	95-50-1	10
2,2'-oxybis(1-chloropropane)	108-60-1	10
N-Nitroso-di-n-propylamine	621-64-7	10
Hexachloroethane	67-72-1	10
Nitrobenzene	98-95-3	10
Isophorone	78-59-1	10
2-Nitrophenol	88-75-5	10
2,4-Dimethylphenol	105-67-9	10
Bis(2-chloroethoxy)methane	111-91-1	10
2,4-Dichlorophenol	120-83-2	10
1,2,4-Trichlorobenzene	120-82-1	10
Naphthalene	91-20-3	10
Hexachlorobutadiene	87-68-3	10
4-Chloro-3-methylphenol	59-50-7	10
Hexachlorocyclopentadiene	77-47-4	50
2,4,6-Trichlorophenol	88-06-2	10
2-Chloronaphthalene	91-58-7	10
Dimethyl phthalate	131-11-3	10
Acenaphthylene	208-96-8	10
Acenaphthene	83-32-9	10
2,4-Dinitrophenol	51-28-5	50
4-Nitrophenol	100-02-7	50
2,4-Dinitrotoluene	121-14-2	10
2,6-Dinitrotoluene	606-20-2	10
Diethylphthalate	84-66-2	10
4-Chlorophenyl phenyl ether	7005-72-3	10
Fluorene	86-73-7	10
4,6-Dinitro-2-methylphenol	534-52-1	50
N-Nitrosodiphenylamine	86-30-6	10
4-Bromophenyl phenyl ether	101-55-3	10
Hexachlorobenzene	118-74-1	10
Pentachlorophenol	87-86-5	50
Phenanthrene	85-01-8	10
Anthracene	120-12-7	10
Di-n-butyl phthalate	84-74-2	10
Fluoranthene	206-44-0	10
Benzidine	92-87-5	100
Pyrene	129-00-0	10

Analytes	CAS Number	Aqueous µg/L
Butyl benzyl phthalate	85-68-7	10
3,3'-Dichlorobenzidine	91-94-1	50
Benzo(a)anthracene	56-55-3	10
Bis(2-ethylhexyl)phthalate	117-81-7	10
Chrysene	218-01-9	10
Di-n-octylphthalate	117-84-0	10
Benzo(b)fluoranthene	205-99-2	10
Benzo(k)fluoranthene	207-08-9	10
Benzo(a)pyrene	50-32-8	10
Indeno(1,2,3-cd)pyrene	193-39-5	10
Dibenz(a,h)anthracene	53-70-3	10
Benzo(g,h,i)perylene	191-24-2	10

Table A-2. Method 625 LCS and MS compounds and spike concentrations.

LCS Compounds	Spiking Level, ng/µL in extract[1]
Phenol	100
Bis(2-chloroethyl)ether	100
2-Chlorophenol	100
1,3-Dichlorobenzene	100
1,4-Dichlorobenzene	100
1,2-Dichlorobenzene	100
2,2'-oxybis(1-chloropropane)	100
N-Nitroso-di-n-propylamine	100
Hexachloroethane	100
Nitrobenzene	100
Isophorone	100
2-Nitrophenol	100
2,4-Dimethylphenol	100
Bis(2-chloroethoxy)methane	100
2,4-Dichlorophenol	100
1,2,4-Trichlorobenzene	100
Naphthalene	100
Hexachlorobutadiene	100
4-Chloro-3-methylphenol	100
Hexachlorocyclopentadiene	100
2,4,6-Trichlorophenol	100
2-Chloronaphthalene	100
Dimethyl phthalate	100
Acenaphthylene	100
Acenaphthene	100
2,4-Dinitrophenol	100
4-Nitrophenol	100
2,4-Dinitrotoluene	100
2,6-Dinitrotoluene	100
Diethylphthalate	100
4-Chlorophenyl phenyl ether	100
Fluorene	100
4,6-Dinitro-2-methylphenol	100
N-Nitrosodiphenylamine	100
4-Bromophenyl phenyl ether	100
Hexachlorobenzene	100
Pentachlorophenol	100
Phenanthrene	100
Anthracene	100
Di-n-butyl phthalate	100
Fluoranthene	100
Benzidine	100
Pyrene	100
Butyl benzyl phthalate	100
3,3'-Dichlorobenzidine	100

LCS Compounds	Spiking Level, ng/µL in extract[1]
Benzo(a)anthracene	100
Bis(2-ethylhexyl)phthalate	100
Chrysene	100
Di-n-octylphthalate	100
Benzo(b)fluoranthene	100
Benzo(k)fluoranthene	100
Benzo(a)pyrene	100
Indeno(1,2,3-cd)pyrene	100
Dibenz(a,h)anthracene	100
Benzo(g,h,i)perylene	100

[1] **Levels are 50 and 75 ng/µL if 2 µL injection is used**

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STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: GAS CHROMATOGRAPHIC ANALYSIS BASED ON METHOD 8000B, 8021B, 8081A, 8082, 8151A, 8310, 8141A, 8015B, 608, 610 and Wisconsin DNR Modified DRO Method

(SUPERSEDES: Revision 5.5 Dated 03/16/01)

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1. SCOPE AND APPLICATION

This SOP describes procedures for analysis of organic analytes by Gas Chromatography (GC). The procedures are based on SW-846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA). Individual analytes and methods are described in the appendices. Appendices B, C, and E include criteria for the analysis of wastewater by Methods 608 and 610. Appendix G describes procedures for the analysis of petroleum hydrocarbons by SW-846 methodology and the Wisconsin DNR Modified DRO method. Appendix H includes criteria for the analysis of non-halogenated organic compounds by Method 8015B, Direct Injection. Appendix I describes the analysis of Phillips 66 analytes by Method 8015B.

2. SUMMARY OF METHOD

In general, semivolatile analytes in aqueous samples are prepared for analysis using continuous or separatory funnel liquid / liquid extraction or solid phase extraction (SOP # CORP-OP-0001NCNC) Solid samples are prepared using sonication, Soxhlet or pressurized fluid extraction (SOP # CORP-OP-0001NC). Volatile analytes are prepared for analysis using purge and trap methodology (Appendix A).

After the initial preparation step, the sample is introduced to the GC and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. Internal or external standardization procedures are used as specified in the method appendices.

3. DEFINITIONS

Definitions of terms used in this SOP may be found in the glossary of the STL North Canton Laboratory Quality Manual (LQM), current version.

4. INTERFERENCES

Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. In addition, some purge and trap autosamplers are susceptible to port specific contamination. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results. In particular, this is a problem with non-selective detectors such as the Flame Ionization Detector (FID). See the appendices for interferences specific to individual tests and suggested corrective actions.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL North Canton associates. The following requirements must be met:

Eye protection that prevents spalsh, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately. Refer to the STL North Canton Chemical Hygiene plan for a complete description of personal protection equipment.

The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. Specific hazards are covered in the appendices.

5.1.1. Opened containers of neat standards will be handled in a fume hood.

5.2. Sample extracts and standards, which are in a flammable solvent, shall be stored in an explosion-proof refrigerator.

5.3. When using hydrogen gas as a carrier, all precautions listed in the CHP shall be observed.

5.4. Standard preparation and dilution shall be performed inside an operating fume hood.

6. EQUIPMENT AND SUPPLIES

An analytical system complete with a gas chromatograph is required. A data system capable of measuring peak area and/or height is required. Recommended equipment and supplies for individual methods are listed in each method appendix.

7. REAGENTS AND STANDARDS

7.1. Stock Standards

Stock standards are purchased as certified solutions or prepared from pure solutions. Stock standards for method 8021B are stored at -10 to -20°C. Other stock standard solutions are stored as recommended by the manufacturer. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.

Semivolatile stock standard solutions must be replaced after one year. Stock standards of gases must be replaced at least every week, unless the acceptability of the standard is demonstrated (Less than 20% drift from the initial calibration is an acceptable demonstration). Other volatile stock standards must be replaced every 6 months or sooner if comparison with check standards prepared from an independent source indicates a problem.

7.1.1. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampoule is opened, if the standard is supplied in a sealed ampoule. If a vendor-supplied standard has an earlier expiration date then that date is used.

7.2. Calibration Standards

7.2.1. Volatile Calibration Standards

The procedure for preparation of volatile standards is given in Appendix A.

7.2.2. Semivolatile Calibration Standards

Semivolatile calibration standards are prepared as dilutions of the stock standards. Surrogates and internal standards are used as specified in the method appendices. Semivolatile calibration solutions must be refrigerated at $\leq$6°C and protected from light. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.

7.3. Gases for carrier and make-up: Hydrogen, Helium, Nitrogen, Argon/Methane.

7.4. Quality control (QC) Standards

QC standards (matrix spiking and LCS standards) are prepared and stored in the same way as calibration standards. They must be made from a stock independent from the calibration standards.

8. SAMPLE PRESERVATION AND STORAGE

Semivolatile extracts must be refrigerated at $\leq$6°C and analyzed within 40 days of the end of the extraction. Volatile sample storage conditions and holding times are given in Appendix A.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2. Batch Definition

Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the STL North Canton QC Program document (QA-003) for further details of the batch definition.

9.2.1. Quality Control Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD a LCSD may be substituted.

9.3. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery +/- 3 standard deviations, unless that limit is tighter than the calibration criteria, in which case limits may be widened. Refer to policy QA-003 for more details.

9.3.1. These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported unless the dilution is more than 5X.

9.3.2. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.3.3. Refer to the QC Program document (QA-003) for further details of control limits.

9.4. Surrogates

All methods must use surrogates to the extent possible. Surrogate recoveries in samples and QC samples must be assessed to ensure that recoveries are within established limits. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.

- Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem. Repreparation is not necessary is there is obvious chromatographic interference.
- The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.4.1. If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in-control result is reported.

9.4.2. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then repreparation or flagging of the data is required.

9.4.3. Refer to the STL North Canton QC Program document (QA-003) for further details of the corrective actions.

9.5. Method Blanks

For each batch of samples, analyze a method blank. The method blank consists of reagent water for aqueous semivolatile samples, and sodium sulfate for semivolatile soils tests (Refer to SOP No. CORP-OP-0001NCNC for details). For low level volatiles, the method blank consists of reagent water. For medium level volatiles, the method blank consists of methanol as described in Appendix A. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone, phthalate esters) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

Re-extraction and reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.5.1. Refer to the STL North Canton QC Program document (QA-003) for further details of the corrective actions.

9.6. Instrument Blanks

9.6.1. An instrument blank must be analyzed during any 12-hour period of analysis that does not contain a method blank.

9.6.2. An instrument blank consists of the appropriate solvent with internal standards added. If internal standards are not used the surrogates should be added.

9.6.3. Control criteria are the same as for the method blank, except that only reanalysis of affected samples would be required, not re-extraction.

9.7. Laboratory Control Samples (LCS)

For each batch of samples, analyze a LCS. The LCS contains a representative subset of the analytes of interest, and must contain the same analytes as the matrix spike. The LCS may also contain the full set of analytes with a subset of control analytes. If any control analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and reanalysis of the batch; however, if the matrix spike and matrix spike duplicate are within limits; the batch may be acceptable.

9.7.1. Refer to the STL North Canton QC Program document (QA-003) for further details of the corrective action.

9.7.2. If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in control result is reported.

9.7.3. LCS compound lists are included in the appendices.

9.8. Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in the appendices. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed.
- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed.
- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, unless the matrix spike components would then be above the calibration range.

9.8.1. If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in control result is reported.

9.9. Quality Assurance Summaries

Certain clients may require specific project or program QC that may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.10. STL North Canton QC Program

Further details of QC and corrective action guidelines are presented in the STL QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION AND STANDARDIZATION

Internal or external calibration may be used. Internal calibration is recommended unless the sample matrix is likely to interfere with the quantitation of the internal standard. In either event prepare standards containing each analyte of interest at a minimum of five concentration levels. The low level standard should be at or

below the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in the appendices.

10.1. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, changing PID lamps or FID jets or replacing the ECD detector. A new calibration is not required after clipping the column, replacing the septum or syringe, or other minor maintenance.

10.2. With the exception of 10.3 below, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least 5 points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points. Third order calibrations require at least seven points.

10.3. A level may be removed from the calibration if the reason can be clearly documented, for example a broken vial or no purge run. A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration. All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

10.4. External standard calibration

Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach, introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak height or area response to the mass or concentration injected may be used to prepare a calibration curve.

$$Calibration\ Factor\,(CF) = \frac{Area\ or\ Height\ of\ Peak}{Mass\ Injected\,(ng)}$$

Some data systems may use the inverse of this formula. This is acceptable so long as the same formula is used for standards and samples. It is also possible to use the concentration of the standard rather than the mass injected. (This would require changes in the equations used to calculate the sample concentrations). Use of peak area or height must be consistent. However, if matrix interferences would make quantitation using peak area inaccurate for a particular sample, then peak height may be used as a substitute.

10.5. Internal standard calibration

10.5.1. The internal standard approach assumes that variations in instrument sensitivity, amount injected etc. can be corrected by determining the ratio of the response of the analyte to the response of an internal standard that has been added to the extract. To use this approach, select one or more internal standard(s) that are similar in analytical behavior to the compounds of interest. Recommended internal standards are given in the appendices. The analyst must demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. If the sample matrix interferes with quantitation of the internal standard, then the external standard approach must be used instead. In this event use the response factors from the previous continuing calibration to quantitate the analytes in the sample with the interference (applies only to the sample with the interference).

10.5.2. Introduce each calibration standard into the GC using the technique that will be used for samples. Response factors (RF) for each compound are calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

A_s = Response for the analyte to be measured
A_{is} = Response for the internal standard
C_{is} = Concentration of internal standard
C_s = Concentration of the analyte to be determined in the standard

10.6. Calibration curve fits

Average response factor, linear regression, or quadratic curves may be used to fit the data. Average response factor may be used if the average % RSD of the response factors or calibration factors of all the analytes in the calibration standard taken together is $\leq$ 20%. The average %RSD is calculated by summing the RSD value for each analyte and dividing by the total number of analytes.

10.6.1. In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits should only be used if the analyst has reason to believe that the average RF model does not fit the normal concentration/response behavior of the detector.

10.6.2. Average response factor

The average response factor may be used if the average percent relative standard deviation (%RSD) of all the response factors taken together is $\leq$ 20%.

The equation for average response factor is:

$$Average\ response\ factor = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where: n = Number of calibration levels

$\sum_{i=1}^{n} RF_i$ = Sum of response factors for each calibration level

10.6.3. Linear regression

The linear fit uses the following functions:

10.6.3.1. External Standard

$$y = ax + b$$

or

$$x = \frac{(y - b)}{a}$$

Where: y = Instrument response
x = Concentration
a = Slope
b = Intercept

10.6.3.2. Internal Standard

$$C_s = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{a}$$

Where: C_s = Concentration in the sample
A_s = Area of target peak in the sample
A_{is} = Area of internal standard in the sample
C_{is} = Concentration of the internal standard

10.6.4. Quadratic curve

The quadratic curve uses the following functions:

10.6.4.1. External standard

$$y = ax + cx^2 + b$$

Where c is the curvature

10.6.4.2. Internal Standard

$$y = a\left(\frac{A_s \times C_{is}}{A_{is}}\right) + c\left(\frac{A_s \times C_{is}}{A_{is}}\right)^2 + b$$

10.7. Evaluation of calibration curves

10.7.1. The percent relative standard error (%RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation.

10.7.2. The least squares regression line is calculated and used to calculate the predicted concentration for each level. The percent relative standard error is calculated as follows:

$$\%\ RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:
N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
C_i = True concentration for level i
PC_i = Predicted concentration for level i

Note that when average response factors are used, %RSE is equivalent to %RSD.

10.8. The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- If a curve is used, the intercept of the curve at zero response must be less than $\pm$ the reporting limit for the analyte.
- The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be $\leq$ 20%.
- Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (r) and Coefficient of Determination (r^2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on r. As a result a curve may have a very good correlation coefficient (>0.995), while also having > 100% error at the low point.

10.9. Weighting of data points

10.9.1. In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.10. Non-standard analytes are sometimes requested. For these analytes, it may be acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. This action must be with client approval. If the analyte is detected in any of the samples, a five point initial calibration must be generated and the sample(s) reanalyzed for quantitation.

10.11. Calibration Verification

10.11.1. 12 hour Calibration

The working calibration curve or RF must be verified by the analysis of a mid point calibration standard at the beginning, after every 12 hours, and at the end of the analysis sequence. The center of each retention time window is updated with each 12-hour calibration or calibration verification.

10.11.2. Calibration Verification

It may be appropriate to analyze a mid point standard more frequently than every 12 hours. If these calibration verification standards are analyzed, requirements are the same as the 12-hour calibration with the exception that retention times are not updated.

10.11.3. Any individual compounds with %D < 15% meet the calibration criteria. The calibration verification is also acceptable if the average of the %D for all the analytes is < 15%. This average is calculated by summing the entire absolute %D results in the calibration (including surrogates) and dividing by the number of analytes. Any analyte that is reportable as found must have a % difference of < 15% in the calibration verification or 12 hour calibration, on the column used for quantitation. Refer to section 12.1.2 for which result to report.

10.11.4. It is not necessary to run a calibration verification standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.11.5. Samples quantitated by external standard methods must be bracketed by calibration verification standards that meet the criteria listed above. The bracketing standards on the column used for calibration must meet the same criteria as the opening standards. Bracketing is not necessary for internal standard methods.

10.11.6. If the analyst notes that a CCV has failed and can document the reason for failure (e.g. no purge, broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria then the preceding samples have been successfully bracketed. If adjustments to the instrument are performed before the repeat CCV then the proceeding samples have not been successfully bracketed but analysis may continue.

10.11.7. In general, it is not advisable to analyze repeat CCVs on unattended runs. If repeat CCVs are analyzed then the first will serve as the bracketing standard for the preceding samples and the last will serve as the CCV for the following samples.

10.11.8. If highly contaminated samples are expected it is acceptable to analyze blanks or primers at any point in the run.

10.11.9. % Difference calculation

% Difference for internal and external methods is calculated as follows

Internal Standard:

$$\%D = \frac{RF_c - \overline{RF}}{\overline{RF}} \times 100$$

External standard:

$$\%D = \frac{CF_c - \overline{CF}}{\overline{CF}} \times 100$$

Where RF_c and CF_c are the response and calibration factors from the continuing calibration

$\overline{RF}$ and $\overline{CF}$ are the average response and calibration factors from the initial calibration

10.11.10. % Drift calculation

% Drift is used for comparing the continuing calibration to a linear or quadratic curve. The criteria for % drift are the same as for % difference

$$\%\ Drift = \frac{Calculated\ Conc.\ -\ Theoretical\ Conc.}{Theoretical\ Conc.} \times 100\%$$

10.11.11. Corrective Actions for Continuing Calibration

If the overall average %D of all analytes is greater than ± 15% corrective action must be taken. This may include clipping the column, changing the liner or other minor instrument adjustments, followed by reanalyzing the standard. If the overall average %D still varies by more than ± 15%, a new calibration curve must be prepared.

10.11.12. Corrective Action for Samples

For internal standard methods, any samples injected after a standard not meeting the calibration criteria must be re-injected.

For external standard methods, any samples injected after the last good continuing calibration standard must be re-injected.

If the average %D for all the analytes in the calibration is over 15%, but all of the analytes requested for a particular sample have %D ≤ 15%, then the analysis is acceptable for that sample.

11. PROCEDURE

11.1. Extraction

Extraction procedures are referenced in the appendices.

11.2. Cleanup

Cleanup procedures are referenced in the appendices.

11.3. Gas Chromatography

Chromatographic conditions for individual methods are presented in the appendices.

11.4. Sample Introduction

In general, volatiles analytes are introduced using purge and trap as described in Appendix A. Semivolatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

11.5. Analytical Sequence

An analytical sequence starts with an initial calibration or a daily calibration. Refer to the individual method appendices for method specific details of daily calibrations and analytical sequences.

11.5.1. The daily calibration includes analysis of standards containing all single response analytes and updating the retention time windows.

11.5.2. If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a daily calibration.

11.6. Retention Time Windows

11.6.1. Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day over a three-day period. Calculate the standard deviation of the three retention times for each analyte (relative retention times may also be used). For multi-response analytes (e.g., Aroclors) use the retention time of major peaks. Plus or minus three times the standard deviation of the retention times of each analyte defines the retention time window.

11.6.2. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows are updated with the mid- point of the initial calibration and each 12-hour calibration. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

11.6.3. If the retention time window as calculated above is less than +/- 0.05 minutes, use +/- 0.05 minutes as the retention time window. This allows for slight variations in retention times caused by sample matrix.

11.6.4. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

11.6.5. Corrective Action for Retention Times

The retention times of all compounds in the 12 hour calibration or calibration verification standard must be within the retention time window. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed unless the following conditions are met for any compound that elutes outside the retention time window:

The retention time of that compound in the standard must be within a retention time range equal to twice the original window.

No peak that would be reportable may be present on the sample chromatogram within an elution time range equal to three times the original retention time window.

11.7. Daily Retention Time Windows

The center of the retention time windows determined in Section 11.6 are adjusted to the retention time of each analyte as determined in the 12 hour calibration standards or continuing calibration verification standards. (See the method 8081A and 8082 appendices for exceptions for multi-response components.) The retention time windows must be updated at the beginning of each analytical sequence and with each 12-hour calibration or continuing calibration verification.

11.8. Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP CORP-OP-0001NC for determination of percent moisture.

11.9. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a supervisor and QA/QC manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. The nonconformance is also addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative Identification

12.1.1. Tentative identification occurs when a peak is found within the retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if J flags are required. Normally confirmation is required on a second column, but if the detector is sufficiently specific or if the sample matrix is well enough defined, single column analysis may be adequate. In some cases GC/MS confirmation may be required. Client specific requirements may also define the need for second column confirmation and / or GC/MS confirmation. Refer to the appendices for test specific requirements for confirmation. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmatory column, at a concentration greater than the reporting limit (MDL if J flag confirmation required).

12.1.2. Dual column quantitation

For confirmed results, two approaches are available to the analyst.
A) The primary column approach
Or
B) The better result approach
Both are acceptable to avoid the reporting of erroneous or unconfirmed data.

12.1.2.1. Primary column approach:

The result from the primary column is normally reported. The result from the secondary column is reported if any of the following three bulleted possibilities are true.

- There is obvious chromatographic interference on the primary column
- The result on the primary column is > 40% greater than the result on the secondary column
- Continuing or bracketing standard fails on the primary column but is acceptable on the secondary column. (If the primary column result is > 40% higher than the secondary and the primary column calibration fails, then the sample must be evaluated for reanalysis.)

12.1.2.2. Better result approach

The lower of the two results is normally reported. The lower result is considered better because the higher result is generally higher because of chromatographic interference. The higher result is reported if any of the following two bulleted possibilities are true.

- There is obvious chromatographic interference on the column with the lower result
- The continuing or bracketing calibration on the column with the lower result fails. (If the higher result is > 40% higher and the calibration on the column with the lower result fails, then the sample must be evaluated for reanalysis.)

12.1.3. If the Relative percent difference (RPD) between the response on the two columns is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect and the results are qualified. RPD is calculated using the following formula:

$$RPD = \frac{R_1 - R_2}{\frac{1}{2}(R_1 + R_2)}$$

Where R=Result

12.1.4. Multi-response Analytes

For multi-response analytes, the analyst should use the retention time window, but should rely primarily on pattern recognition. The pattern of peaks will normally serve as confirmation.

12.1.5. The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.2. Calibration Range

If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix.

12.3. Dilutions

Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.3.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and only minor matrix peaks are, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgement is required to determine the most concentrated dilution that will not result in instrument contamination.

12.3.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions may be reported at client request, if the lower dilutions will not cause detector saturation, column overload, or carryover. Analyst judgement and client site history will factors in the reporting of dual dilutions.

12.4. Interferences

If peak detection is prevented by interferences, further cleanup should be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.5. Internal Standard Criteria for Continuing Calibration

If internal standard calibration is used, then the internal standard response in a continuing calibration standard must be within 50 to 150% of the response in the mid level of the initial calibration.

12.6. Calculations

Capabilities of individual data systems may require the use of different formulas than those presented here. When this is the case, the calculations used must be shown to be equivalent and must be documented in an appendix attached to this document.

12.6.1. External Standard Calculations

12.6.1.1. Aqueous samples

$$Concentration\ (mg/L) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times V_s)}$$

Where:

A_x = Response for the analyte in the sample
V_i = Volume of extract injected, µL
D_f = Dilution factor
V_t = Volume of total extract, µL
V_s = Volume of sample extracted or purged, mL
CF = Calibration factor, area or height/ng, Section 10.1

12.6.1.2. Non-aqueous Samples

$$Concentration\ (mg/kg) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times W \times D)}$$

Where:

W = Weight of sample extracted or purged, g

$$D = \frac{100 - \%\,Moisture}{100}$$ (D = 1 if wet weight is required)

12.6.2. Internal Standard Calculations

12.6.2.1. Aqueous Samples

$$Concentration\ (mg/L) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times V_s)}$$

Where:

C_{is} = Amount of internal standard added, ng
A_{is} = Response of the internal standard
RF = Response factor for analyte

12.6.2.2. Non-aqueous Samples

$$Concentration\ (mg/kg) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times W \times D)}$$

12.6.3. Surrogate Recovery

Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation:

$$\%\ \text{Recovery} = \frac{Concentration\ (or\ amount)\ found}{Concentration\ (or\ amount)\ spiked} \times 100$$

13. METHOD PERFORMANCE

13.1. Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: QA-005.

13.2. Initial Demonstration

Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in each appendix.

13.2.3. If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3. Training Qualification

The group/team leader has the responsibility to ensure that an analyst who has been properly trained in its use and has the required experience performs this procedure.

14. POLLUTION PREVENTION

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, and Section 8000B

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.

17.2. Modifications from Previous Revision

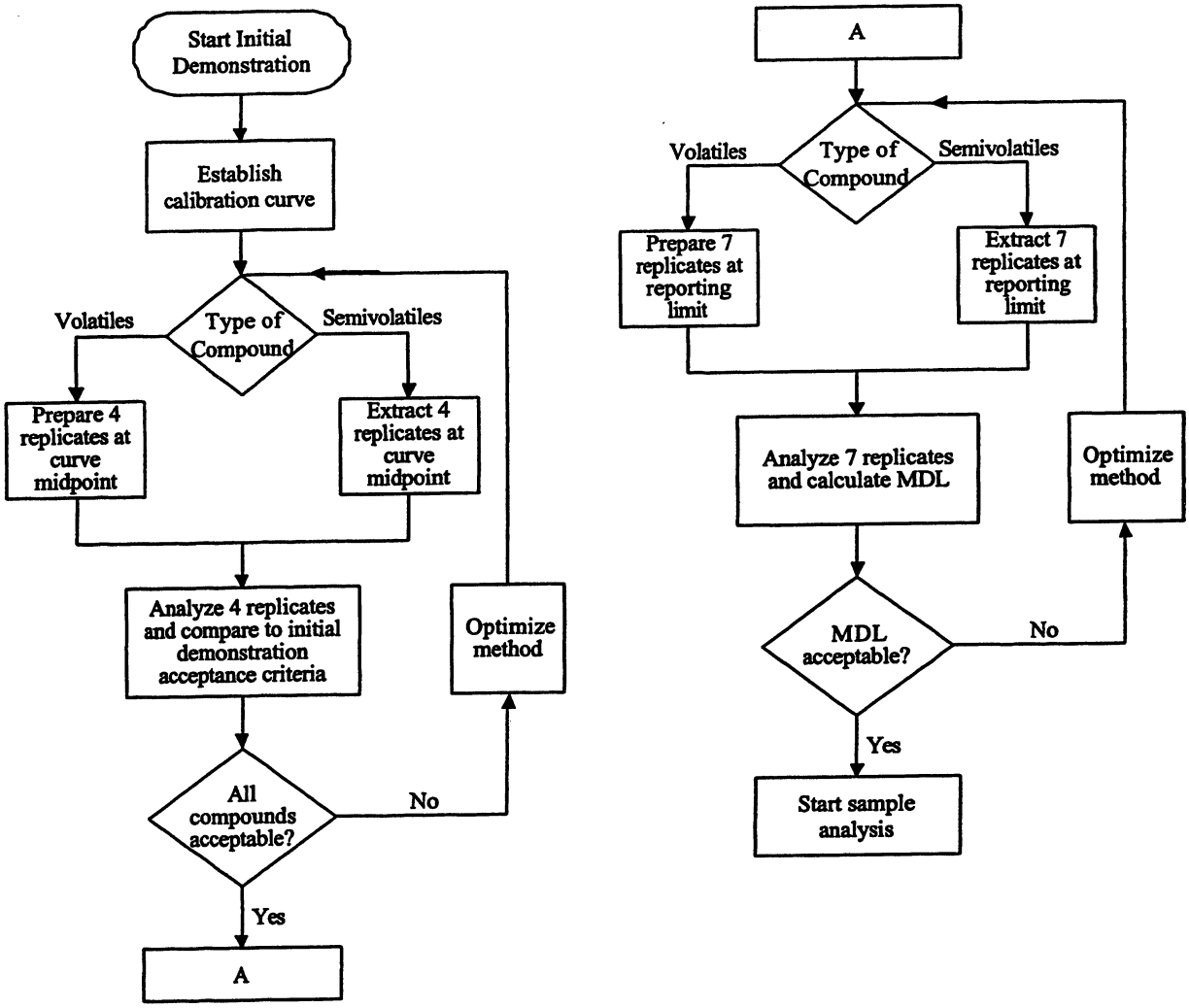
The calibration criteria in section 10.11 have been rewritten to improve consistency with SW-846 and to improve clarity.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

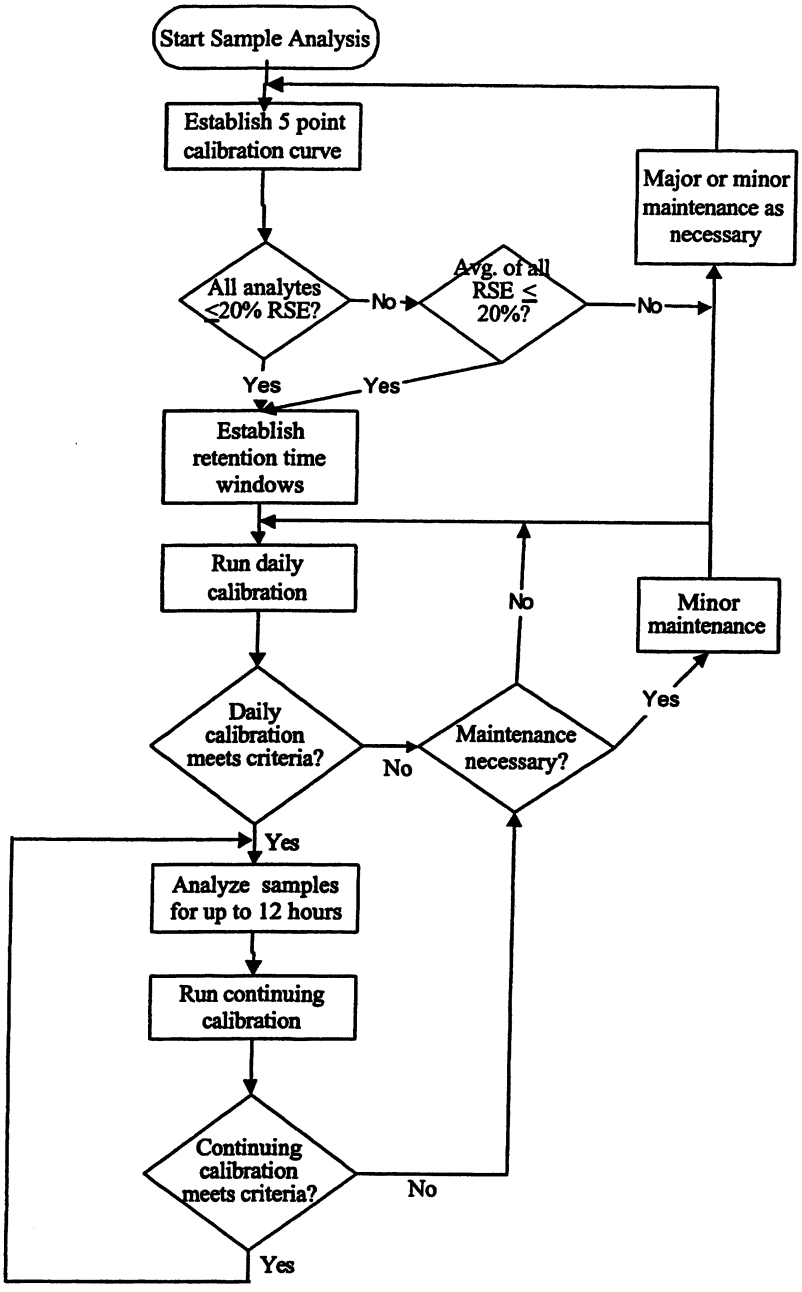
17.4. Flow Diagrams

17.4.1. Initial demonstration and MDL[1]



[1] This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.

17.4.2. Sample Analysis[1]



[1] This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.

1. SCOPE AND APPLICATION

1.1. This method describes sample preparation and extraction for the analysis of volatile organics by a purge and trap procedure, following method 8021B. However, where required by a client QAPP this section may also be used to analyze aromatic volatiles by discontinued methods 8020A and 8010B. All requirements of the 8000B section of this SOP must be met except when superseded by this Appendix. Refer to Table A-1 for the individual analytes normally determined by these procedures.

1.2. Compounds within the scope of this method have boiling points below 200°C and are soluble or slightly soluble in water. Classes of compounds best suited to purge-and-trap analysis include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

1.3. Water samples and soils samples with low levels of contamination may be analyzed directly by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in soil may be determined by the medium level methanol extraction procedure.

1.4. This method also describes the preparation of water-miscible liquids, non-water-miscible liquids, solids, wastes, and soils/sediments for analysis by the purge-and-trap procedure.

1.5. The associated LIMS method code is QR.

2. SUMMARY OF METHOD

2.1. An inert gas is bubbled through the sample at ambient temperature or at 40°C (40°C required for low-level soils), and the volatile components are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and back-flushed with inert gas to desorb the components onto a gas chromatographic column. Analytes are detected using a photoionization Detector, an electrolytic conductivity detector or a combination of both.

2.2. For soil samples, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water. It is then analyzed by purge-and-trap GC following the normal water method. If very low detection limits are needed for soil samples then direct purge using sodium bisulfate preservation may be necessary.

3. DEFINITIONS

Refer to the STL North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this SOP.

4. INTERFERENCES

4.1. Refer to section 4 of the method 8000B part of this SOP for general information on chromatographic interferences.

4.2. Impurities in the purge gas, and from organic compounds out-gassing from the plumbing ahead of the trap, account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

4.3. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank

prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

4.4. Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by an analysis of organic-free reagent water to check for cross-contamination. The trap and other parts of the system are subject to contamination. Therefore, frequent bake-out and purging of the system may be required.

4.5. When utilizing an autosampler system, which has multiple ports for sample analysis, it is likely that only a single stage or port may be contaminated by a highly concentrated sample. If a port is suspect, a water blank should be analyzed to verify lack of contamination. If the water blank and subsequent blanks on that port show contamination consistent with the concentrated sample, further maintenance is required. This may include replacing or cleaning the multi-port valve, transfer lines, etc.

4.6. A holding blank is kept in the sample refrigerator. This is analyzed and replaced every 14 days. If the holding blank does not meet the method blank criteria, the source of contamination must be found and corrected. Evaluation of all samples analyzed in the 14-day period prior to the analysis of the contaminated holding blank is required.

4.7. Acidification of samples may result in hydrolysis of 2-chloroethyl-vinyl ether.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B section of this SOP for general safety requirements.

5.2. Often, purge vessels on purge-and-trap instrumentation are pressurized by the time analysis is completed. Therefore, vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.3. The toxicity or carcinogenicity of each chemical used in this procedure has not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

Methanol -- Flammable and toxic

5.4. The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: Benzene, Carbon Tetrachloride, 1,4-Dichlorobenzene, 1,2-Dichloroethane, Hexachlorobutadiene, 1,1,2,2-Tetrachloroethane, 1,1,2-Trichloroethane, Chloroform, 1,2-Dibromoethane, Tetrachloroethene, Trichloroethene, Vinyl Chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood.

5.5. Methanol shall not be used in a CaptAir hood.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes -- 10µL, 25µL, 100µL, 250µL, 500µL, and 1000µL. These should be equipped with a 20 gauge (0.006" ID) needle. These will be used to measure and dispense methanolic solutions and aqueous samples.

6.2. Gas tight syringes -- 5 mL and 25 mL. Used for measuring sample volumes.

6.3. Purge and Trap Apparatus -- A device capable of extracting volatile compounds, trapping on a sorbent trap, and introducing onto a gas chromatograph.

6.4. Purge and Trap Autosampler -- In order to maintain high sample throughput, an autosampler is highly recommended.

6.5. Trap -- The trap used is dependent on the class of compound to be analyzed. Refer to Table A-2 for suggested traps for specific tests.

6.6. Purge Vessels -- These are dependent on the purge and trap unit/autosampler used. Both disposable culture tubes (needle sparge units) and specially designed vessels with fritted bottoms may be used. Follow the manufacturer's suggestions for configuration.

6.7. Columns - Refer to Table A-2 for details of columns.

6.8. Volumetric flasks, Class A: 5 mL to 250 mL

6.9. pH paper

6.10. Balance capable of weighing to 0.01g for samples.

7. REAGENTS AND SUPPLIES

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.

7.2. Organic Free Water

Organic free water is defined as water in which an interferent is not observed at the reporting limit of the compounds of interest. Suggested methods for generating organic free water include:

- Filtration through a carbon bed.
- Continuously sparging water with helium or nitrogen.
- Use of commercial water purification systems.

Other methods may be used, so long as the requirement that the water show no interference is met. The procedure used should be documented in a lab specific attachment.

7.3. Sodium Bisulfate

7.4. Methanol -- Purge and Trap Grade

7.5. Standards

Refer to tables A-5 and A-6 for details of surrogate, matrix spiking and internal standards. Calibration standard levels are not specified, since they may depend on the sensitivity and linear range of specific detectors. However, the low level standard must be equivalent to the reporting limits specified in Table A-1.

7.5.1. Volatile standards are prepared by injecting a measured volume of the stock standard into a syringe containing the appropriate volume of organic free water. The calibration standard is then loaded into the purge device.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample collection.

8.2. Water samples are normally preserved at pH < 2 with 1:1 hydrochloric acid. If residual chlorine is present, 2 drops of 10% sodium thiosulfate are added.

8.3. Solid samples are field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. At specific client request, unpreserved soil samples may be accepted.

8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take an EnCore sample. (The 5 g or 25 g sampler can be used, depending on client preference). Following shipment back to the lab the soil is preserved in methanol. This is the medium level procedure. If very low detection limits are needed (< 50 µg/kg for most analytes) then it will be necessary to use two additional 5 g EnCore samplers or to use field preservation.

8.5. Sample collection for medium level analysis using EnCore samplers.

8.5.1. Ship one 5 g (or 25 g) EnCore sampler per field sample position.

8.5.2. An additional bottle must be shipped for percent moisture determination.

8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25 g) sample into a tared VOA vial containing 5 mL methanol (25 mL methanol for the 25 g sampler). Obtain the weight of the soil added to the vial and note on the label.

8.5.4. Add the correct amount of surrogate spiking mixture. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.)

8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.) The addition of spike introduces a slight error, (0.4%) which can be neglected, into the calculations.

8.5.6. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (100 µL of spike to 25 mL methanol or 20 µL spike to 5 mL methanol).

8.5.7. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.5.8. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4+2°C until analysis.

8.6. Sample collection for medium level analysis using field methanol preservation

8.6.1. Prepare a VOA vial by adding 5 mL purge and trap grade methanol. (If a 25 g sample is to be used, add 25 mL methanol to the VOA vial).

8.6.2. Seal the bottle and attach a label.

8.6.3. Weigh the bottle to the nearest 0.01g and note the weight on the label.

8.6.4. Ship with appropriate sampling instructions.

8.6.5. Each sample will require an additional bottle with no preservative for percent moisture determination.

8.6.6. At client request, the methanol addition and weighing may also be performed in the field.

8.6.7. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.6.8. Add the correct amount of surrogate spiking mixture. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.)

8.6.9. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.) The addition of spike introduces a slight error, (0.4%) which can be neglected, into the calculations.

8.6.10. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (100 µL of spike to 25 mL methanol or 20 µL spike to 5 mL methanol).

8.6.11. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.6.12. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4+2°C until analysis.

8.7. Low level procedure

8.7.1. If low detection limits are required (typically < 50 µg/kg) sodium bisulfate preservation must be used. However, it is also necessary to take a sample for the medium level (methanol preserved) procedure, in case the concentration of analytes in the soil is above the calibration range of the low-level procedure.

8.7.2. A purge and trap autosampler capable of sampling from a sealed vial is required for analysis of samples collected using this method. (Varian Archon or O.I. 4552).

8.7.3. The soil sample is taken using a 5g EnCore sampling device and returned to the lab. It is recommended that two EnCore samplers be used for each field sample position, to allow for any reruns than may be necessary. A separate sample for % moisture determination is also necessary.

8.7.4. Prepare VOA vials by adding a magnetic stir bar, approximately 1 g of sodium bisulfate and 5 mL of reagent water.

8.7.5. Seal the vial and attach a label. The label must not cover the neck of the vial or the autosampler will malfunction.

8.7.6. Weigh the vial to the nearest 0.01g and note the weight on the label.

8.7.7. Extrude the soil sample from the EnCore sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil and note on the label.

8.7.8. **Note:** Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case at a specific site, add 5 mL of water instead, and freeze at ≥ -10°C until analysis.

8.7.9. Alternatively the sodium bisulfate preservation may be performed in the field. Ship at least two vials per sample. The field samplers must determine the weight of soil sampled. Each sample will require an additional bottle with no preservative for percent moisture determination, and an additional bottle preserved with methanol for the medium level procedure.

8.8. Aqueous samples are stored in glass containers with Teflon lined septa at 4°C +/- 2°C, with minimum headspace.

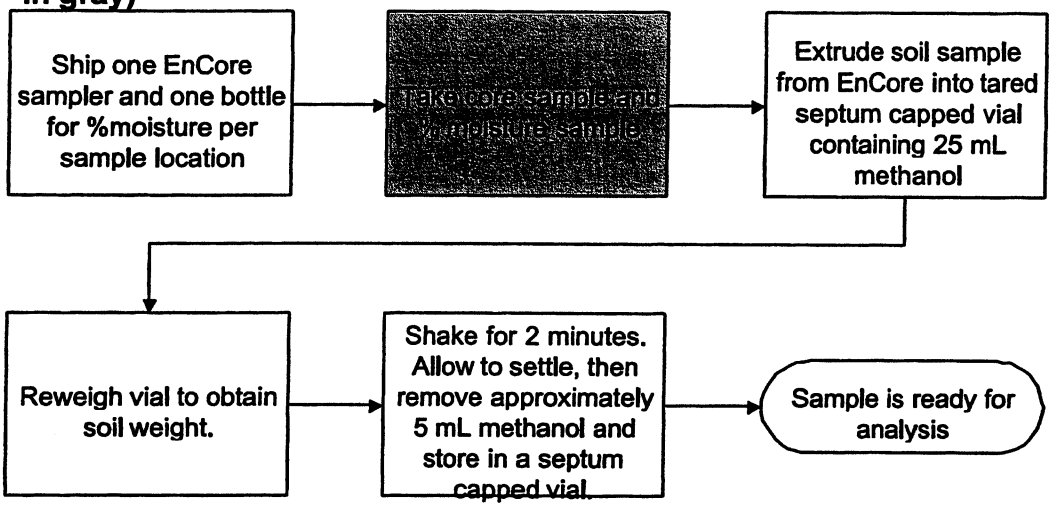
8.9. Medium level solid extracts are aliquoted into 2 - 5 mL glass vials with Teflon lined caps and stored at 4°C +/- 2°C. The extracts are stored with minimum headspace.

8.10. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14 day holding time. However they should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative). Maximum holding time for the EnCore sampler (before the sample is added to methanol or sodium bisulfate) is 48 hours.

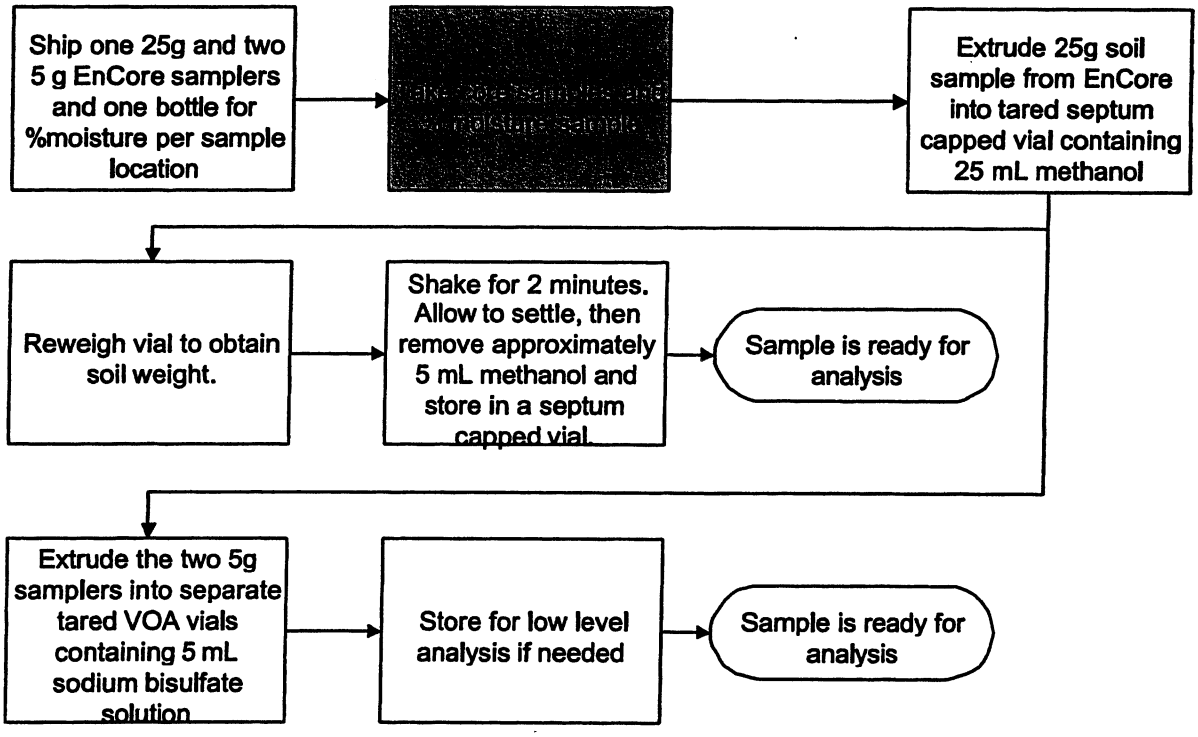
8.11. A holding blank is stored with the samples. This is analyzed and replaced if any of the trip blanks show any contamination. Otherwise it is replaced every 14 days.

Note: Freezing is not allowed for Ohio VAP solids.

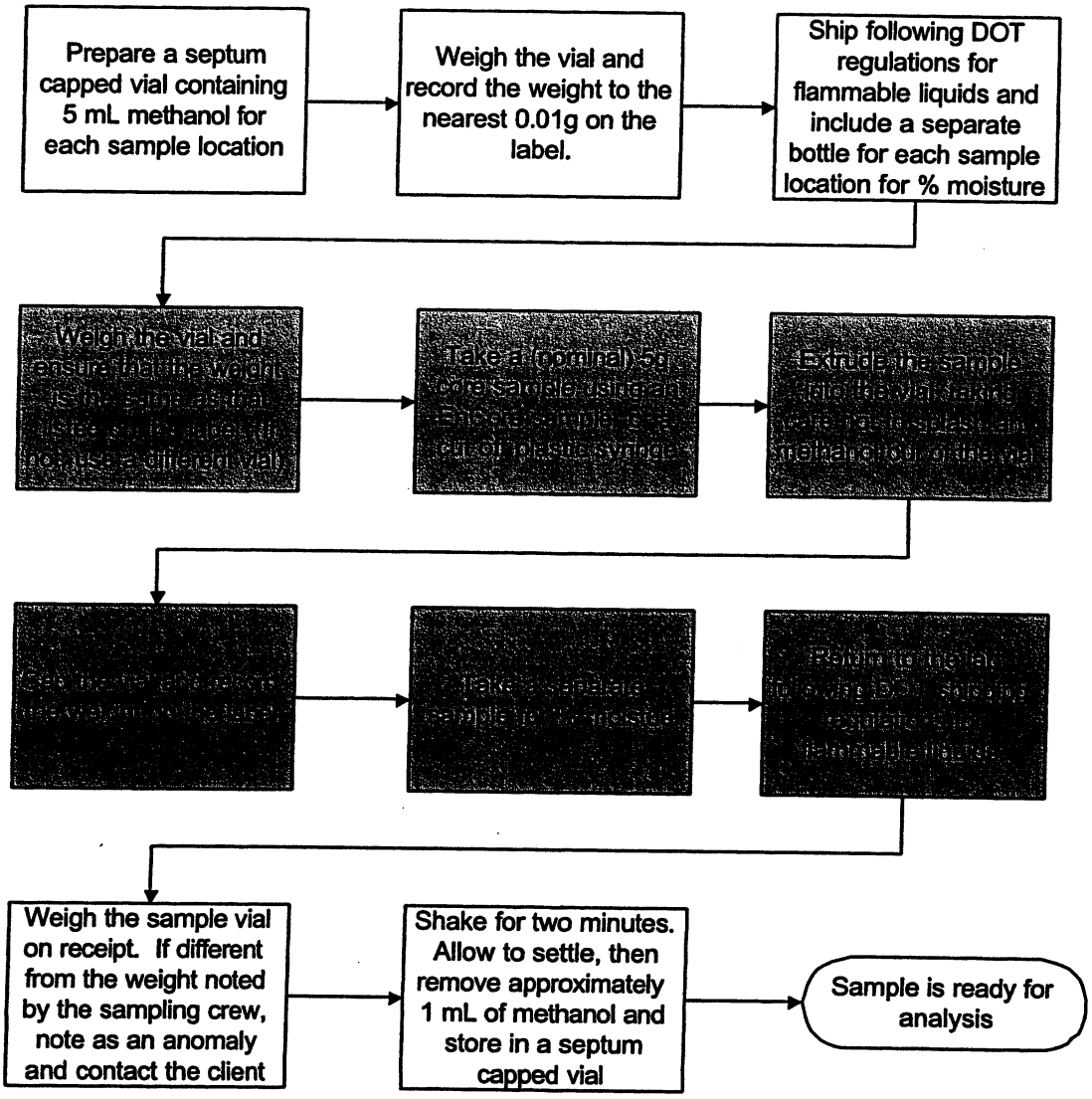
EnCore procedure when low level is not required (field steps in gray)



EnCore procedure when low level is required



Field methanol extraction procedure (field steps in gray)



9. QUALITY CONTROL

9.1. Refer to the method 8000B section of this SOP, section 9, for general quality control procedures, including batch definition, requirements for method blanks, LCS, matrix spikes, surrogates, and control limits.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to the method 8000B section of this SOP, section 10, for general calibration procedures.

10.2. Gas Chromatograph Operating Conditions

Various column configurations are possible. If dual column confirmation is necessary, the sample may be split using a Y splitter at the injector end to direct the sample to two columns and two detectors. For simultaneous determination of aromatic and halogenated volatiles, a single column is used and the PID and ELCD detectors are connected in series.

10.2.1. Refer to Table A-2, A-3 and A-4 for GC operating conditions.

10.3. Initial Calibration

10.3.1. Refer to Section 10 of the 8000B section of this SOP for details of initial calibration criteria.

10.3.2. Low level soil samples must be purged at 40°C; therefore the calibration curve must also be purged at 40°C. In addition, the low level soil calibration solutions should contain approximately the same amount of sodium bisulfate as the samples.

10.3.3. The low level calibration must be at the reporting limit or below. The remaining standards encompass the working range of the detector.

10.3.4. Calibrate the instrument using the same volume that will be used during sample analysis.

10.4. Calibration Verification

10.4.1. A mid level calibration standard is used for the calibration verification. The gases have 20 % D criteria rather than the 15% used for other analytes.

10.4.2. A calibration verification run is performed after every 10 samples for this method.

10.4.3. Bracketing of samples with calibration verification runs is only necessary for external standard analysis.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for general procedural requirements.

11.2. Analytical Sequence

The analytical sequence starts with an initial calibration of at least five points, or a 12 hour calibration that meets % difference criteria from an existing initial calibration.

11.3. Confirmation

The PID and ELCD detectors are sufficiently selective that second column confirmation is not always necessary. Requirements for second column confirmation should be decided in consultation with the

client. If the PID and ELCD are used in series confirmatory information for many analytes can be gained by comparing the relative response from the two detectors.

11.4. Aqueous Sample Analysis (Purge and Trap units using sparge vessels)

11.4.1. Depending on the sensitivity of the instrument and capabilities of the purge and trap device, 5, 10, 20, or 25 mL sample volumes may be analyzed. A 5 mL sample volume is recommended.

11.4.2. Rinse a 5 mL (or 25 mL for larger sample volumes) gas-tight syringe with organic free water. Fill the syringe with the sample to be analyzed, and compress to volume.

11.4.3. Check and document the pH of the sample remaining in the VOA vial after loading the syringe.

11.4.4. This procedure invalidates the contents of the VOA vial for further analysis, unless an aliquot is transferred to a smaller VOA vial with no headspace (e.g., 20 mL) at the same time the analysis aliquot is removed.

11.4.5. Spike with the appropriate volume of surrogate/internal standard solution and spike solution (if required) through the barrel of the syringe. The method blank is spiked with surrogates only, the LCS and matrix spikes with the surrogate and matrix spiking solutions. Refer to Tables A-5 and A-6 for volumes and concentrations of spiking solutions.

11.4.6. Load onto the purge and trap device and start the run.

11.4.7. If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. When a sample has a high response for a compound, an organic free water blank should follow analysis. It is recognized that during automated unattended analysis, this may not occur. If any potential carryover hits are present in samples following highly contaminated samples, the sample must be reanalyzed to determine if any of these hits are a result of carryover or are actually present in the sample.

11.4.8. Dilutions may be made in gas tight syringes unless the volume of sample used is less than 5 µL, in which case dilution in volumetric flasks will be necessary.

11.4.8.1. Spike with the same volume of surrogate/internal standard solution as used for undiluted samples prior to loading onto the purge and trap device.

11.4.8.2. For Matrix spike / matrix spike duplicates where the sample requires dilution, the sample is spiked after the dilution is performed.

11.5. Aqueous and Soil Sample Analysis (Purge and Trap units that sample directly from the VOA vial)

11.5.1. Units, which sample from the VOA vial, should be equipped with a module, which automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

11.5.2. If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise the internal and surrogate standards must be added to the vial. *Note:* Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to analyzed as soils.

11.5.3. Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.5.4. Check the pH of the sample remaining in the VOA vial after analysis is completed.

11.6. Low-Level Solids Analysis using discrete autosamplers

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

This method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogate and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.6.1. Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

11.6.2. Weigh out 5 g (or other appropriate aliquot) of sample into a disposable culture tube or other purge vessel. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 1.0 g. If the sample is contaminated with analytes such that a purge amount less than 1.0 g is appropriate, use the medium level method described in section 11.7.

11.6.3. Connect the purge vessel to the purge and trap device.

11.6.4. Rinse a 5 mL gas-tight syringe with organic free water, and fill. Compress to 5 mL. Add surrogate/internal standard (and matrix spike solutions if required.) (See Tables A-5, A-6, A-7 and A-8.) Add directly to the sample from 11.6.2.

11.6.5. The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

11.6.6. Add the heater jacket or other heating device and start the purge and trap unit.

11.6.7. Soil samples that have low IS recovery when analyzed (<50%) should be reanalyzed once to confirm matrix effect. If external standard calibration is used, samples with surrogate recovery below the control limit should be reanalyzed once to confirm matrix effect.

11.7. Methanol Extract Soils

11.7.1. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe.

12. DATA ANALYSIS AND CALCULATIONS

Refer to section 12 of the 8000B section of this SOP.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the 8000B section of this SOP.

14. POLLUTION PREVENTION

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Sections 5000, 5030B, 5035 and 8021B

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.2. Modifications from previous revision

17.2.1. No revisions were made to this appendix.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4. Tables

Table A-1

Standard Analyte List

Test	Compound	CAS number	Reporting Limit, µg/L or µg/kg		
			Aqueous	Low Soil	Medium Soil
Halogenated volatiles by 8021B	Bromodichloromethane	75-27-4	1.0	1.0	50
	Bromoform	75-25-2	1.0	1.0	50
	Bromomethane	74-83-9	1.0	1.0	50
	Carbon Tetrachloride	56-23-5	1.0	1.0	50
	Chlorobenzene	108-90-7	1.0	1.0	50
	Chloroethane	70-00-3	1.0	1.0	50
	2-Chloroethyl vinyl ether	110-75-8	5.0	5.0	250
	Chloroform	67-66-3	1.0	1.0	50
	Chloromethane	74-87-3	1.0	1.0	50
	Dibromochloromethane	124-48-1	1.0	1.0	50
	1,2-Dichlorobenzene	95-50-1	1.0	1.0	50
	1,3-Dichlorobenzene	541-73-1	1.0	1.0	50
	1,4-Dichlorobenzene	106-46-7	1.0	1.0	50
	Dichlorodifluoromethane	75-71-8	1.0	1.0	50
	1,1-Dichloroethane	75-34-3	1.0	1.0	50
	1,2-Dichloroethane	107-06-2	1.0	1.0	50
	1,1-Dichloroethene	75-45-4	1.0	1.0	50
	cis-1,2 Dichloroethene	156-59-4	1.0	1.0	50
	trans-1,2-Dichloroethene	156-60-5	1.0	1.0	50
	Dichloromethane(DCM)	75-09-2	5.0	5.0	250
	1,2-Dichloropropane	78-87-5	1.0	1.0	50
	cis-1,3-Dichloropropene	10061-01-5	1.0	1.0	50
	trans-1,3-Dichloropropene	10061-02-6	1.0	1.0	50
	1,1,2,2-Tetrachloroethane	79-34-5	1.0	1.0	50
	Tetrachloroethene	127-18-4	1.0	1.0	50
	1,1,1-Trichloroethane	71-55-6	1.0	1.0	50
	1,1,2-Trichloroethane	79-00-5	1.0	1.0	50
	Trichloroethene	79-01-6	1.0	1.0	50
	Trichlorofluoromethane	75-69-4	1.0	1.0	50
	Vinyl Chloride	75-01-4	1.0	1.0	50
Additional halogenated volatiles	Benzyl Chloride	100-44-7	5.0	5.0	250
	Bromobenzene	108-86-1	1.0	1.0	50
	Dibromomethane	74-95-3	1.0	1.0	50
	1,1,1,2-Tetrachloroethane	630-20-6	1.0	1.0	50
	Freon 113	76-13-1	1.0	1.0	50
	1,2,3-Trichloropropane	96-18-4	1.0	1.0	50
BTEX by 8021B	Benzene	71-43-2	1.0	1.0	50
	Ethyl Benzene	100-41-4	1.0	1.0	50
	Toluene	108-88-3	1.0	1.0	50
	Xylenes (total)	1330-20-7	1.0	1.0	50
Aromatic	Benzene	71-43-2	1.0	1.0	50

Table A-1

Standard Analyte List

Test	Compound	CAS number	Reporting Limit, µg/L or µg/kg		
			Aqueous	Low Soil	Medium Soil
volatiles by 8021B					
	Chlorobenzene	108-90-7	1.0	1.0	50
	1,2-Dichlorobenzene	75-34-3	1.0	1.0	50
	1,3-Dichlorobenzene	107-06-2	1.0	1.0	50
	1,4-Dichlorobenzene	75-45-4	1.0	1.0	50
	Ethyl Benzene	100-41-4	1.0	1.0	50
	Toluene	108-88-3	1.0	1.0	50
	Xylenes (total)	1330-20-7	1.0	1.0	50
Additional aromatic and unsaturated volatiles	1,2,4 Trimethylbenzene	95-63-6	1.0	1.0	50
	1,3,5 Trimethylbenzene	108-67-8	1.0	1.0	50
	Acetone	67-64-1	10	10	500
	MEK (2-butanone)	78-93-3	5.0	5.0	250
	MIBK (4-methyl-2-pentanone)	108-10-1	5.0	5.0	250
	Naphthalene	91-20-3	2.0	2.0	250
	Styrene	100-42-5	1.0	1.0	50
	Methyl tert-butyl ether (MTBE)	1634-04-4	1.0	1.0	50
Combined halogenated and aromatic volatiles by 8021B	Benzene	71-43-2	1.0	1.0	50
	Bromobenzene	108-86-1	1.0	1.0	50
	Bromochloromethane	74-97-5	1.0	1.0	50
	Bromodichloromethane	75-27-4	1.0	1.0	50
	Bromoform	75-25-2	1.0	1.0	50
	Bromomethane	74-83-9	1.0	1.0	50
	n-butylbenzene	104-51-8	1.0	1.0	50
	sec-Butylbenzene	135-98-8	1.0	1.0	50
	tert-Butylbenzene	98-06-6	1.0	1.0	50
	Carbon Tetrachloride	56-23-5	1.0	1.0	50
	Chlorobenzene	108-90-7	1.0	1.0	50
	Chlorodibromomethane	124-48-1	1.0	1.0	50
	Chloroethane	75-00-3	1.0	1.0	50
	Chloroform	67-66-3	1.0	1.0	50
	Chloromethane	74-87-3	1.0	1.0	50
	2-Chlorotoluene	95-49-8	1.0	1.0	50
	4-Chlorotoluene	106-43-4	1.0	1.0	50
	1,2-Dibromo-3-Chloropropane(DBCP)	96-12-8	1.0	1.0	50
	1,2-Dibromoethane(EDB)	106-93-4	1.0	1.0	50
	Dibromomethane	74-95-3	1.0	1.0	50
	1,2-Dichlorobenzene	95-50-1	1.0	1.0	50
	1,3-Dichlorobenzene	541-73-1	1.0	1.0	50
	1,4-Dichlorobenzene	106-46-7	1.0	1.0	50
	Dichlorodifluoromethane	75-71-8	1.0	1.0	50
	1,1-Dichloroethane	75-34-3	1.0	1.0	50
	1,2-Dichloroethane	107-06-2	1.0	1.0	50
	1,1-Dichloroethene	75-35-4	1.0	1.0	50

Table A-1
Standard Analyte List

Test	Compound	CAS number	Reporting Limit, µg/L or µg/kg		
			Aqueous	Low Soil	Medium Soil
	cis-1,2-Dichloroethene	156-59-4	1.0	1.0	50
	trans-1,2-Dichloroethene	156-60-5	1.0	1.0	50
	1,2-Dichloropropane	78-87-5	1.0	1.0	50
	1,3-Dichloropropane	142-28-9	1.0	1.0	50
	2,2-Dichloropropane	590-20-7	1.0	1.0	50
	1,1-Dichloropropene	563-58-6	1.0	1.0	50
	cis-1,3-Dichloropropene	10061-01-5	1.0	1.0	50
	trans-1,3-Dichloropropene	10061-02-6	1.0	1.0	50
	Ethylbenzene	100-41-4	1.0	1.0	50
	Hexachlorobutadiene	87-68-3	1.0	1.0	50
	Isopropylbenzene	98-82-8	1.0	1.0	50
	p-Isopropyltoluene	99-87-6	1.0	1.0	50
	Methylene Chloride	75-09-2	5.0	5.0	250
	Naphthalene	91-20-3	2.0	2.0	250
	n-Propylbenzene	10306501	1.0	1.0	50
	Styrene	100-42-5	1.0	1.0	50
	1,1,1,2-Tetrachloroethane	630-20-6	1.0	1.0	50
	1,1,2,2-Tetrachloroethane	79-34-5	1.0	1.0	50
	Tetrachloroethene	127-18-4	1.0	1.0	50
	Toluene	108-88-3	1.0	1.0	50
	1,2,3-Trichlorobenzene	87-61-6	1.0	1.0	50
	1,2,4-Trichlorobenzene	120-82-1	1.0	1.0	50
	1,1,1-Trichloroethane	71-55-6	1.0	1.0	50
	1,1,2-Trichloroethane	79-00-5	1.0	1.0	50
	Trichloroethene	79-01-6	1.0	1.0	50
	Trichlorofluoromethane	75-69-4	1.0	1.0	50
	1,2,3-Trichloropropane	96-18-4	1.0	1.0	50
	1,2,4-Trimethylbenzene	95-63-6	1.0	1.0	50
	1,3,5-Trimethylbenzene	108-67-8	1.0	1.0	50
	Vinyl Chloride	75-01-4	1.0	1.0	50
	Xylenes (total)	1330-20-7	1.0	1.0	50

Table A-2
Recommended Conditions for Aromatic Volatiles

Parameter	Recommended Conditions
Temperature program	50°C, 1min, 10°C/min to 200°C,1min
Column 1	Rtx-502.2 or DB-502.2 60m x 0.53mm 3.0um
Column 2	Rtx-1 or DB-1 60m x 0.53mm 3.0 um
Carrier gas	Helium or hydrogen
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-3 Recommended Conditions for Method Halogenated Volatiles	
Parameter	Recommended Conditions
Temperature program	35°C, 12 min, then 4°C/min to 200°C, hold for 5 min
Column 1	DB-VRX or RTX-502.2 105m x 0.53 mm id df = 3.0um
Column 2	DB-1 or RTX-1 105m x 0.53 mm ID df = 3.0um
Column 3	Rtx - Volatiles 120m x 0.53mm ID df=2.0um
Carrier gas	Helium
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-4 Recommended Conditions for Method Combined Aromatic and Halogenated Volatiles	
Parameter	Recommended Conditions
Temperature program	35°C, 12 min, then 4°C/min to 200°C, hold for 5 min
Column 1	DB-VRX or RTX-502.2 105m x 0.53 mm id df = 3.0um
Column 2	DB-1 or RTX-1 105m x 0.53 mm ID df = 3.0um
Column 3	Rtx - Volatiles 120m x 0.53mm ID df=2.0um
Carrier gas	Helium
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-5
Surrogate and Internal Standard Concentrations

Standard	Components	Working Solution µg/mL	Spike amount µL (for 5 mL purge)	Final concentration µg/L (µg/kg)
Aromatic volatiles IS/SS	4-Chlorotoluene (SS) 1-Chloro-4-fluorobenzene (IS)	20 40	5	20 40
Halogenated volatiles IS/SS	4-chlorotoluene (SS) 1-Chloro-4-fluorobenzene (IS)	20 40	5	20 40
Combined Aromatic and halogenated volatiles IS/SS	Fluorobenzene (SS)	20	5	20
	1,4-Dichlorobutane (SS)	20	5	20
	1-Chloro-4-fluorobenzene (IS)	40	10	40

It may be necessary to select different surrogates in order to minimize sample interferences. 1-chloro-4-fluorobenzene and 4-chlorotoluene are fairly well resolved from analytes listed in this SOP. However 4-chlorotoluene may sometimes be requested as a target analyte. Other surrogates that may be considered, and issues associated with their use are:

Bromochloromethane: Elutes very close to chloroform and cis-1, 2-dichloroethene on the 502.2 column. May be a target analyte.

1,2-Bromochloroethane:

1-Chloro-2-fluorobenzene: Elutes close to ethylbenzene on DB-1 or Rtx-1 and close to m,p-xylene on 502.2

a,a,a-Trifluorotoluene: Good for aromatic volatiles, coelutes or very close to trichloroethene

Bromofluorobenzene: Close to 1,1,2,2-trichloroethane and 1,2,3-trichloropropane on the 502.2 column. Good on DB-1 or Rtx-1.

2-Bromo-1-chloropropane: May coelute with 1,1,2-trichloroethane

Table A-6
Concentrations for LCS and MS/MSD compounds

Standard	Components	Working Solution µg/mL	Spike amount µL (5 mL purge)	Final concentration µg/L (µg/kg)
Aromatic	Benzene	20	5	20
	Toluene	20		20
	Chlorobenzene	20		20
Halogenated	Chlorobenzene	20	5	20
	1,1-Dichloroethene	20		20
	Trichloroethene	20		20
Combination aromatic / halogenated	Benzene	20	5	20
	Toluene	20		20
	Chlorobenzene	20		20
	1,1-Dichloroethene	20		20
	Trichloroethene	20		20

1. SCOPE AND APPLICATION

This SOP Appendix describes procedures to be used when SW-846 Method 8081A is applied to the analysis of organochlorine pesticides by GC/ECD. This Appendix may also to be applied when discontinued SW-846 Method 8080A is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate STL North Canton sample extraction SOPs. (CORP-OP-0001NC) Criteria for the analysis of wastewater by Method 608 is also included in this appendix.

Table B-1 lists compounds, which are routinely determined by this method, and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

At client request, this method may also be used for the analysis of PCBs (Arochlors) in combination with pesticides, although these are normally analyzed following method 8082, as described in Appendix C of this SOP. In any event, if samples for PCB analysis do not need the acid clean up procedure, then the same injection may be used for method 8081B and 8082, assuming all calibration and QC requirements for both methods are met. Extracts that have been acid cleaned may not be analyzed for pesticides, since several of the pesticides will be degraded.

1.1. The associated LIMS method codes are QJ (8081A) and DM (608).

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of organochlorine pesticides. The pesticides are injected onto the column and separated and detected by electron capture detection. Quantitation may be by internal or external standard methods.

3. DEFINITIONS

Refer to the STL North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Avoiding contact with any plastic materials minimizes interferences from phthalates.

4.3. Sulfur will interfere and can be removed using procedures described in SOP CORP-OP-0001NC.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), and Sulfur cleanup (Method 3660). These cleanup procedures are included in SOP # CORP-OP-0001NC. Using hexane / acetone as the extraction solvent (rather than hexane / methylene chloride) will reduce the amount of interferences extracted.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

5.2. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

5.3. The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, and the BHCs. Primary standards of these toxic compounds should be prepared in a hood.

5.4. All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

5.5. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the Director, EH&S shall be immediately notified and a letter sent to the NRC or local state agency.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. A ^{63}Ni electron capture detector is required.

6.2. Refer to Table B-2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.

7.2. Refer to Table B-3 for details of calibration standards.

7.3. Surrogate Standards

Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Refer to tables B-5 and B-6 for details of surrogate standards.

7.4. Column Degradation Evaluation Mix

A mid-level standard containing 4,4'-DDT and Endrin and not containing any of their breakdown products must be prepared for evaluation of degradation of these compounds by the GC column and injection port. This mix must be replaced after one year, or whenever corrective action to columns fails to eliminate the breakdown of the compounds, whichever is shorter. This solution also contains the surrogates. Refer to Table B-4 for details of the column degradation evaluation mix.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

Refer to Section 9 of the 8000B section of this SOP.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. Refer to Table B-2 for details of GC operating conditions. The conditions listed should result in resolution of all analytes listed in Table B-1 on both columns. Closely eluting pairs are DDE and Dieldrin on the Rtx-5 or DB-5 column and Endosulfan II and DDD on the 1701 column.

Note: Method 608 requires a minimum of three concentration levels for initial calibrations. The relative standard deviation, RSD must be less than 10%.

10.3. Column Degradation Evaluation

Before any calibration runs, either initial or 12 hour, The column evaluation mix must be injected before each initial or daily calibration. The degradation of DDT and endrin must be calculated (see equations 9 and 10) and each shown to be less than 15% before calibration can proceed. This is only necessary if the target compound list includes DDT, Endrin, or any of their degradation products.

If the breakdown of DDT and/or endrin exceeds the limits given above, corrective action must be taken. This action may include:

- Replacement of the injection port liner or the glass wool.
- Cutting off a portion of the injection end of a capillary column.
- Replacing the GC column.

10.4. Initial Calibration

Refer to Section 10 of the 8000B section of this SOP for details of calibration procedures.

10.4.1. Refer to Table B-7 for the initial calibration analytical sequence.

10.4.2. The response for each single-peak analyte will be calculated by the procedures described in the general method for GC analysis.

10.4.3. The surrogate calibration curve is calculated from the Individual AB mix. Surrogates in the other calibration standards are used only as retention time markers. If there are resolution problems, then the A and B mixes may be analyzed separately.

10.4.4. For multi-component pesticides:

Single point calibration is used for multi-component pesticides (typically toxaphene and technical chlordane). Two options are possible; the same quantitation option must be used for standards and samples. Refer to section 12.3 for guidance on which option to use.

10.4.5. For multi-component analytes, the mid level standard must be analyzed as part of the initial calibration. This single point calibration is used to quantitate multi-component analytes.

10.4.6. The analyst may include a full 5 point calibration for any of the multi-component analytes with the initial calibration.

10.5. 12 hour Calibration Verification

The 12 hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration. A mid level calibration standard is used for the 12 hour calibration. Refer to the 8000B section of this SOP for acceptance criteria.

Note : Method 608 requires that the working calibration curve must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ± 15%, the test must be repeated using a fresh calibration standard.

10.5.1. At a minimum, the 12 hour calibration includes analysis of the breakdown mix followed by mid level standards of any single and multi-component analytes.

10.5.2. The retention time windows for any analytes included in the 12 hour calibration are updated.

10.6. Continuing Calibration

The AB calibration mix is analyzed as the continuing calibration standard. At a minimum, this is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. If 12 hours elapse analyze the 12 hour standard sequence instead. The continuing calibration standard need not include multi-component analytes. If instrument drift is expected due to sample matrix or other factors, it may be advisable to analyze the continuing calibration standard more frequently.

10.6.1. A mid level calibration standard is used for the continuing calibration.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for general procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP No. CORP-OP-0001NC.

11.3. Cleanup

Cleanup procedures are described in SOP No. CORP-OP-0001NC.

11.4. Suggested gas chromatographic conditions are given in Table B-2.

11.5. Allow extracts to warm to ambient temperature before injection.

11.6. The suggested analytical sequence is given in Table B-7.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. Identification of Multi-component Analytes

Retention time windows are also used for identification of multi-component analytes, but the "fingerprint" produced by major peaks of those compounds in the standard is used in tandem with the retention times to identify the compounds. The ratios of the areas of the major peaks are also taken into consideration. Identification of these compounds may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.3. Quantitation of Multi-component Analytes

Use 3-10 major peaks or total area for quantitation as described in section 10.4.4, initial calibration of multi-component analytes.

12.3.1. If there are no interfering peaks within the envelope of the multi-component analyte, the total area of the standards and samples may be used for quantitation. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area.

12.3.1.1. Multiple peak option

This option is particularly valuable if toxaphene is identified but interferences make quantitation based on total area difficult. Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks. Alternatively, find the response of each of the 3-10 peaks per multi-peak pesticide, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be coeluting with contaminant peaks from the quantitation. (i.e. peaks which are significantly larger than would be expected from the rest of the pattern.)

Chlordane may be quantitated either using the multiple peak option (12.3.1.1) total area option (12.3.1.2.) or by quantitation of the major components, α-chlordane, γ-chlordane and heptachlor.

12.3.1.2. Total area option

The total area of the standards and samples may be used for quantitation of multi-component analytes. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area. This option should not be used if there are significant interference peaks within the multi-component pattern in the samples. The retention time window for total area measurement must contain at least 90% of the area of the analyte.

12.4. Second column confirmation multi-component analytes will only be performed when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence.

12.5. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX). Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

12.6. Calculation of Column Degradation/% Breakdown (%B)

Equation 9

$$DDT\,\%B = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100$$

where:
A_{DDD}, A_{DDE}, and A_{DDT} = the response of the peaks for 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT in the column degradation evaluation mix.

Equation 10

$$Endrin\,\%B = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_{E}} \times 100$$

where:
A_{EK}, A_{EA}, and A_E = the response of endrin ketone, endrin aldehyde, and endrin in the column degradation evaluation mix.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the main body of this SOP. Example performance limits are listed in Table B-8. The spiking level should be equivalent to a mid level calibration.

14. POLLUTION PREVENTION

Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, Update III, December 1996, Method 8081A

16.2. CFR136A, Method 608 – Organochlorine Pesticides and PCBs

17. MISCELLANEOUS

17.1. Modifications from Reference Method

None

17.2. Modifications from Previous Revisions

17.2.1. No revisions were made to this appendix.

17.3. Tables

Table B-1
Standard Analyte list and Reporting Limits

Compound	Reporting Limit, µg/L or µg/kg		
	water	soil	waste
Aldrin	0.05	1.7	50
α-BHC	0.05	1.7	50
β-BHC	0.05	1.7	50
δ-BHC	0.05	1.7	50
γ-BHC (Lindane)	0.05	1.7	50
α-Chlordane	0.05	1.7	50
γ-Chlordane	0.05	1.7	50
Chlordane (technical)	0.5	17	500
4,4'-DDD	0.05	1.7	50
4,4'-DDE	0.05	1.7	50
4,4'-DDT	0.05	1.7	50
Dieldrin	0.05	1.7	50
Endosulfan I	0.05	1.7	50
Endosulfan II	0.05	1.7	50
Endosulfan Sulfate	0.05	1.7	50
Endrin	0.05	1.7	50
Endrin Aldehyde	0.05	1.7	50
Heptachlor	0.05	1.7	50
Heptachlor Epoxide	0.05	1.7	50
Methoxychlor	0.1	3.3	100
Toxaphene	2.0	67	2000
APPENDIX IX ADD ONs			
Diallate	1.0	33	1000
Isodrin	0.1	3.3	100
Chlorobenzilate	0.1	3.3	100
Kepone[1]	1.0	33	1000

[1] Kepone is sometimes requested for analysis by method 8081A. However kepone may produce peaks with broad tails that elute later than the standard by up to a minute (presumably due to hemi-acetal formation). As a result kepone analysis by 8081A is unreliable and not recommended. Analysis by method 8270C is a possible alternative. Note: alpha chlordane, gamma chlordane, and endrin ketone are not required for some projects.

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.		Final Vol.
Ground water	1000 mL	10 mL	
Low-level Soil	30 g		10 mL
High-level soil / waste	1 g		10 mL

Table B-2	
Parameter	**Recommended Conditions**
Injection port temp	220°C
Detector temp	325°C
Temperature program	120°C for 1 min, 8.5°C/min to 285°C, , 6 min hold
Column 1	Rtx-CLPesticides 30m x 0.32mm id, 0.5µm
Column 2	Rtx-35 30m x 0.32 mm id, 0.5µm
Column 3	DB-608, 30m X 0.32 mm, 0.25µm
Injection	2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table B-3						
Calibration Levels ng/mL						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[2]
Individual Mix AB[1]						
Aldrin	5	10	25	50	100	200
g-BHC (Lindane)	5	10	25	50	100	200
Heptachlor	5	10	25	50	100	200
Methoxychlor	10	20	50	100	200	400
Dieldrin	5	10	25	50	100	200
Endosulfan I	5	10	25	50	100	200
Endosulfan II	5	10	25	50	100	200
4,4'-DDT	5	10	25	50	100	200
Endrin Aldehyde	5	10	25	50	100	200
Endrin Ketone	5	10	25	50	100	200
β-BHC	5	10	25	50	100	200
δ-BHC	5	10	25	50	100	200
α-BHC	5	10	25	50	100	200
4,4'-DDD	5	10	25	50	100	200
4,4'-DDE	5	10	25	50	100	200
Endosulfan Sulfate	5	10	25	50	100	200
Endrin	5	10	25	50	100	200
α-Chlordane[3]	5	10	25	50	100	200
γ-Chlordane[3]	5	10	25	50	100	200
Multi-component Standards						
Chlordane (Technical)			250[4]			
Toxaphene			1000[5]			
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl	5	10	25	50	100	200

[1] Standards may be split into an A and B mix if resolution of all compounds on both columns is not obtained.

[2] Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.

[3] Compounds may be used in lieu of running a daily technical Chlordane standard for samples that are non-detect for technical Chlordane.

[4] This standard may be used for quantitation of technical chlordane between 50 and 1000 ng/mL. If the chlordane is more concentrated, the extract must be diluted and reanalyzed.

[5] This standard may be used for quantitation of toxaphene between 200 and 4000 ng/mL. If the toxaphene is more concentrated, the extract must be diluted and reanalyzed.

Table B-4	
Column Degradation Evaluation Mix ng/mL	
Component	Concentration
4,4'-DDT	25
Endrin	25
Tetrachloro-m-xylene (Surrogate)	20
Decachlorobiphenyl (Surrogate)	20

Table B-5			
LCS/Matrix Spike and Surrogate Spike levels µg/L or µg/kg			
	Aqueous	Soil	Waste
gamma BHC (Lindane)	0.20	33.3	200
Aldrin	0.20	33.3	200
Heptachlor	0.20	33.3	200
Dieldrin	0.50	33.3	500
Endrin	0.50	33.3	500
4,4'DDT	0.50	33.3	500
Tetrachloro-m-xylene (Surrogate)	0.20	33.3	200
Decachlorobiphenyl (Surrogate)	0.20	33.3	200

Table B-6		
LCS/Matrix Spike and Surrogate Spike levels for TCLP µg/L or µg/kg		
	Aqueous	Waste
Heptachlor	5	500
Heptachlor epoxide	5	500
Lindane	5	500
Endrin	5	500
Methoxychlor	10	1000

Table B-7
Suggested Analytical Sequence

Initial Calibration

Solvent blank (optional)	
Breakdown Mix	
Individual mix AB	All levels
Technical Chlordane	Level 3[1]
Toxaphene	Level 3[1]
Up to 20 samples unless 12 hours comes first)	
Solvent blank (optional)	
Individual mix AB	Mid level (Continuing calibration)
Samples	
After 12 hours:	
Breakdown mix	
Individual mix AB	
Any other single component analytes	

Any multi-component analytes

[1] A five point curve for any of the multi-component analytes may be included
If Arochlors are included, a 5 point calibration for Arochlor 1016/1260 should be included with the initial calibration and a single point for the other Arochlors. The mid point 1016/1260 mix is included with the daily calibration (every 12 hours).

Note: A solvent blank or primer may be analyzed at any time during the sequence when highly contaminated samples are expected. A solvent blank or primer may not be analyzed as routine immediately prior to standards.

12 hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Individual mix AB, and the breakdown mix must be run before the continuing calibration.

Table B-8		
Example Performance limits, four replicate initial demonstration of capability		
Compound	Initial demonstration, mean recovery limits	Initial demonstration, RSD limits
Aldrin	46-112	21
alpha-BHC	51-122	24
beta-BHC	61-120	32
delta-BHC	49.5-118.5	36
gamma-BHC	57-116	23
Chlordane	44.8-108.6	20
4,4'-DDD	52-126	28
4,4'-DDE	46-120	27.5
4,4'-DDT	54-137	36
Dieldrin	42.5-124.5	38
Endosulfan I	43-141	24.5
Endosulfan II	78-171	61
Endosulfan Sulfate	62-132	27
Endrin	49-126	37
Heptachlor	57-100	20
Heptachlor Epoxide	43.5-131.5	25.4
Toxaphene	44.4-111.2	20

1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8000B is applied to the analysis of polychlorinated biphenyls (PCB) by GC/ECD. This Appendix is to be applied when SW-846 Method 8082 is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate STL sample extraction SOPs. (CORP-OP-0001NC). The PCBs are determined and quantitated as Arochlor mixes. Criteria for the analysis of wastewater by Method 608 is also included in this appendix.

Table C-1 lists compounds, which are routinely determined by this method, and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

Note: SW-846 method 8082 provides incomplete guidance for determination of individual PCB congeners. This SOP does not include directions for congener specific analysis.

1.2. The associated LIMS method codes are QH (8082) and DM (608).

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of PCBs. The PCBs are injected onto the column and separated and detected by electron capture detection. Quantitation is by the external standard method.

3. DEFINITIONS

Refer to the STL North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Avoiding contact with any plastic materials minimizes interferences from phthalates.

4.3. Sulfur will interfere and can be removed using procedures described in SOP CORP-OP-0001NC.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), and Sulfur cleanup (Method 3660). These cleanup procedures are included in SOP # CORP-OP-0001NC.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

5.2. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

5.3. All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

5.4. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the Director, EH&S shall be immediately notified and a letter sent to the NRC or local state agency.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. A ^{63}Ni electron capture detector is required.

6.2. Refer to Table C-2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.

7.2. Refer to Table C-3 for details of calibration standards.

7.3. Surrogate Standards

Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Other surrogates may be used at client request. Refer to Table C-4 for details of surrogate standards.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

Refer to Section 9 of the 8000B section of this SOP.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. Initial Calibration

10.2.1. Refer to Table C-5 for the initial calibration analytical sequence.

10.2.2. The response for each Arochlor will be calculated by the procedures described in the general method for GC analysis, with the following modifications.

10.2.3. A five point calibration of the Arochlor 1016/1260 mix is generated with at least mid level single points for the other Aroclor mixes. The average response factor is used to quantitate Arochlors 1260 and 1016, other Arochlors are quantitated from the mid level single point.

Note: Method 608 requires a minimum of three concentration levels for initial calibrations. The relative standard deviation, RSD must be less than 10%.

10.2.4. The analyst may include a full 5 point calibration for any of the Arochlors with the initial calibration.

10.2.5. The high and low standards for the initial 5 point calibration of 1016 / 1260 define the acceptable quantitation range for the other Arochlors. If any Arochlor is determined above this concentration the extract must be diluted and reanalyzed.

10.2.6. If the analyst knows that a specific Arochlor is of interest for a particular project, that Arochlor may be used for the five point calibration rather than the 1016 / 1260 mix.

10.2.7. The surrogate calibration curve is calculated from the Aroclor 1016/1260 mix. Surrogates in the other calibration standards are used only as retention time markers.

10.2.8. Two options are possible for quantitation of Aroclors. The same quantitation option must be used for standards and samples.

10.2.8.1. Multiple peak option

Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks. Alternatively, find the response of each of the 3-10 peaks per Aroclor, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be coeluting with contaminant peaks from the quantitation. (i.e. peaks which are significantly larger than would be expected from the rest of the pattern.)

10.2.8.2. Total area option

The total area of the standards and samples may be used for quantitation of multi-component analytes. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area. This option should not be used if there are significant interference peaks within the multi-component pattern in the samples. The retention time window for total area measurement must contain at least 90% of the area of the analyte.

10.3. 12 hour Calibration

The 12 hour calibration verification must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration.

10.3.1. At a minimum, the 12 hour calibration includes analysis of the Arochlor 1260 / 1016 mix.

10.3.2. It is adequate to verify calibration with a mixture of Aroclors 1016 and 1260. If a specific Aroclor is expected, it may be included in the daily calibration check.

10.3.3. The retention time windows for any analytes included in the daily calibration and CCVs are updated.

10.3.4. For this method samples must be bracketed with successful calibration verification runs.

10.4. Calibration verification

The Arochlor 1260/1016 calibration mix is analyzed as the calibration verification standard. This is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. (Depending on the type of samples, it may be advisable to analyze verifications more frequently in order to minimize reruns.).

10.4.1. A mid level standard is used for the calibration verification.

Note : Method 608 requires that the working calibration curve must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ± 15%, the test must be repeated using a fresh calibration standard.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for general procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP No. CORP-OP-0001NC.

11.3. Cleanup

Cleanup procedures are described in SOP No. CORP-OP-0001NC.

11.4. Suggested gas chromatographic conditions are given in Table C-2.

11.5. Allow extracts to warm to ambient temperature before injection.

11.6. The suggested analytical sequence is given in Table C-5.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Identification of Arochlors

Retention time windows are used for identification of Arochlors, but the "fingerprint" produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

A clearly identifiable Arochlor pattern serves as confirmation of single column GC analysis. Dual column confirmation may be used for specific program requirements or by client request.

12.2. Quantitation of Arochlors

Use 3-10 major peaks or total area for quantitation

If the analyst believes that a combination of Aroclor 1254 and 1260, or a combination of 1242, 1248 and 1232 is present, then only the predominant Arochlor is quantitated and reported, but the suspicion of multiple Aroclors is discussed in the narrative. If well separated Aroclor patterns are present, and then both Aroclors are quantitated and reported.

12.3. If there are no interfering peaks within the envelope of the Arochlor, the total area of the standards and samples may be used for quantitation. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area.

12.4. Second column confirmation of Aroclors will only be performed when requested by the client. The appearance of the multiple peaks in the sample usually serves as a confirmation of Aroclor presence.

12.5. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX). Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

13.2. Method detection limits (MDL) are determined for all Arochlors.

14. POLLUTION PREVENTION

Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, Update III, December 1996, Method 8082

16.2. CFR136A, Method 608, Organochlorine Pesticides and PCBs.

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Method 8082 includes limited direction for congener specific quantitation. This is outside the scope of this SOP.

17.2. Modifications from Previous Revisions

No changes were made to this Appendix

17.3. Tables

Table C-1
Standard Analyte list and Reporting Limits

Compound	Reporting Limit, µg/L or µg/kg		
	water	soil	waste
Aroclor-1016	1.0	33	1000
Aroclor-1221	1.0	33	1000
Aroclor-1232	1.0	33	1000
Aroclor 1242	1.0	33	1000
Aroclor-1248	1.0	33	1000
Aroclor-1254	1.0	33	1000
Aroclor-1260	1.0	33	1000

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.
Ground water	1000 mL	10 mL
Low-level Soil	30 g	10 mL
High-level soil / waste	1 g	10 mL

Table C-2

Parameter	Recommended Conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	70°C for 0.5min, 30°C/min to 190°C, 2.5°C/min to 225, 18°C/min to 280°C, 3 min hold
Column 1	DB-5 or Rtx-5 30m x 0.32mm id, 0.5µm
Column 2	DB-1701 or Rtx 1701 30m x 0.32 mm id, 0.25µm
Column 3	DB-608, 30m X 0.32 mm, 0.25µm
Injection	1-2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table C-3						
Calibration Levels ng/mL						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[1]
Aroclor 1016/1260	100	200	500	1000	2000	4000
Aroclor 1242[2]			500			
Aroclor 1221 +1254[2]			500			
Aroclor 1232[2]			500			
Aroclor 1248[2]			500			
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl	5	10	25	50	100	200

[1] Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.

[2] Aroclors may be quantitated within the range 100 to 2000 ng/mL (4000ng/mL if the level 6 1016/1260 standard is included). If the Aroclor is more concentrated, it must be reanalyzed at a dilution.

Table C-4			
LCS/Matrix Spike and Surrogate Spike levels for Aroclor analysis with Acid Cleanup µg/L or µg/kg			
	Aqueous	Soil	Waste
Aroclor 1016/1260	10	333	10,000
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	200

Table C-5 Michigan Analyte List and Reporting Limits[1]		
	Reporting Limit	
Compound	water (µg/L)	soil (µg/Kg)
Aroclor-1016	0.2	330
Aroclor-1221	0.2	330
Aroclor-1232	0.4	330
Aroclor 1242	0.2	330
Aroclor-1248	0.2	330
Aroclor-1254	0.2	330
Aroclor-1260	0.2	330

[1] Reporting Limits are only for samples performed under the Michigan program

Table C-5
Suggested Analytical Sequence

Initial Calibration

Injection #		
1	Solvent blank (optional)	
2	Aroclor 1016/1260	Level 1
3	Aroclor 1016/1260	Level 2
4	Aroclor 1016/1260	Level 3
5	Aroclor 1016/1260	Level 4
6	Aroclor 1016/1260	Level 5
7	Aroclor 1232	Level 3
8	Aroclor 1242	Level 3
9	Aroclor 1248	Level 3
10	Aroclor 1221/1254	Level 3
11-30	Sample 1-20 (or as many samples as can be analyzed in 12 hours Solvent blank (optional)	
32	Aroclor 1016/1260	Level 3

etc

Note: A solvent blank or primer may be analyzed at any time during the sequence when highly contaminated samples are expected. A solvent blank or primer may not be analyzed as routine immediately prior to standards.

12 hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Aroclor 1260 / 1016 mix. Mid level standards of any other Aroclors expected to be present in the samples are also injected.

1. SCOPE AND APPLICATION

This method is applicable to the gas chromatographic determination of Chlorinated phenoxy acid herbicides in extracts prepared by SOP CORP-OP-0001NC. The herbicides listed in Table D1 are routinely analyzed. Other chlorinated acids may be analyzed by this method if the quality control criteria in Section 9 and the initial demonstration of method performance in Section 13 are met.

1.1. The associated LIMS method code is QS.

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of phenoxy acid herbicides by gas chromatography. The herbicides, as their methyl esters, are injected onto the column, separated, and detected by electron capture detectors. Quantitation is by the external standard method.

3. DEFINITIONS

Refer to the STL North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

4.2. Chlorinated acids and phenols cause the most direct interference with this method.

4.3. Sulfur may interfere and may be removed by the procedure described in SOP#CORP-OP-0001NC.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. A Ni_{63} electron capture detector is required.

6.2. Refer to Table D2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to section 7 of the 8000B section of this SOP for general information on reagents and standards.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).

9.2. Refer to Table D-3 for the components and levels of the LCS and MS mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. Calibration standards are prepared from purchased standards in the methyl ester form.

10.3. The low level standard must be at or below the laboratory reporting limit. Other standards are chosen to bracket the expected range of concentrations found in samples, without saturating the detector or leading to excessive carryover.

10.4. Refer to Table D-2, for details of GC operating conditions.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP #CORP-OP-0001NC.

11.3. Cleanup

The alkaline hydrolysis and subsequent extraction of the basic solution described in the extraction procedure provides an effective cleanup.

11.4. Analytical Sequence

The analytical sequence starts with an initial calibration of at least five points, or a daily calibration that meets % difference criteria from an existing initial calibration.

11.4.1. The daily calibration must be analyzed at least once every 24 hours when samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 24 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a daily calibration.

11.4.2. The daily calibration consists of mid level standards of all analytes of interest. Retention time windows must be updated with the daily calibration.

11.4.3. After every 12 hours a continuing calibration is analyzed. The continuing calibration consists of mid level standards of all analytes of interest. Retention time windows are updated with continuing calibrations.

11.5. Gas Chromatography

Chromatographic conditions are listed in Table D-2.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. The herbicides are analyzed as their methyl esters, but reported as the free acid. For this reason it is necessary to correct the results for the molecular weight of the ester versus the free acid. This is achieved through the concentrations of the calibration standards. For example the 20µg/L calibration standard for 2,4-D contains 21.3 µg/L of the methyl ester. No further correction is necessary.

12.3. A routine 10X dilution occurs on final extracts for all samples. Due to a QuantIMS limitation, the dilution factor field in QuantIMS cannot be used when a dilution is routine, because the dilution factor is automatically applied to all reference values creating reporting problems. For the herbicide analysis, the extract volume will be 10mL and an aliquot at 10X dilution will be analyzed. The final extract volume recorded on the laboratory bench sheet will be recorded as 100mL to avoid using the dilution factor field in QuantIMS.

13. METHOD PERFORMANCE

13.1. The EPA for this method has not published multiple laboratory performance data. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

14. POLLUTION PREVENTION

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

Method 8151A, SW-846, Update III, December 1996

17. MISCELLANEOUS

17.1. Modifications from Reference Method

Refer to the method 8000B section of this SOP for modifications from the reference method.

17.2. Modifications from Previous Revision

The calibration procedure has been changed to require esterification of the calibration standards

17.3. Tables

Table D-1				
Standard Analyte list				
Compound	CAS Number	Reporting Limit, µg/L or µg/kg		
		Aqueous	Soil	Waste
2,4-D	94-75-7	4	80	4000
2,4-DB	94-82-6	4	80	4000
2,4,5-TP (Silvex)	93-72-1	1	20	1000
2,4,5-T	93-76-5	1	20	1000
Dalapon	75-99-0	2	40	2000
Dicamba	1918-00-9	2	40	2000
Dichloroprop	120-36-5	4	80	4000
Dinoseb	88-85-7	0.6	12	600
MCPA	94-74-6	400	8000	400,000
MCPP	93-65-2	400	8000	400,000

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.	Dilution Factor
Ground water	1000 mL 10 mL	20	
Low-level Soil without GPC	50 g	10 mL	20
High-level soil / waste	1 g	10 mL	20

Specific reporting limits are highly matrix dependent. The reporting limits listed above are provided for guidance only and may not always be achievable. For special projects, the extracts may be analyzed without any dilution, resulting in reporting limits 20 times lower than those in Table D-1.

Table D-2	
Instrumental Conditions	
PARAMETER	Recommended conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	80,2/30/170,0/1/180,1
Column 1	DB-5MS or RTX 5 30x0.32, 0.5um
Column 2	DB-1701 or Rtx-1701
Injection	1-2µL
Carrier gas	Helium / Hydrogen
Make up gas	Nitrogen

Recommended conditions should result in resolution of all analytes listed in Table D-1.

The reporting limits listed in Table D-1 will be achieved with these calibration levels and a 20 fold dilution of the sample extract. Lower reporting limits can be achieved with lesser dilutions of the sample extract.

Table D-3			
LCS/Matrix Spike and Surrogate Spike levels µg/L or µg/kg[1]			
	Aqueous	Soil	Waste
2,4-D	16	800	16000
Silvex	4	200	4000
2,4,5-T	4	200	4000
2,4-DB	16	800	16000
Dalapon	8	400	8000
DCAA (surrogate)	16	800	16000

[1] LCS, MS and SS spikes are as the free acid.

1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8000B is applied to the analysis of polynuclear aromatic hydrocarbons (PAH) by High Performance Liquid Chromatography. This Appendix is to be applied when SW-846 Method 8310 or EPA Method 610 is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate STL North Canton sample extraction SOPs. (CORP-OP-0001NC).

Table E-1 lists compounds, which are routinely determined by this method, and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed. Note: Reporting limits are subject to change due to compound sensitivity, calibration levels, MDLs, and other factors.

1.2. The associated LIMS method codes are SG (8310) and VT (610).

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of polynuclear aromatic hydrocarbons by high performance liquid chromatography. The compounds are injected onto a column, separated, and detected by ultraviolet and fluorescence detectors. Quantitation is by the external standard method.

3. DEFINITIONS

Refer to the STL North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

5.2. The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: Benzo(a)anthracene, Benzo(a)pyrene, and Dibenzo(a,h)anthracene. Primary standards of these toxic compounds should be prepared in a hood.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. An Ultraviolet detector and Fluorescence detector is required.

6.2. Refer to Table E2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to section 7 of the 8000B section of this SOP for general information on reagents and standards.

7.2. Refer to Table E-3 and E-4 for details of calibration and other standards.

7.3. Surrogate Standards

Terphenyl-d14 and Benzo(e)pyrene are the surrogate standards. Refer to table E-3 for details of surrogate standards.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

Refer to section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).

9.2. Refer to Table E-3 for the components and levels of the LCS and MS mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

Note: The Initial Calibration requirement for Method 610 is as follows: If the ratio of response to amount injected (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

10.2. Calibration standards are prepared from purchased solutions. Table E-4 lists the calibration levels.

10.3. The low level standard must be at or below the laboratory reporting limit. Other standards are chosen to bracket the expected range of concentrations found in samples, without saturating the detector or leading to excessive carryover.

10.4. Refer to Table E-2, for details of LC operating conditions.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP #CORP-OP-0001NC.

11.3. Cleanup

11.4. Analytical Sequence

The analytical sequence starts with an initial calibration of at least five points, or a daily calibration that meets % difference criteria from an existing initial calibration.

Note: Method 610 requires that continuing calibration verifications may not vary from the predicted response for any parameter by more than ± 15%. The average response may not be used for Method 610.

11.4.1. The daily calibration must be analyzed at least once every 24 hours when samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 24 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a daily calibration.

11.4.2. The daily calibration consists of mid level standards of all analytes of interest. Retention time windows must be updated with the daily calibration.

11.4.3. After every 12 hours a continuing calibration is analyzed. The continuing calibration consists of mid level standards of all analytes of interest. Retention time windows are updated with continuing calibrations.

11.5. High Performance Liquid Chromatography

Chromatographic conditions are listed in Table E-2.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. Surrogate recovery results are calculated and reported for Terphenyl-d14 and Benzo(e)pyrene unless it is determined that sample interference has adversely affected the quantitation of one of the surrogates. One surrogate must be within QC criteria. Corrective action is only necessary if Terphenyl-d14 and Benzo(e)pyrene are both outside of acceptance limits.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required as referenced under Section 13.1

14. POLLUTION PREVENTION

Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, Update III, December 1996, Method 8310.

16.2. CFR136 Appendix A Method 610-Polynuclear Aromatic Hydrocarbons.

17. MISCELLANEOUS

17.1. Reporting limits

17.1.1. The lower standard reporting limits are listed in Table

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Elution time

17.2.1. For any positive detection in a sample, the chromatogram is overlaid on screen with the nearest standard for elution time matching and pattern recognition using the Target system.

17.3. Analyte detection

17.3.1. Some compounds are detected only on the ultraviolet detector. For other compounds, the lower of the concentrations calculated from both detectors is reported, the second detector serving as confirmation of the analytes presence.

17.3.2. For any compound that is detected only on the ultraviolet detector, confirmation analysis is not possible. These detected compounds must be narrated that the quantitation is not confirmed by a second detector (Fluorescence).

17.3.3. Some compounds, due to calibration levels and detector limitations, can only be confirmed using the Fluorescence detector. The result will always be reported from the UV detector. These compounds will be narrated, when applicable. Table E-5 lists the compounds and respective detectors.

17.4. Troubleshooting guide

17.4.1. Consult the instrument manufacturer's operating manual for guidance.

17.5. Tables

Table E-1
Standard Analyte list

Compound	CAS Number	Reporting Limit, µg/L or µg/kg	
		Aqueous	Soil
Acenaphthene	83-32-9	1	100
Acenaphthylene	208-96-8	1	100
Anthracene	120-12-7	2	100
Benzo(a)anthracene	56-55-3	0.1	5
Benzo(a)pyrene	50-32-8	0.1	5
Benzo(b)fluoranthene	205-99-2	0.1	5
Benzo(g,h,i)perylene	191-24-2	0.1	10
Benzo(k)fluoranthene	207-08-9	0.1	1.7
Chrysene	218-01-9	0.1	5
Dibenzo(a,h)anthracene	53-70-3	0.1	5
Fluoranthene	206-44-0	0.1	10
Fluorene	86-73-7	1	100
Indeno(1,2,3-cd)pyrene	193-39-5	0.1	5
Naphthalene	91-20-3	2	100
Phenanthrene	85-01-8	1	100
Pyrene	129-00-0	0.1	5
1-Methylnaphthalene*	90-12-0	2	100
2-Methylnaphthalene*	91-57-6	2	100

Table E-2
Instrumental Conditions

PARAMETER	Recommended conditions			
Injection volume	20 uL			
Detector	HP Series 1100 Fluorescence Detector			
Detector	HP Series 1100 Variable Wavelength Detector (UV)			
Solvent Program	Time	% Water	% Acetonitrile	Flow
	0 min.	50	50	1.5 mL/m
	5 min.	45	55	1.5 mL/m
	10 min.	35	65	2.0 mL/m
	15 min.	25	75	2.0 mL/m

	20 min. 25 min. 30 min.	15 5 45	85 95 55	2.0 mL/m 2.0 mL/m 2.0 mL/m
Column 1	PAH Hypersil 5 micron			
Injection	20 uL			
Solvent	Acetonitrile, HPLC grade			
Solvent	Reagent Water, HPLC grade			

Table E-3 LCS/Matrix Spike and Surrogate Spike Compounds

Compound	Concentration	Compound	Concentration
Acenaphthene	10 ug/mL	Dibenzo(a,h)anthracene	2 ug/mL
Acenaphthylene	10 ug/mL	**Fluoranthene**	2 ug/mL
Anthracene	10 ug/mL	Fluorene	2 ug/mL
Benzo(a)anthracene	2 ug/mL	**Indeno(1,2,3-cd)pyrene**	2 ug/mL
Benzo(a)pyrene	2 ug/mL	**Naphthalene**	10 ug/mL
Benzo(b)fluoranthene	2 ug/mL	**Phenanthrene**	2 ug/mL
Benzo(g,h,i)perylene	2 ug/mL	Pyrene	2 ug/mL
Benzo(k)fluoranthene	2 ug/mL	Terphenyl-d14*	1 ug/mL
Chrysene	2 ug/mL	Benzo(e)pyrene*	5 ug/mL

Bold print denotes control compounds in the LCS/LCSD and MS/MSD.

* Denotes surrogate compounds

Table E-4 Calibration Standards

Component	ST	1:2	1:5	1:10	1:20	1:50	1:100
Naphthalene	50	25	10	5	2.5	1	0.5
Acenaphthylene	50	25	10	5	2.5	1	0.5
Terphenyl d14 (surrogate)	50	25	10	5	2.5	1	0.5
Acenaphthene	10	5	2	1	0.5	0.2	0.1
Fluorene	10	5	2	1	0.5	0.2	0.1
Phenanthrene	10	5	2	1	0.5	0.2	0.1
Anthracene	10	5	2	1	0.5	0.2	0.1
Chrysene	10	5	2	1	0.5	0.2	0.1
1-Methylnaphthalene	50	25	10	5	2.5	1	0.5
Fluoranthene	10	5	2	1	0.5	0.2	0.1
Pyrene	10	5	2	1	0.5	0.2	0.1
Benzo(a)anthracene	10	5	2	1	0.5	0.2	0.1
Benzo(e)pyrene (surrogate)	50	25	10	5	2.5	1	0.5
Benzo(b)fluoranthene	10	5	2	1	0.5	0.2	0.1
Benzo(k)fluoranthene	10	5	2	1	0.5	0.2	0.1

Table E-4 Calibration Standards

Component	ST	1:2	1:5	1:10	1:20	1:50	1:100
Benzo(a)pyrene	10	5	2	1	0.5	0.2	0.1
Dibenzo(a,h)anthracene	10	5	2	1	0.5	0.2	0.1
Benzo(g,h,i)perylene	10	5	2	1	0.5	0.2	0.1
Indeno(1,2,3-cd)pyrene	10	5	2	1	0.5	0.2	0.1
2-Methylnaphthalene	50	25	10	5	2.5	1	0.5

Table E-5 Compound/Detector

Compound	Ultraviolet 254 nm	Fluorescence 280 nm
Acenaphthene	X	X
Acenaphthylene	X	
Anthracene	X	Retention Time confirmation only
Benzo(a)anthracene	X	X
Benzo(a)pyrene	X	X
Benzo(b)fluoranthene	X	X
Benzo(g,h,i)perylene	X	X
Benzo(k)fluoranthene	X	X
Chrysene	X	X
Dibenzo(a,h)anthracene	X	X
Fluoranthene	X	X
Fluorene	X	X
Indeno(1,2,3-cd)pyrene	X	X
Naphthalene	X	X
Phenanthrene	X	X
Pyrene	X	X
1-Methylnaphthalene	X	X
2-Methylnaphthalene	X	X

1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of the concentration of certain Organophosphorus Pesticides in waters, wastewaters, oils, soils, and sludges. It is based on SW846 Method 8141A. Table F1 shows reporting limits for compounds routinely analyzed by this method. The compounds include, but are not limited to, those shown in Table F1.

1.2. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

1.3. The associated LIMS method code is P2.

2. SUMMARY OF METHOD

2.1. An aliquot of prepared sample is injected in a gas chromatograph (GC) and compounds in the effluent are detected by a flame photometric detector. Appropriate preparation techniques are described in SOP CORP-OP-0001NC. Ultrasonic Extraction (Method 3550) is **NOT** an appropriate sample preparation for Method 8141 and should not be used because of the potential for destruction of target analytes during the ultrasonic extraction process.

3. DEFINITIONS

Refer to the STL North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

4.2. Analytical difficulties encountered for target analysis include:

4.2.1. The water solubility of Dichlorvos (DDVP) is 10 g/L at 20°C, and recovery is poor from aqueous solution.

4.2.2. Naled is converted to Dichlorvos (DDVP) on column by debromination. This reaction may also occur during sample workup.

4.2.3. Trichlorfon rearranges and is dehydrochlorinated in acidic, neutral, or basic media to form Dichlorvos (DDVP) and hydrochloric acid. If this method is to be used for the determination of organophosphates in the presence of Trichlorfon, the analyst should be aware of the possibility of rearrangement to Dichlorvos to prevent misidentification.

4.2.4. Merphos is a single component pesticide that is readily oxidized to Merphos oxone. Chromatographic analysis of Merphos almost always results in two peaks.

5. SAFETY

5.1. Refer to Section 5 of the Method 8000B SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP.

6.2. Refer to Table F2 for Instrument settings.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to Section 7 of the 8000B section of this SOP for general information on reagents and standards.

7.2. Refer to Table F-3 for details of calibration and other standards.

7.3. Surrogate Standards

Triphenyl phosphate is the surrogate standard. Refer to table F-4 for details of the surrogate standard.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

Refer to section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).

9.2. Refer to Table F-4 for the components and levels of the LCS and MS mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. Calibration standards are made up from purchased solutions. Table F-3 lists the calibration levels.

10.3. The low level standard must be at or below the laboratory reporting limit. Other standards are chosen to bracket the expected range of concentrations found in samples, without saturating the detector or leading to excessive carryover.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP #CORP-OP-0001NC.

11.3. Cleanup

11.4. Analytical Sequence

The analytical sequence starts with an initial calibration of at least five points, or a daily calibration that meets % difference criteria from an existing initial calibration.

11.4.1. The daily calibration must be analyzed at least once every 24 hours when samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 24 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a daily calibration.

11.4.2. The daily calibration consists of mid level standards of all analytes of interest. Retention time windows must be updated with the daily calibration.

11.4.3. After every 12 hours a continuing calibration is analyzed. The continuing calibration consists of mid level standards of all analytes of interest. Retention time windows are updated with continuing calibrations.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. Surrogate recovery results are calculated and reported for Triphenylphosphate unless it is determined that sample interference has adversely affected the quantitation of one of the surrogates. The surrogate must be within QC criteria. Corrective action is only necessary if Triphenylphosphate is outside of acceptance limits.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

14. POLLUTION PREVENTION

Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

SW846, Update III, December 1996, Method 8141A.

17. MISCELLANEOUS

17.1. Reporting limits

17.1.1. The lower standard reporting limits are listed in Table F-1

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.1.2.1. The nature of the FPD detector contributes to high dilutions for Method 8141A. There is a phenomenon known as quenching that occurs. This happens when light absorption occurs in the flame of the FPD due to hydrocarbons, sulfur, and certain light absorbing compounds.

When this happens the analytes of interest do not reach the photomultiplier tube and are not detected even though they may be present.

17.2. Elution time

17.2.1. For any positive detection in a sample, the chromatogram is overlaid on screen with the nearest standard for elution time matching and pattern recognition using the Target system.

17.3. Troubleshooting guide

17.3.1. Consult the instrument manufacturer's operating manual for guidance.

Table F1: Organophosphorus Pesticides Routinely Analyzed and Reporting Limits

Compound	CAS Number	Reporting Limits	
		Water, µg/L	Solid, µg/kg
Azinphos methyl	86-50-0	1.0	33
Bolstar (Suprofos)	35400-43-2	1.0	33
Chlorpyrifos	2921-88-2	1.0	33
Coumaphos	56-72-4	1..0	33
Demeton, O and S	8065-48-3	1.0	33
Diazinon	333-41-5	1.0	33
Dichlorvos	62-73-7	1.0	33
Disulfoton	298-04-4	1.0	33
Ethoprop	13194-48-4	1.0	33
Fensulfothion	115-90-2	1.0	33
Fenthion	55-38-9	1.0	33
Malathion	121-75-5	1.0	33
Merphos	150-50-5	1.0	33
Methyl Parathion	298-00-0	1.0	33
Mevinphos	7786-34-7	1.0	33
Naled	300-76-5	1.0	33
Phorate	298-02-2	1.0	33
Ronnel	299-84-3	1.0	33
Stirophos	22248-79-9	1.0	33
Tokuthion	34643-46-4	1.0	33
Trichloronate	327-98-0	1.0	33
O,O,O-Trientyl phosphorothioate	126-68-1	1.0	33
Thionazin	297-97-2	1.0	33
Sulfotepp	3689-24-5	1.0	33
Dimethoate	60-51-5	1.0	33
Parathion	56-38-2	1.0	33
Famphur	52-85-7	1.0	33

Table F2: Instrumental Conditions	
PARAMETER	Recommended conditions
Injection port temp	175°C
Detector temp	230°C
Initial temp	135°C
Temperature program	(A) 5°C/minute (B) 20°C/minute
Final Temp	(A) 245°C (B) 295°C
Final Hold Time	(A) 0 minutes (B) 7 minutes
Column 1	RTX-OPP, 30 meter, 0.32 mm, 0.5 µm film
Column 2	RTX-1, 30 meter, 0.32 mm, 0.5 µm film
Injection	1-2µL
Carrier gas	Helium / Hydrogen
Make up gas	Nitrogen

Table F3 Initial Calibration Concentrations (ng/uL)

COMPOUND	LEVEL1	LEVEL2	LEVEL3	LEVEL4	LEVEL5	LEVEL6	LEVEL7
o,o,o-Triethylp-hosphate	0.2	0.5	1	2	5	10	20
Dichlorvos	0.2	0.5	1	2	5	10	20
Mevinphos	0.2	0.5	1	2	5	10	20
Thionazin	0.2	0.5	1	2	5	10	20
Ethoprop	0.2	0.5	1	2	5	10	20
Naled	0.2	0.5	1	2	5	10	20
Sulfotepp	0.2	0.5	1	2	5	10	20
Phorate	0.2	0.5	1	2	5	10	20
Demeton	0.2	0.5	1	2	5	10	20
Dimethoate	0.2	0.5	1	2	5	10	20
Diazinon	0.2	0.5	1	2	5	10	20
Disulfoton	0.2	0.5	1	2	5	10	20
Methyl Parathion	0.2	0.5	1	2	5	10	20
Ronnel	0.2	0.5	1	2	5	10	20
Fenthion	0.2	0.5	1	2	5	10	20
Chlorpyrofos	0.2	0.5	1	2	5	10	20
Parathion	0.2	0.5	1	2	5	10	20
Malathion	0.2	0.5	1	2	5	10	20
Trichloronate	0.2	0.5	1	2	5	10	20
Merphos	0.2	0.5	1	2	5	10	20
Stirophos	0.2	0.5	1	2	5	10	20
Tokuthion	0.2	0.5	1	2	5	10	20
Fensulfothion	0.2	0.5	1	2	5	10	20
Bolstar	0.2	0.5	1	2	5	10	20
Famphur	0.2	0.5	1	2	5	10	20
Azinphos methyl	0.2	0.5	1	2	5	10	20
Coumaphos	0.2	0.5	1	2	5	10	20

Table F4: LCS/Matrix Spike and Surrogate Spike Compounds – 20 ug/mL

Compound	Compound
Thinazin	Sulfotepp
Phorate	Disulfoton
Methyl Parathion	Parathion
Famphur	O,O,O-Triethylphosphate
Dimethoate	Triphenyl Phosphate - Surrogate

1. Scope and Application

1.1. This method is applicable to the determination of the concentration and tentative identification of petroleum hydrocarbon mixes in waters, wastewaters, soils, and sludges.

1.2. This SOP is based on SW-846 Method 8015B, Modified, Revision 3, December 1996 and Wisconsin DNR Modified DRO method.

1.3. The associated LIMS method codes are HS (8015 MOD), KI (8015B), and C6 (Wisconsin DRO).

2. Summary of Method

2.1. This method provides gas chromatographic conditions for detection and identification of total petroleum hydrocarbons. Prior to the use of this method, appropriate sample preparation techniques are used.

2.2. Wisconsin DRO Method is designed to measure the concentration of diesel range organics in water and soil. This corresponds to a hydrocarbon range of C_{10}-C_{28} and a boiling point range between approximately 170 °C and 430 °C.

2.3. An aliquot of the prepared sample is injected into a gas chromatograph (GC) and compounds in the effluent are detected by a flame ionization detector (FID).

3. Definitions

3.1. Refer to the glossary in the STL North Canton Laboratory Quality Manual (LQM), current version.

3.2. Wisconsin DNR DRO Method Definition - Diesel Range Organics (DRO) – All the chromatographic response falling between the onset of the n-decane (n-C_{10}) peak and the conclusion of the n-octacosane (n-C_{28}) peak. Quantitation is based on a direct comparison of the total area within this range to the total area of the Diesel Component Standard.

3.3. Wisconsin DNR DRO Method Definition –Diesel Component Standard: A ten component blend of typical diesel compounds. This standard serves as a quantitation standard and is used to establish a retention time window for diesel range organics.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

5. SAFETY

5.1. Refer to Section 5 of the Method 8000B SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP.

6.2. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to Section 7 of the 8000B section of this SOP.

7.2. The petroleum hydrocarbons are purchased from a chemical supplier when available. When no chemical supplier is available, the fuels are purchased from public sources.

7.3. The Diesel Component Stock Standard for the Wisconsin DRO method may be commercially prepared standards if the standards are certified by the manufacturer or by an independent source.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

8.1. Refer to Section 8 of the 8000B section of this SOP.

8.2. Wisconsin DNR DRO aqueous samples must be preserved with HCL such that the pH is < 2.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).

9.2. LCS recoveries are calculated from a Diesel Calibration. The Wisconsin DNR DRO method specifies the recovery of the LCS-water spike must be between 75%-115% and the RPD <20%. LCS Soil recoveries must fall between 70%-120% of the known concentration and the RSD must be <20%.

9.2.1. The Wisconsin DNR DRO Method requires a Duplicate Laboratory Control Spike with each batch. MS/MSD samples are not required for this method. One LCS must be run at the beginning of the batch of samples and the other at the end.

9.3. MS/MSD recoveries are calculated from a Diesel calibration.

9.4. Surrogates

9.4.1. Because of the nature of the TPH analysis, whereas certain petroleum mixtures can override the C9 surrogate, the C9 surrogate recoveries are advisory. Re-extraction due to surrogate recoveries is determined by analyst judgement.

NOTE: Ohio VAP rules require reanalysis when surrogate recoveries are outside of control limits.

10. CALIBRATION AND STANDARDIZATION

10.1. Recommended Instrument Conditions

10.1.1. Hydrogen carrier gas - flow rate 5 - 6 mL/min

10.1.2. Detector gas mixture - air hydrogen mixture in a 10:1 ratio, air 80 - 120 mL/min, hydrogen 8 -12 mL/min

10.1.3. Temperature Program - refer to Appendix

10.1.4. Injection volume - 1 µL

10.2. Initial Calibration

10.2.1. Analyze a five point Diesel calibration standard and a five point DRO calibration standard referring to the recommended instrument conditions. The calibration concentrations are 100, 200, 500, 1000, and 2000 ng/uL. A 5000ng/uL standard may be analyzed if needed. The retention time window of C10-C32 shall be used for the Diesel calibration.

10.2.2. The Wisconsin DNR DRO method requires the retention time window be defined as beginning 0.1 minutes before the onset of the n-decane peak and ending 0.1 minutes after the conclusion of the n-octacosane peak in the calibration run.

10.2.3. Wisconsin DNR DRO quantitation is based on a direct comparison of the total area within the retention time window to the total area of the Diesel Component Standard.

10.2.4. Wisconsin DNR DRO integration must be "baseline to baseline" as opposed to a "valley to valley". Baseline to baseline is defined here as a flat baseline drawn parallel to the x-axis of chromatographic graph that includes all responses within the retention time window.

10.2.5. A linear regression calibration curve must be prepared for Wisconsin DNR DRO analysis. The curve must have a correlation coefficient of at least 99%.

10.3. Continuing Calibration

10.3.1. The calibration verification must bracket the LCS, MS, MSD extracts and must meet passing criteria. The Wisconsin DNR DRO method requires the calibration verification must not vary from the calibration curve by more than 20%.

10.4. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP #CORP-OP-0001NC.

11.3. Cleanup

11.4. Analytical Sequence – Refer to Section 11 in the 8000B Section of this SOP.

11.5. Petroleum Hydrocarbon Identification and/or Fingerprinting

11.5.1. To identify the type of petroleum hydrocarbon, compare the chromatographic peak pattern to the patterns of known petroleum hydrocarbons analyzed under identical chromatographic conditions. Samples are quantified against diesel, but fingerprinting may be done when client requested.

11.5.2. Positive matching may not be possible, even using site-specific hydrocarbons. Degradation of the pattern can occur during environmental exposure of the fuel. See Table 2 for possible fingerprints.

11.6. Sample Quantification

11.6.1. Samples are quantified against the initial calibration of diesel or DRO on a single column.

11.6.2. The total height or area of the hydrocarbon is determined in the same manner used for the hydrocarbon standard.

11.6.3. If the amount of sample injected into the GC exceeds the working range of the calibration curve, an appropriate dilution is performed before reanalysis.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. Surrogate recovery results are calculated and reported for Nonane (C-9) unless it is determined that sample interference has adversely affected the quantitation of one of the surrogates. The surrogate must be within QC criteria. Corrective action is only necessary if Nonane (C-9) is outside of acceptance limits.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

14. POLLUTION PREVENTION

Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, Method 8015B, Nonhalogenated Organics Using GC/FID, Test Methods for Evaluating Solid Waste, Third Edition, USEPA

16.2. Method for Determining Diesel Range Organics, Modified DRO, Wisconsin DNR, Public Hearing Draft, September 1995.

16.3. Related SOP

16.3.1. CORP-OP-0001NC, Extraction and Cleanup of Organic Compounds from Waters and Soils, Based on SW846 3500 Series, 3600 Series, 8150, 8151, and 600 Series Methods

Table G1: Suggested GC Temperature Program for TPH analysis

Initial Temperature	40°C
Initial Hold Time	4 minutes
Temperature Program	10°C/minute
Final Temperature	280°C
Final Hold Time	10 minutes

Table G2: Reporting Limits for TPH Analysis

Analyte	Reporting Limits		
	Water (µg/L)	Solids (mg/kg)	Waste Dilution (mg/kg)
TPH (as Diesel) or DRO	100	3.3	200
C10-C32 (Ohio VAP only)	100	3.3	200
Fingerprint Compounds[1]			
Mineral Spirits	Kerosene	Motor Oil	
Hydraulic Oil	Jet Fuel	Stoddard Solvent	
DRO Spiking Solution			
Decane	Dodecane	Tetradecane	
Hexadecane	Octobecane	Eicosane	
Docosane	Tetracosane	Hexacosane	
Octacosane			

[1] This list represents most of the common petroleum hydrocarbons. The list may be expanded to include other petroleum hydrocarbons.

1. Scope and Application

1.1. This method is applicable to the determination of the concentration of various Non-halogenated Organic Compounds in waters, wastes, sludges, and solids. It is based on SW-846 Method 8015B.

1.2. The applicable LIMs method codes are J7 (GC/FID 8015) and QU (Semivolatile Organics, 8015B). The preparation code is 88.

2. Summary of Method

2.1. This method provides gas chromatographic conditions for the detection of various nonhalogenated organic compounds. Samples are introduced to the GC by direct injection. Detection is achieved by a flame ionization detector (FID).

3. Definitions

3.1. Refer to the glossary in the STL North Canton Laboratory Quality Manual (LQM).

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP.

6.2. Recommended Columns:

6.2.1. RTX Stabilwax, fused silica, 60 m x 0.53, 0.5 µm film thickness, or equivalent column.

6.2.2. Stabilwax-DA, fused silica, 60 m x 0.32, 0.5 µm film thickness, or equivalent column.

6.3. Detectors: Flame ionization (FID)

6.4. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to Section 7 of the 8000B section of this SOP.

7.1.1. Reagent water

7.2. Standards

7.2.1. Refer to Table H2.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

8.1. Refer to section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).

10. CALIBRATION AND STANDARDIZATION

10.1. Recommended Instrument Conditions

10.1.1. The following conditions are recommended. The following table lists specific information for various compounds.

10.1.2. RTX- Stabilwax and Stabilwax-DA Columns:

Carrier Gas:	Hydrogen
Initial Temp:	45 °C
Initial Hold:	3 mins
Ramp Rate A:	5 °C/min
Ramp Rate B:	30 °C/min
Final Hold A:	0 min
Final Hold B:	3 mins
Analysis Time:	17.83 mins
Injector Temp:	275 °C
FID Temp:	300 °C
Injection Vol:	1 µL

10.2. Initial Calibration

10.2.1. For each non-halogenated organic compound and surrogate standard, analyze five or more calibration standards referring to the recommended GC conditions in Section 10.1. One of the standards analyzed should be at or near the concentration which corresponds to the calibration range.

10.2.2. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for procedural requirements.

11.2. Sample Preparation Summary

11.2.1. Samples received fall into three general categories: waters, soils, or wastes.

11.3. Sample Preparation Procedure

11.3.1. Waters

11.3.1.1. Add surrogate solution to the sample to achieve a concentration of 100 mg/L.

11.3.2. Sediments/soils and waste

11.3.2.1. Mix the contents of the sample container with a narrow metal or wood spatula. Weigh 5 g (wet weight) into a tarred culture tube. Use a top-loading balance. Record the weight to 0.01 gram.

11.3.2.2. Quickly add 5 mL of reagent water. Add surrogate standard. Cap the vial and vortex to mix for two minutes.

11.3.2.3. If extract is cloudy or has suspended sediment particles, refrigerate and allow sample to sit for a maximum of 24 hours. Filter sample if necessary.

11.4. Sample Analysis

11.4.1. Preliminary Evaluation

11.4.1.1. The sample or sample extract is introduced to the GC column by direct inject techniques. The concentration of the sample components is then calculated from the resulting chromatograms.

11.4.2. Analytical Sequence – Refer to Section 11 in the 8000B Section of this SOP.

11.4.3. Inject 1 µL of the sample extract or diluted sample into the GC using the same operating conditions and techniques as those used in the calibration of the instrument.

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.5.2. All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. Surrogate recovery results are calculated and reported unless it is determined that sample interference has adversely affected the quantitation of the surrogate. The surrogate must be within QC criteria. Corrective action is only necessary if the surrogate is outside of acceptance limits.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

14. POLLUTION PREVENTION

Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, Method 8015B, Nonhalogenated Organics Using GC/FID, Test Methods for Evaluating Solid Waste, Third Edition, USEPA

17. Miscellaneous (Tables, Appendices, Etc...)

Table H1 Non-halogenated Organic Compounds, Reporting Limits[1]

Compound	CAS Number	Reporting Limits	
		Water, mg/L	Solid, mg/kg
2-Methoxyethanol	109-86-4	1.0	1.0
Methanol	67-56-1	1.0	0.5
Isopropyl alcohol	67-63-0	1.0	0.5
n-Propyl alcohol	71-23-8	1.0	0.5
Ethanol	64-17-5	1.0	0.5
n-Butanol	71-36-3	1.0	0.5
1,4-Dioxane	123-91-1	1.0	0.5

Compound	CAS Number	Reporting Limits	
		Water, mg/L	Solid, mg/kg
Ethylene oxide	75-21-8	1.0	0.5
iso-Butanol	78-83-1	1.0	0.5

[1] If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

Table H2 Non-halogenated Organic Compounds Working Standards

COMPOUND	LEVEL1	LEVEL2	LEVEL3	LEVEL4	LEVEL5	LEVEL 6
2-Methoxyethanol	1.0	2.0	5.0	10.0	20.0	50.0
Methanol	1.0	2.0	5.0	10.0	20.0	50.0
Isopropyl alcohol	1.0	2.0	5.0	10.0	20.0	50.0
n-Propyl alcohol	1.0	2.0	5.0	10.0	20.0	50.0
Ethanol	1.0	2.0	5.0	10.0	20.0	50.0
n-Butanol	1.0	2.0	5.0	10.0	20.0	50.0
1,4-Dioxane	1.0	2.0	5.0	10.0	20.0	50.0
Ethylene oxide	1.0	2.0	5.0	10.0	20.0	50.0
iso-Butanol	1.0	2.0	5.0	10.0	20.0	50.0

1. Scope and Application

1.1. This method is applicable to the determination of the concentration of Sulfolane and N-Methyl-2-pyrrolidone in water and solid samples. It is based on SW846 Method 8015B. The working linear range is 50 to 1000 µg/L. Table I1 lists the reporting limits associated with this method.

1.2. The applicable LIMS method code is KU.

2. Summary of Method

2.1. This method provides gas chromatographic conditions for the detection of mg/L levels of Phillips 66 compounds in water. Prior to use of this method, appropriate sample extraction techniques must be used.

3. Definitions

3.1. Refer to the glossary in the STL North Canton Laboratory Quality Manual (LQM), current version.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

5.2. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.2.1. Chemicals that have been classified as **carcinogens**, or **potential carcinogens**, under OSHA include: **Methylene Chloride.**

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP.

6.2. Gas Chromatograph

6.2.1. Gas Chromatograph: Modified to accept capillary columns

6.2.2. Data System: Capable of peak integration

6.2.3. Gas Chromatographic Column: 30 m x 0.32 mm ID RTX-5 fused silica capillary column

6.2.4. Autosampler: Capable of reproducible injections

6.2.5. Carrier Gas: Hydrogen

6.2.6. Detector: Flame ionization (FID)

6.3. Volumetric Flasks: 10, 50, and 100 mL

6.4. Microsyringe: 10 µL

6.5. Pipettes: Disposable µL, Pasteur

6.6. Autosampler Vials: 1 mL with 11 mm crimp cap, Teflon®/silicone septum liner.

7. REAGENTS AND STANDARDS

7.1. Refer to Section 7 of the 8000B section of this SOP.

7.2. Reagents

7.2.1. Methylene Chloride: Pesticide grade or equivalent

7.3. Standards

7.3.1. Refer to Section 7 of the 8000B section of this SOP.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

8.1. Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).

9.2. Surrogates are not used for this analysis.

10. CALIBRATION AND STANDARDIZATION

10.1. Initial Calibration

10.1.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for procedural requirements.

11.2. Sample Analysis

11.2.1. Summary

11.2.1.1. The sample extract is injected onto the GC column. The compounds are then identified and quantitated.

11.2.2. Recommended Instrument Conditions

11.2.2.1. GC Conditions

Initial Temperature:	45°C
Initial Hold Time:	4 minutes
Temperature Program:	15°C/minute
Final Temperature:	300°C, hold 2 minutes
Final Time:	23 minutes
Carrier Gas:	Hydrogen
Injection Volume:	1 µL

11.2.3. Sample Analysis Procedure

11.2.3.1. Preliminary Evaluation

The sample extracts may be screened to determine the level of analyte present. If the level of analyte exceeds the working range of the calibration curve, an appropriate dilution is performed to bring the level within the calibration range.

11.2.4. Inject 1 µL of the sample extract or diluted sample into the GC using the same conditions as those used in calibration.

11.2.5. Identification

11.2.5.1. Analytes of interest are identified by comparing retention times with known standards.

11.2.5.2. A single column is used for identification.

11.2.6. Sample Quantification

11.2.6.1. Refer to Section 11 in the 8000B Section of this SOP

11.3. Analytical Documentation

11.3.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.3.2. All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.3.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

14. POLLUTION PREVENTION

14.1. Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, Method 8015B, Nonhalogenated Organics Using GC/FID, Test Methods for Evaluating Solid Waste, Third Edition, USEPA

17. Miscellaneous (Tables, Appendices, Etc...)

17.1. Reporting limits

17.1.1. The lower reporting limits are shown in Table I1

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

TABLE I1
PHILLIPS 66 REPORTING LIMITS

Compound	Reporting Limits, µg/L
Tetramethylene sulfone (Sulfolane)	50
N-Methyl-2-pyrrolidone	50

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SOP No. CORP-IP-0003NC
Revision No. 1.3
Revision Date: 09/25/01

STL STANDARD OPERATING PROCEDURE

TITLE: ACID DIGESTION OF AQUEOUS SAMPLES BY SW846 AND MCAWW 200 SERIES METHODS

(Supersedes: Revision 1.2 Dated 03/20/00)

Prepared by: _____ 9/26/01
Date

Reviewed by: _____ 9.26.01
Technology Specialist Date

Reviewed by: _____ 9/26/01
Quality Assurance Manager Date

Reviewed by: _____ 9-27-01
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Reviewed by: _____ 9-28-01
Laboratory Director Date

Reviewed by: _____
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Date

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of aqueous samples for the analysis of certain metals by Graphite Furnace Atomic Absorption (GFAA), Flame Atomic Absorption (FLAA), Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP), and Inductively Coupled Plasma-Mass Spectrometry (ICP/MS) using the MCAWW 200 series methods (NPDES) and SW846 Methods 3005A, 3010A, 3020A and 7060A/7740 (RCRA).

1.2. The applicability of each of these preparation protocols to specific analytes is detailed in Tables I and II (Appendix A) and the applicable determinative methods are illustrated by Figures 6 and 7 (Section 17). Additional elements may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.

1.3. This SOP provides procedures applicable to the preparation of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, certain aqueous sludges, wastes, and biological tissues, and leachates/extracts.

1.4. SW-846 Method 3005A is used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by FLAA, ICP and GFAA (antimony only).

1.5. MCAWW Method 200.7 Section 9.4 is used to prepare surface water, domestic and industrial waste samples for total recoverable and dissolved metals determination by ICP.

1.6. SW-846 Method 3010A is used to prepare aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for total metals analysis by FLAA or ICP.

1.7. MCAWW Method 200.7 Section 9.3 is used to prepare surface water and wastes that contain suspended solids for total metals analysis by ICP.

1.8. SW-846 Method 3020A is used to prepare aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for total metals by GFAA, or ICP/MS.

1.9. MCAWW Method 200.0 Section 4.1.3 is used to prepare surface water and wastes that contain suspended solids for total metals analysis by GFAA.

1.10. MCAWW Method 200.0 Section 4.1.4 is used to surface water, domestic and industrial waste samples for total recoverable and dissolved metals determination by GFAA.

1.11. SW-846 Methods 7060A and 7740, respectively, contain the procedure for the preparation of aqueous samples for arsenic and selenium.

1.12. MCAWW Methods 206.2 and 270.2, respectively, contain the procedure for the preparation of aqueous samples for arsenic and selenium.

1.13. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators do require digestion of dissolved samples and this must be clarified before project initiation.

2. SUMMARY OF METHOD

2.1. Method 3005A / Method 200.7 Section 9.4 - Preparation for Total Recoverable or Dissolved Metals Analysis by FLAA or ICP Spectroscopy

2.1.1. A representative aliquot of sample is heated with nitric and hydrochloric acids and substantially reduced in volume. The digestate is filtered (if necessary) and diluted to volume.

2.2. Method 3010A / Method 200.7 Section 9.3 - Preparation for Total Metals Analysis by FLAA or ICP Spectroscopy

2.2.1. A representative aliquot of sample is refluxed with nitric acid. After the digestate has been reduced to a low volume, it is refluxed with hydrochloric acid, filtered (if necessary) and brought up to volume.

2.3. Method 3020A / Method 200.0 Section 4.1.3 - Preparation for Total Metals for Analysis by GFAA Spectroscopy and ICP/MS.

2.3.1. A representative aliquot of sample is refluxed with nitric acid. After the digestate has been reduced to a low volume, it is cooled, filtered (if necessary) and brought up to volume.

2.4. Methods 7060A/206.2 and Methods 7740/270.2 - Preparation for Arsenic/Selenium Analysis by GFAA

2.4.1. A representative aliquot of sample is heated with nitric acid and peroxide until the digestate has been reduced to a low volume. The sample is cooled, filtered (if necessary) and brought up to volume.

2.5. Method 200.0 Section 4.1.4 - Total Recoverable GFAA Preparation (NPDES)

2.5.1. A representative aliquot of sample is heated with nitric acid and until the digestate has been reduced to a low volume. The sample is cooled, filtered (if necessary) and brought up to volume.

3. DEFINITIONS

Additional definitions of terms used in this SOP may be found in the glossary of the QAMP.

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following digestion.

3.4. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include: metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2. The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix D for additional contamination control guidelines.

4.3. Boron and silica from the glassware will migrate into the sample solution during and following sample processing. For critical low level determinations of boron and silica, only quartz and/or plastic labware should be used.

4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents and other matrices may not be digested using these methods if they are not soluble with acids. If physical interferences are present, they should be documented.

4.5. Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.6. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs the sample must be reprepared. Antimony is easily lost by volatilization from hydrochloric acid media.

4.7. Precipitation of silver chloride (AgCl) may occur when chloride ions and high concentrations of silver (i.e., greater than 1 mg/L) are present in the sample.

4.8. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. The following materials are known to be **corrosive:**

hydrochloric acid and nitric acid.

5.3.2. The following materials are known to be **oxidizing agents:**

nitric acid and hydrogen peroxide.

5.3.3. All sample digestions, including cooling of digestates, must be carried out in a fume hood.

5.4. The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood. The analyst should also be aware of the potential for a vigorous reaction.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor.

5.7. Always carry bulk concentrated acid bottles in appropriate impact proof containers.

5.8. Acid/peroxide spills must be neutralized immediately, flushed with water and cleaned up using appropriate spill kits.

5.9. Discard chipped or broken beakers to prevent injury. Chipped glassware may be fire polished as an alternative to disposal.

5.10. Any and all accidents and spills must be reported to the lab supervisor or EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

6.1. Hot plate, digestion block or other adjustable heating source capable of maintaining a temperature of 95°C (± 4).

6.2. Calibrated thermometer that covers a temperature range of 0-200°C.

6.3. Griffin beakers of assorted sizes or equivalent.

6.4. Watch glasses, ribbed or equivalent.

6.5. Whatman No. 4 filter paper or equivalent.

6.6. Funnels or equivalent filtration apparatus.

6.7. Centrifugation equipment (if desired method of removing particulates is centrifugation).

6.8. Graduated cylinder or equivalent capable of measuring 50 mL within 3% accuracy.

6.9. Analytical balance capable of accurately weighing to the nearest 0.01 grams.

6.10. Repipetors or suitable reagent dispensers.

6.11. Calibrated automatic pipettes with corresponding pipette tips or Class A glass volumetric pipettes.

6.12. Class A volumetric flasks.

6.13. pH indicator strips (pH range 0 - 6).

6.14. Plastic digestate storage bottles.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative SOPs.

7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom STL solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Working ICP LCS/MS spike solution: Prepare the ICP LCS/MS working spike solution from custom stock standards to the final concentration listed in Table III. The working

spike must be prepared in a matrix of 5% HNO_3. This acid (5 mL of concentrated HNO_3 per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working ICP LCS solution must be made fresh every three months.

7.4. Working GFAA LCS/MS spike solution: Prepare the GFAA working LCS spike solution by diluting the custom stock solution (7.2) 200x. The working spike solution must be prepared in a matrix of 5% HNO_3. This acid (5 mL of concentrated HNO_3 per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working GFAA LCS solution must be made fresh every three months.

7.5. The TCLP MS working spike solution is provided directly by the vendor, no further standard preparation is necessary.

7.6. The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom STL solution, the individual facility must purchase a solution from the designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.7. Aqueous laboratory control samples (LCSW) and matrix spike samples are prepared as described in Sections 9.5 and 9.6. Refer to Tables III and IV(Appendix A) for details regarding the stock, working standard and final digestate spike concentrations for ICP and GFAA LCS and matrix spike preparations.

7.8. Nitric acid (HNO_3), concentrated, trace metal grade or better.

7.9. Nitric acid, 1:1 - dilute concentrated HNO_3 with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

7.10. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.11. Hydrochloric acid, 1:1 - dilute concentrated HCl with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

7.12. 30% Hydrogen peroxide (H_2O_2), reagent grade.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. For dissolved metals analysis, the samples should be filtered through a 0.45 um filter prior to preservation. Filtration must be done in the field or within 24 hours of collection.

Note: If a sample being analyzed for dissolved metals is found to contain sediment the analyst should contact their supervisor or group leader. The client should be notified of the problem to decide how to treat the sample.

9. QUALITY CONTROL

Table VI (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using any method contained within this SOP the following requirements must be met:

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDL's must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in STL QA Policy QA-005. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the STL reporting limit.

9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which should contain all the analytes of interest. The results of the initial demonstration study must be acceptable before analysis of samples may begin. The results of the initial demonstration study may be used to

extend a method for the analysis of other elements provided all acceptance criteria are met.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.1.2.2. Calculations and acceptance criteria for QC check samples are given in the determinative SOPs (CORP-MT-0001, CORP-MT-0003).

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS) are not included in the sample count for determining the size of a preparation batch. MS/MSD are not included in the sample count unless there are multiple sets of MS/MSD per batch. In other words, the first MS/MSD are not counted; all additional MS and MSDs are counted as samples.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Criteria for the acceptance of blanks are contained within the individual analytical method SOP's. If the method blank does not meet the criteria contained within the analytical method SOPs; the blank and all associated samples in the batch must be redigested.

9.4.1. Aqueous method blanks are prepared by taking 50 mL or 50 g of reagent water through the appropriate procedure as described in Section 11.

9.4.2. TCLP method blanks are prepared by taking 50 mL or 50 g of leachate fluid through the appropriate procedure as described in Section 11.

9.5. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried

through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOP's. Corrective action when LCS results fail to meet control limits will be repreparation and reanalysis of the batch. Refer to Section 7.3 and 7.4 for instructions on preparation of the aqueous LCS spike solution.

9.5.1. The aqueous LCS is prepared by spiking a 50 mL aliquot of reagent water with 1.0 mL of the working LCS/MS spike solution (7.3 or 7.4). The LCS is then processed through the appropriate procedure as described in Section 11.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis. If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. Corrective action when MS results fail to meet control limits does not include repreparation of samples unless the results indicate that a spiking error may have occurred.

9.6.1. The aqueous matrix spike sample is prepared by spiking a 50 mL aliquot of a sample with 1.0 mL of the working LCS/MS spike solution (7.3 or 7.4). The matrix spike sample is then processed as described in Section 11.

9.6.2. The TCLP matrix spike sample is prepared by spiking a 50 mL aliquot of a leachate with 0.5 mL of the working TCLP spike solution (7.5). The matrix spike sample is then processed as described in Section 11.

NOTE: The TCLP matrix spike must be added prior to preservation of the leachate.

9.6.3. If insufficient sample is available to process a MS/MSD, then a second LCS must be processed. The LCS pair is then evaluated according to the MS/MSD criteria.

9.7. Quality Assurance Summaries - Certain clients may require specific project or program QC that may supersede the SOP requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

10. CALIBRATION AND STANDARDIZATION

10.1 Hotplate temperature must be verified daily for each hotplate used and must be recorded on either the metals preparation log or in a hotplate temperature logbook. The hotplate temperature should be verified by measuring the temperature of a beaker of reagent water placed on each hotplate.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. All digestion procedures must be carried out in a properly functioning hood.

11.4. All samples are to be checked out of sample control with the chain of custody documentation filled out completely.

11.5. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample.

11.6. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating prep, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge like, organic liquid, lots of sediment etc.) contact the lab supervisor or project manager for further instructions. In some cases it may be more appropriate to process these samples as solids.

11.7. If possible prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and reporting group.

11.8. In most cases, both AA and ICP digests are required on each sample. It is recommended that both aliquots be measured out and processed at the same time.

11.9. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

11.10. The following procedure must be followed for all aqueous sample preparations:

11.10.1. Measure sample pH with pH paper on a separate aliquot of sample.

Note: If the sample pH is > 2 pH units, the client must be notified of the anomaly.

Note: If sample pH has already been verified and documented in sample receipt this step may be omitted.

11.10.2. Mix sample by shaking the container.

11.10.3. Measure and transfer 50 mL or 50 g of the sample into a beaker.

Note: This SOP allows for samples to be weighed instead of measured volumetrically.

11.10.4. Measure two extra aliquots of sample selected for the MS/MSD analysis. Spike each aliquot with the appropriate spiking solutions (7.3-7.5,9.6).

11.10.5. Measure and transfer 50 mL of reagent water into a beaker for the method blank.

11.10.6. Measure and transfer 50 mL of reagent water into a beaker for the LCS and add the appropriate spiking solutions (7.3-7.5,9.6).

11.11. Proceed to the appropriate Section for the desired method as follows:

Method 3005A or Method 200.7 Section 9.4	11.12
Method 3010A or Method 200.7 Section 9.3	11.13
Method 3020A or Method 200.0 Section 4.1.3	11.14
Method 7060A/7740 or Method 206.2/270.2	11.15
Method 200.0 Section 4.1.4	11.16

11.12. **Method 3005A / Method 200.7 Section 9.4 - Preparation for Total Recoverable or Dissolved Metals Analysis by FLAA or ICP (See Figures 1, 6 and 7)**

11.12.1. To the sample beaker, add 1 mL of concentrated HNO_3 and 2.5 mL of concentrated HCl.

11.12.2. Cover with ribbed watch glass.

11.12.3. Heat at 95°C (± 4) until volume is reduced to between 15 and 20 mL.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.

11.12.4. Cool the beaker in a fume hood.

11.12.5. Wash down beaker walls and watch glass with reagent water.

11.12.6. Filter sample, if insoluble materials are present, through Whatman 4 filter paper that has been pre-rinsed with dilute nitric acid.

Note: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.12.7. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

11.12.8. Adjust the final volume/mass to 50 mL or 50 g with reagent water. The sample is now ready for analysis

11.13. **Method 3010A / Method 200.7 Section 9.3 - Preparation for Total Metals Analysis by FLAA or ICP Spectroscopy (See Figures 2, 6 and 7)**

11.13.1. To the sample beaker, add 3.0mL of concentrated HNO_3.

11.13.2. Cover with ribbed watch glass.

11.13.3. Place beaker on hotplate 95°C (± 4) and evaporate for 4-5 hours or to low volume of 15-20 mL while ensuring that no portion of the bottom of the beaker is allowed to go dry.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.

11.13.4. If necessary, add another 1.5 ml portion of concentrated HNO_3 and re-cover the beaker. Reflux 15 minutes.

11.13.5. Add 5 mL of 1:1 HCl.

11.13.6. Cover and reflux for an additional 15 minutes to dissolve precipitate or residue. Cool in a fume hood.

11.13.7. Wash down beaker walls and watch glass with reagent water.

11.13.8. Filter sample, if insoluble materials are present, through Whatman 4 filter paper.

Note: If any samples in the QC batch are filtered the method blank and LCS associated with that batch must also be filtered.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.13.9. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

11.13.10. Adjust final volume/mass to 50 mL or 50 g with reagent water. The sample is now ready for analysis.

11.14. **Method 3020A / Method 200.0 Section 4.1.3 - Preparation for Total Metals Analysis by GFAA or Total Recoverable Metals by ICPMS (See Figures 3, 6 and 7)**

11.14.1. To the sample beaker, add 1.5 mL of concentrated HNO_3.

11.14.2. Cover with ribbed watch glass.

11.14.3. Place beaker on hotplate 95°C (± 4) and evaporate to low volume of 15-20 mL while ensuring that no portion of the bottom of the beaker is allowed to go dry.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.

11.14.4. If necessary, add another 1.5 mL portion of concentrated HNO_3. Recover, and reflux 15 minutes. Cool the beaker in a fume hood.

11.14.5. Filter sample, if insoluble materials are present, through Whatman 4 filter paper.

Note: If any samples in the QC batch are filtered the method blank and LCS associated with that batch must also be filtered.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.14.6. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

11.14.7. Adjust final volume to 50 mL with reagent water. The sample is now ready for analysis.

11.15. **Method 7060A/7740 and Method 206.2/270.2 - Preparation for Arsenic and Selenium Analysis by GFAA (See Figures 4, 6 and 7)**

11.15.1. To the sample beaker, add 1 mL of 30 % H_2O_2 and 1.0 mL of 1:1 HNO_3.

11.15.2. Heat, until the digestion is complete, at 95°C (± 4) or until the volume has been reduced to 15-20 mL.

11.15.3. Cool beaker.

11.15.4. Filter sample, if insoluble materials are present, through Whatman 4 filter paper that has been pre-rinsed with dilute nitric acid.

Note: If any samples in the QC batch are filtered the method blank and LCS associated with that batch must also be filtered.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.15.5. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

11.15.6. Adjust final volume to 50 mL with reagent water. The sample is now ready for analysis.

11.16. **Method 200.0 Section 4.1.4 - Preparation for Total Recoverable GFAA Analyses. (See Figures 5 and 7)**

11.16.1. To the sample beaker, add 1.0 mL of 1:1 HNO_3.

11.16.2. Heat, until the digestion is complete, at 95°C (± 4) or until the volume has been reduced to 15 - 20 mL.

11.16.3. Cool beaker.

11.16.4. Filter sample, if insoluble materials are present, though Whatman 4 filter paper.

Note: If any samples in the QC batch are filtered the method blank and LCS associated with that batch must also be filtered.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.16.5. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

11.16.6. Adjust final volume to 50 mL with reagent water. The sample is now ready for analysis.

12. DATA ANALYSIS AND CALCULATIONS

Not Applicable.

13. METHOD PERFORMANCE

13.1. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. In general, the matrix spike recovery should fall within +/- 20 % and the matrix spike duplicates should compare within 20% RPD. Method blanks must meet the criteria specified in determinative SOPs. The laboratory control samples should recover within 20% of the true value until in house control limits are established. Acceptance criteria are given in the determinative SOPs.

13.2. The initial demonstration study as detailed in Section 9.1.2 must be acceptable before the analysis of field samples under this SOP may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

13.3. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The facility EH & S coordinator should be contacted if additional information is required.

15.2. Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, Revision 1, July 1992. Methods 3005A, 3010A, 3020A, 7060A and 7740A.

16.2. Methods for the Chemical Analysis of Water and Waste (MCAWW), 1983.

16.3. CORP-MT-0001, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis of Water and Wastes, Method 6010A and Method 200.7.

16.4. CORP-MT-0003, Graphite Furnace Atomic Absorption Spectroscopy, SW846 Method 7000A and MCAWW 200 Series Methods.

16.5. QA-003, STL QC Program.

16.6. QA-004, Rounding and Significant Figures.

16.7. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference methods.

17.1.1. Modifications applicable to SW-846 reference methods.

17.1.1.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

17.1.1.2. The referenced methods as well as Table 3-1 of SW-846 refer to the use of a 100 mL aliquot for digestion. This SOP requires the use of a 50 mL sample size to reduce waste generation. The use of reduced sample volumes are supported in EPA's document "Response to Public

Comments Background Document, Promulgation of the Second Update to SW-846, Third Edition" dated November 3, 1994. This document stated "flexibility to alter digestion volumes is addressed and "allowed" by the table (3-1) and is also inherently allowed by specific digestion methods. Table 3-1 is only to be used as guidance when collecting samples..." EMSL-Ci has also taken the stance that "reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology." Additionally, in written correspondence from the Office of Solid Waste, Olliver Fordham stated " As a "representative sample" can be assured, scaling causes no loss of precision and accuracy in the analysis."

17.1.2. Modifications Specific to Method 3010A

17.1.2.1. Section 11.13.7 of this SOP requires the sample be reduced to a volume of 15 - 20 mL. Section 7.2 of Method 3010A states the volume should be reduced to 3 mL but also states that no portion of the bottom of the beaker should go dry. The SOP required volume is a closer approximation of the volume required to provide an adequate covering of the beaker so as to prevent the loss of critical analytes through volatilization.

17.1.2.2. The scope of 3010A has been expanded to include silver based on comparison studies with 7760A. Method 3010A consistently demonstrated improved accuracy and precision over Method 7760A in the matrices tested (reagent water, surface water and TCLP leachate) up to a concentration of 1 ppm silver.

17.1.3. Modifications Specific to Method 3020A

17.1.3.1. Section 11.14.8 of this SOP requires the sample be reduced to a volume of 15 - 20 mL. Section 7.2 of Method 3010A states the volume should be reduced to 3 mL but also states that no portion of the bottom of the beaker should go dry. The SOP required volume is a closer approximation of the volume required to provide an adequate covering of the beaker so as to prevent the loss of critical analytes through volatilization.

17.1.4. Modifications Specific to Method 7060A/7740

17.1.4.1. Methods 7060A and 7740A incorporate the use of a two step dilution to accommodate the addition of a nickel nitrate modifier. This SOP performs the dilution directly in one step and omits the addition of the modifier. The modifier is added automatically at the instrument by direct injection into the furnace.

17.1.5. Modifications Specific to MCAWW Methods

It was determined by technical review that several of the MCAWW methods were equivalent to the SW-846 methods and therefore were combined under the scope of this SOP as described in Section 11.0. The nature of the differences were deemed insignificant in regards to the amount of acid added and the evaporative volume based on the flexibility allowed by the methods (i.e., add additional acid as required) and the subjective wording of the methods (i.e., evaporate to near dryness vs. an exact volume).

17.2. Modifications from previous SOP

17.2.1. Added ICP/MS to the digestion procedures.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none. Refer to the Appendices for any facility specific information required to support this SOP.

17.4. Documentation and Record Management

The preparation benchsheet should, at a minimum, include the following information:

- Preparation date, analyst name, matrix, prep type (ICP or GFAA), SOP reference.
- Sample ID; initial weight/volume and final weight/volume.
- Standards Documentation (source, lot, prep date, volume added).
- Analyst Signature.

- Reviewer's Signature and date.

Figure 1. Method 3005A / Method 200.7 Section 9.4 (Section 11.12)

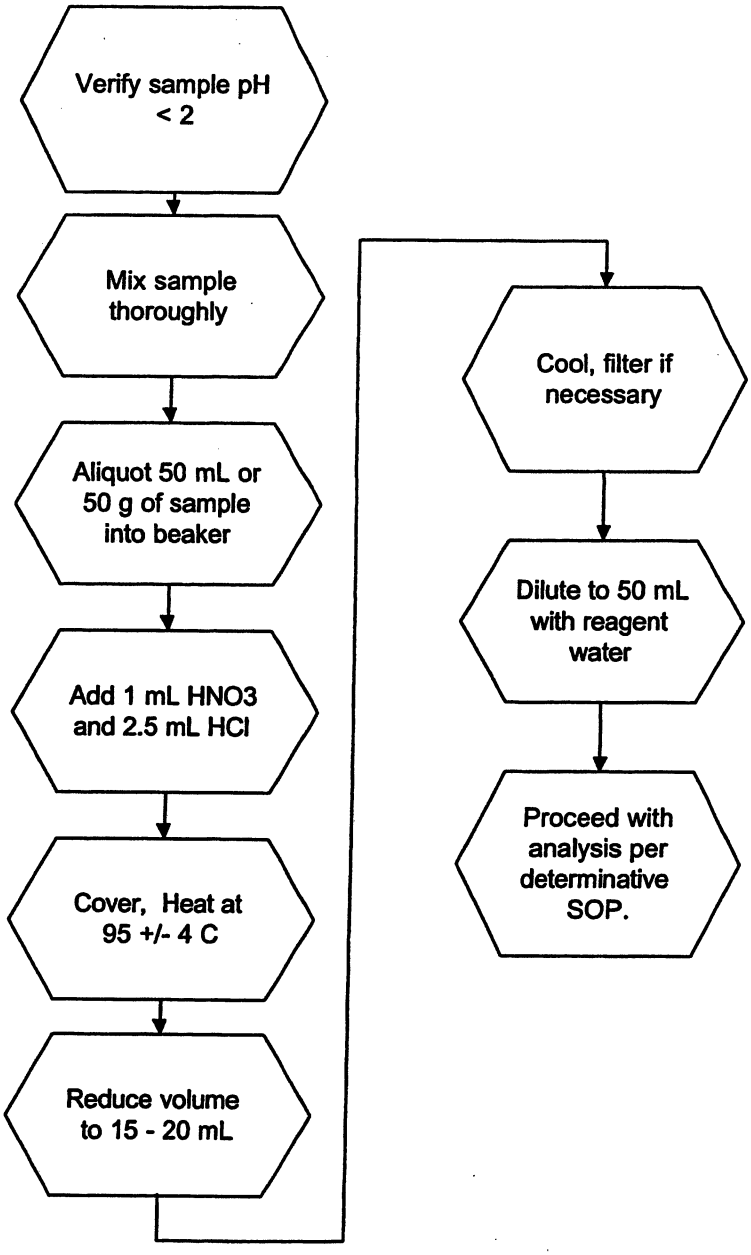


Figure 2. Method 3010A / Method 200.7 Section 9.3 (Section 11.13)

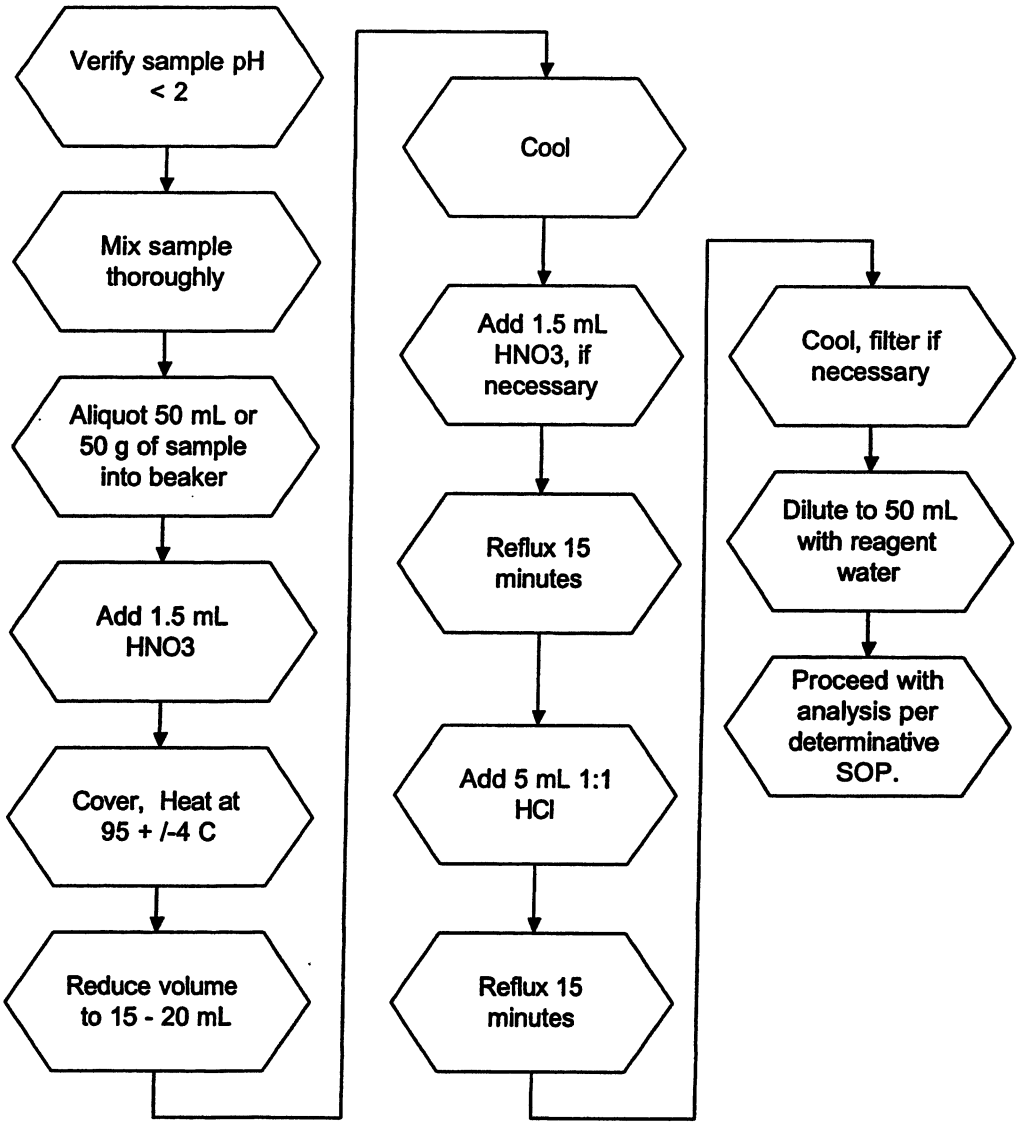


Figure 3. Method 3020A / Method 200.0 Section 4.1.3 (Section 11.14)

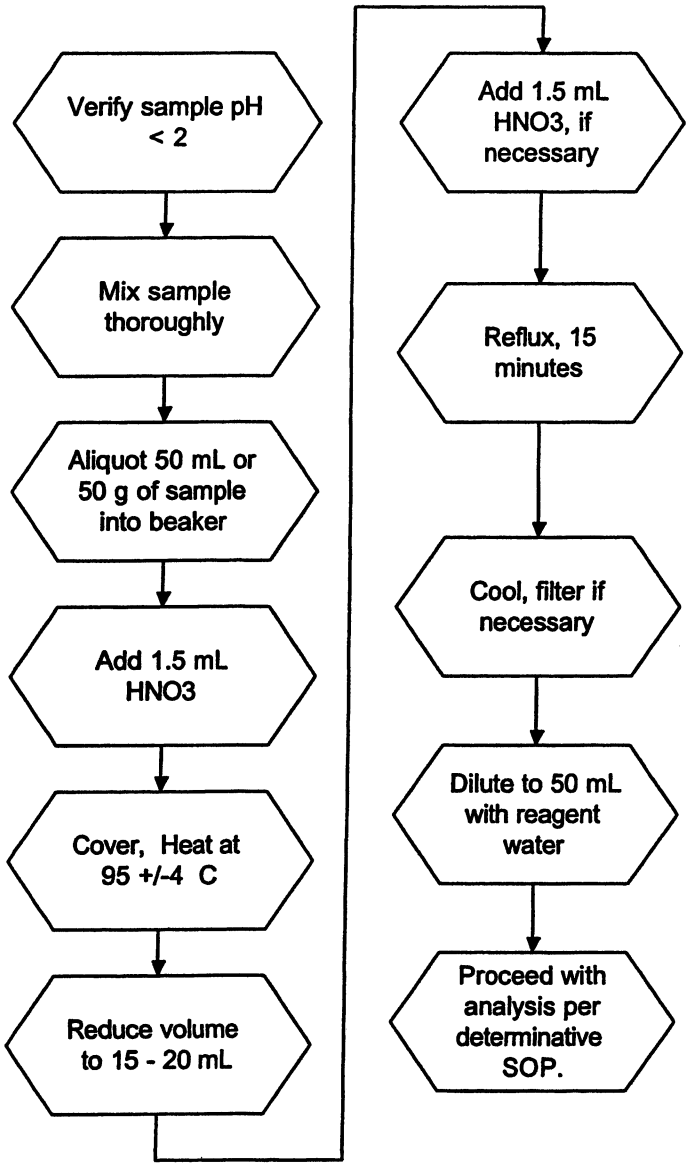


Figure 4. Method 7060A/7740A and Method 206.2/270.2 (Section 11.15)

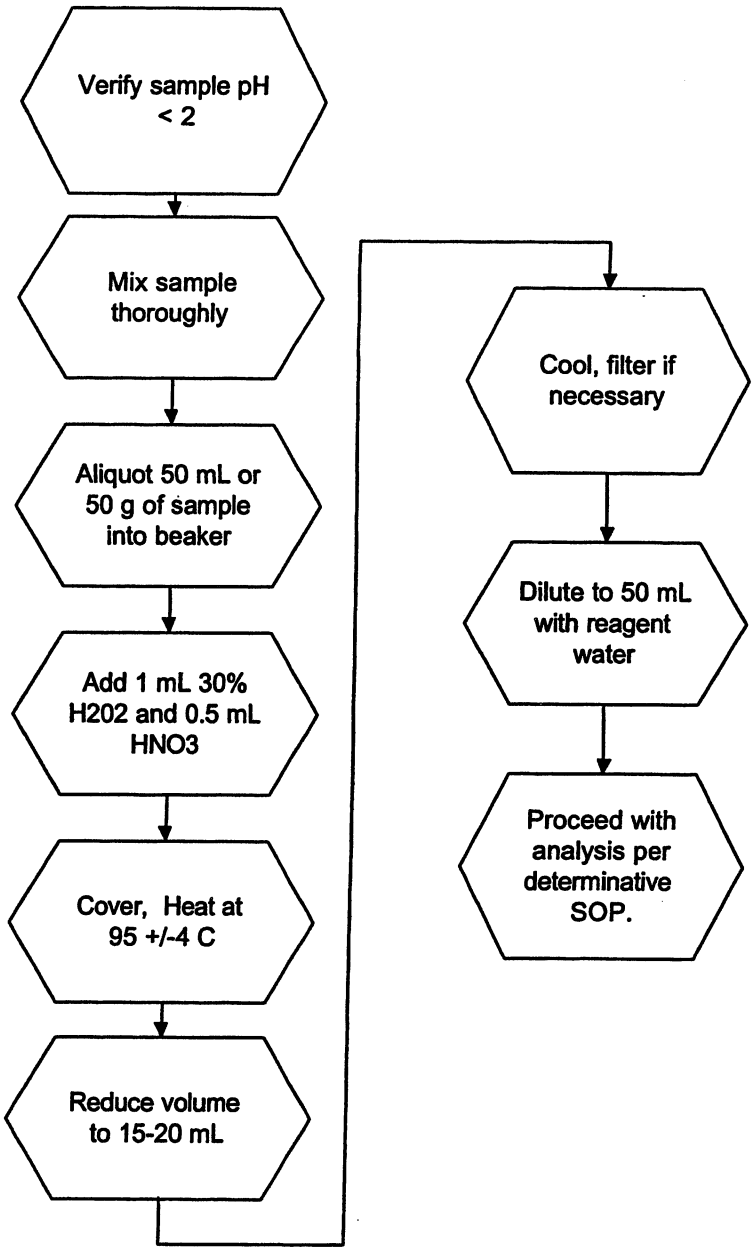


Figure 5. Method 200.0 Section 4.1.4 (Section 11.16)

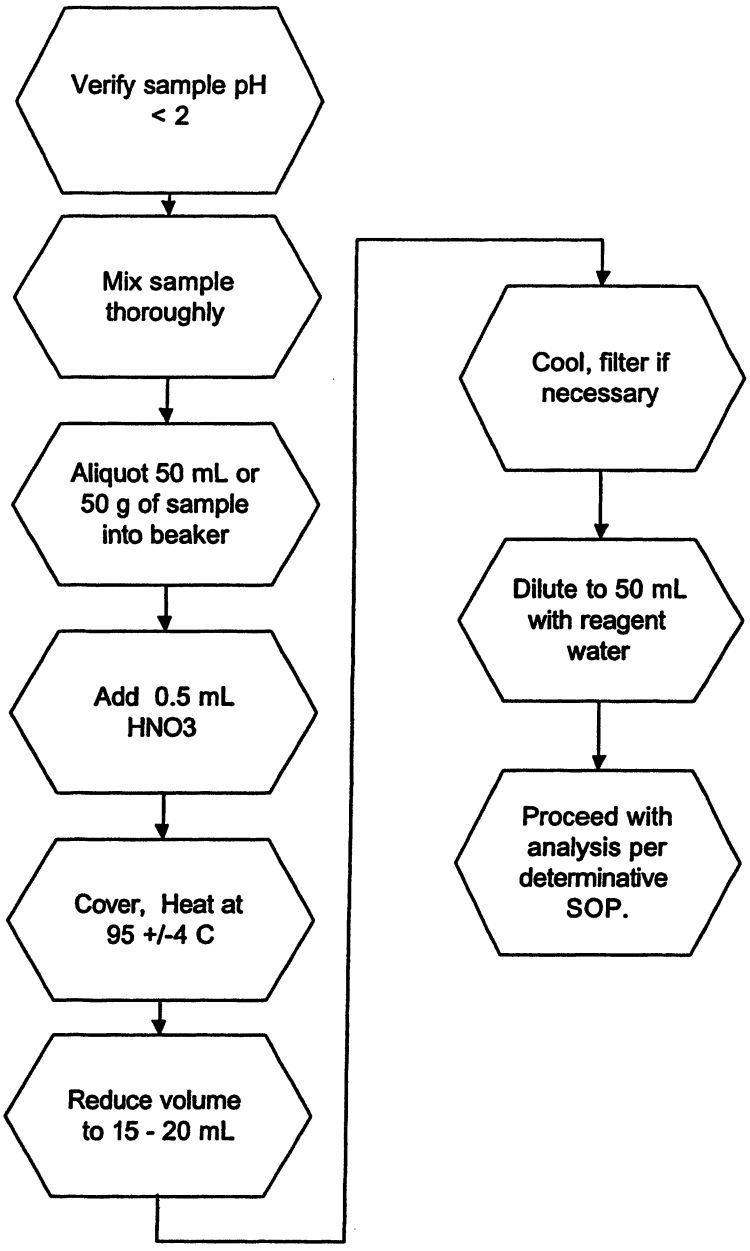
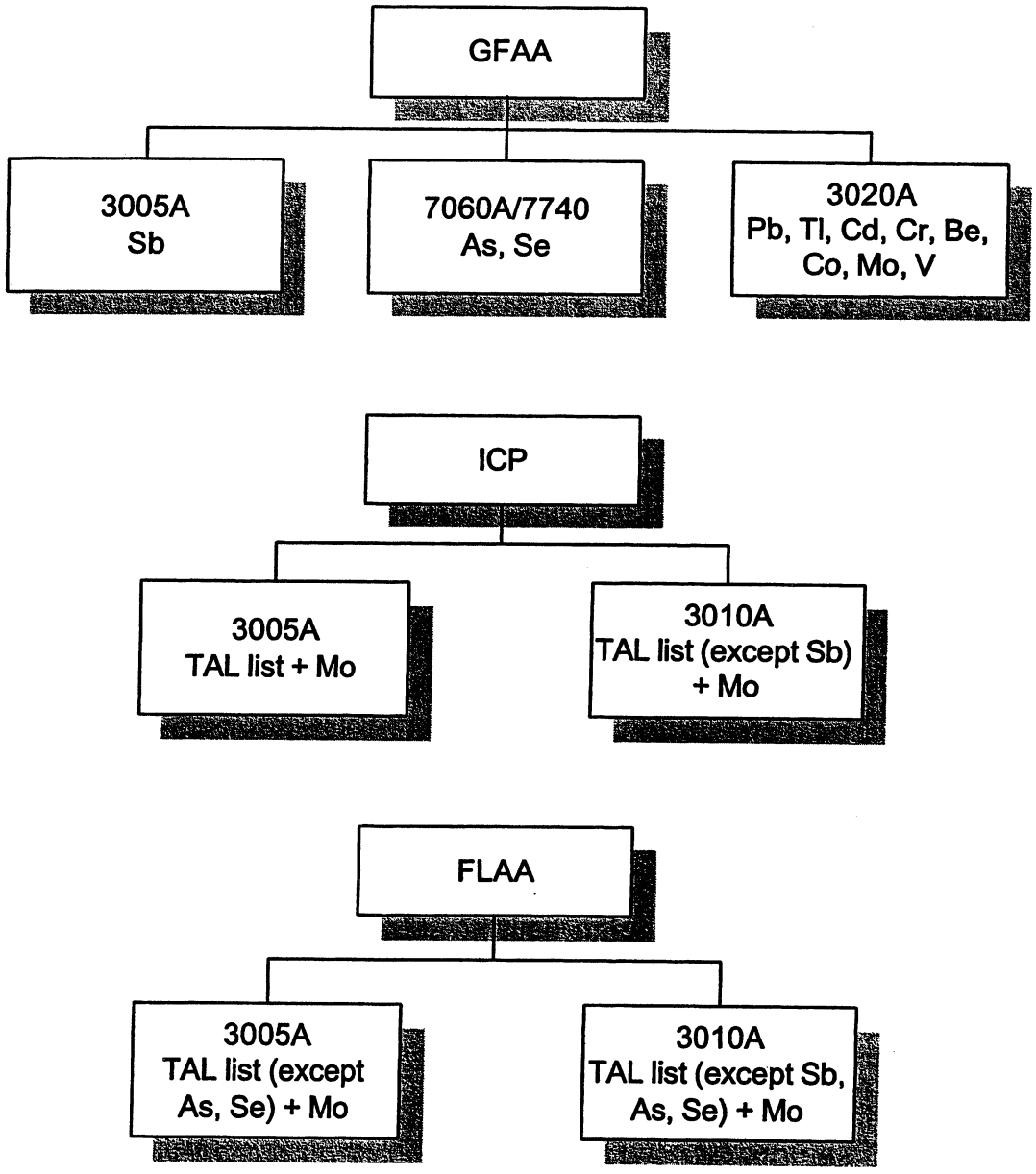
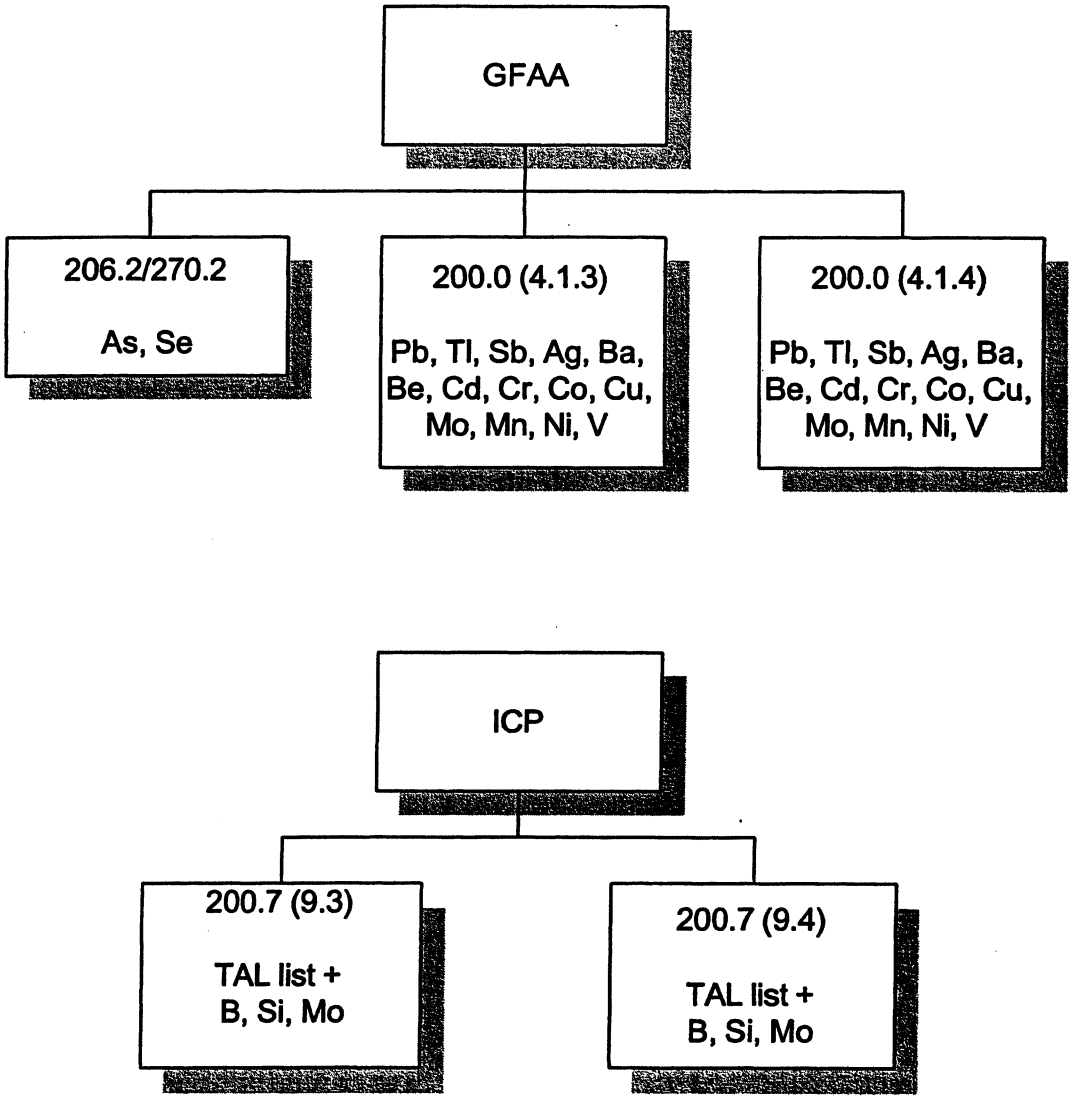


Figure 6. Overview of SW846 Aqueous Preparation Methods by Determinative Method



TAL list: Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn

Figure 7. Overview of MCAWW Aqueous Preparation Methods by Determinative Technique



TAL list: Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn

APPENDIX A

TABLES

TABLE I. Approved Preparation Method Analytes - SW846

ELEMENT	Symbol	CAS Number	3005A	3010A	3020A	7060A 7740
Aluminum	Al	7429-90-5	X	X		
Antimony	Sb	7440-36-0	X			
Arsenic	As	7440-38-2	X	X		X
Barium	Ba	7440-39-3	X	X		
Beryllium	Be	7440-41-7	X	X	X	
Cadmium	Cd	7440-43-9	X	X	X	
Calcium	Ca	7440-70-2	X	X		
Chromium	Cr	7440-47-3	X	X	X	
Cobalt	Co	7440-48-4	X	X	X	
Copper	Cu	7440-50-8	X	X		
Iron	Fe	7439-89-6	X	X		
Lead	Pb	7439-92-1	X	X	X	
Magnesium	Mg	7439-95-4	X	X		
Manganese	Mn	7439-96-5	X	X		
Molybdenum	Mo	7439-98-7	X	X	X	
Nickel	Ni	7440-02-0	X	X		
Potassium	K	7440-09-7	X	X		
Selenium	Se	7782-49-2	X	X		X
Silver	Ag	7440-22-4	X	X		
Sodium	Na	7440-23-5	X	X		
Thallium	Tl	7440-28-0	X	X	X	
Vanadium	V	7440-62-2	X	X	X	
Zinc	Zn	7440-66-6	X	X		

X - Designates that the preparation method is approved for an element

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

TABLE II. Approved Preparation Method Analytes - NPDES

ELEMENT	Symbol	CAS Number	200.7 (9.4)	200.7 (9.3)	200.0 (4.1.4)	200.0 (4.1.3)	206.2 270.2
Aluminum	Al	7429-90-5	X	X			
Antimony	Sb	7440-36-0	X	X	X	X	
Arsenic	As	7440-38-2	X	X			X
Boron	B	7440-42-8	X	X			
Barium	Ba	7440-39-3	X	X	X	X	
Beryllium	Be	7440-41-7	X	X	X	X	
Cadmium	Cd	7440-43-9	X	X	X	X	
Calcium	Ca	7440-70-2	X	X			
Chromium	Cr	7440-47-3	X	X	X	X	
Cobalt	Co	7440-48-4	X	X	X	X	
Copper	Cu	7440-50-8	X	X	X	X	
Iron	Fe	7439-89-6	X	X	X	X	
Lead	Pb	7439-92-1	X	X	X	X	
Magnesium	Mg	7439-95-4	X	X			
Manganese	Mn	7439-96-5	X	X	X	X	
Molybdenum	Mo	7439-98-7	X	X	X	X	
Nickel	Ni	7440-02-0	X	X	X	X	
Potassium	K	7440-09-7	X	X			
Selenium	Se	7782-49-2	X	X			X
Silicon	Si	7631-86-9	X	X			
Silver	Ag	7440-22-4	X	X	X	X	
Sodium	Na	7440-23-5	X	X			
Thallium	Tl	7440-28-0	X	X	X	X	
Vanadium	V	7440-62-2	X	X	X	X	
Zinc	Zn	7440-66-6	X	X			

X - Designates that the preparation method is approved for an element

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

TABLE III. ICP and FLAA Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	Working LCS/MS Standard (mg/L)	Aqueous LCS/ MS Level * (ug/l)
Aluminum	100	2000
Antimony	25	500
Arsenic	100	2000
Barium	100	2000
Beryllium	2.5	50
Cadmium	2.5	50
Calcium	2500	50000
Chromium	10	200
Cobalt	25	500
Copper	12.5	250
Iron	50	1000
Lead	50	500
Magnesium	2500	50000
Manganese	25	500
Molybdenum	50	1000
Nickel	25	500
Phosphorous	500	10000
Potassium	2500	50000
Selenium	100	2000
Silver	2.5	50
Sodium	2500	50000
Thallium	100	2000
Vanadium	25	500
Zinc	25	500
Boron	50	1000
Tin	100	2000
Titanium	50	1000

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 1.0 mL working spike (7.3) to 50 mL of sample.

TABLE IV. GFAA Matrix Spike and Aqueous LCS Spike Levels

ELEMENT	Stock LCS/MS Standard (mg/L)	Working LCS/MS Standard (ug/L)	Aqueous LCS/ MS Level * (ug/l)
Arsenic	400	2000	40
Selenium	400	2000	40
Lead	400	2000	40
Thallium	400	2000	40
Antimony	400	2000	40
Cadmium	40	200	4
Chromium	100	500	10
Silver	50	250	5

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 1.0 mL working spike (7.4) to 50 mL of sample.

TABLE V. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	RL (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)*
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 0.5 mL working spike (7.4) to 50 mL of sample.

TABLE VI. Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: CORP-MT-0001 CORP-MT-0003	Redigest and reanalyze samples associated with the method blank.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: CORP-MT-0001 CORP-MT-0003	Redigest and reanalyze all samples associated with the LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: CORP-MT-0001 CORP-MT-0003	Reprep not required unless preparation error suspected.
Matrix Spike Duplicate	See Matrix Spike	Refer to determinative SOPs: CORP-MT-0001 CORP-MT-0003	See Corrective Action for Matrix Spike.

APPENDIX B

CONTAMINATION CONTROL GUIDELINES

APPENDIX B. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

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Implementation Date: 2/12/01

SOP No. CORP-MT-0001NC
Revision No. 3.2
Revision Date: 01/19/01

STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSES, SW-846 METHOD 6010B AND EPA METHOD 200.7

(SUPERSEDES: REVISION 3.1, REVISION DATE 10/04/00)

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	Corporate Technology	Date

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1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma -Atomic Emission Spectroscopy (ICP-AES) using SW-846 Method 6010B and EPA Method 200.7 . Table I of Appendix A lists the elements appropriate for analysis by Methods 6010B and 200.7. Additional elements may be analyzed under Methods 6010B and 200.7 provided that the method performance criteria presented in Section 13.0 are met.

1.2. ICP analysis provides for the determination of metal concentrations over several orders of magnitude. Detection limits, sensitivity and optimum concentration ranges of the metals will vary with the matrices and instrumentation used. For instance, in comparison to conventional ICP technique, ICP-Trace can achieve detection levels comparable to those determined using the graphite furnace atomic absorption spectroscopy (GFAAS) technique.

1.3. Method 6010B is applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, and TCLP, EP and other leachates/extracts. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators may require digestion of **dissolved samples** and this must be clarified and documented before project initiation. Silver concentrations must be below 2.0 mg/L in aqueous samples and 100 mg/kg in solid matrix samples. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data.

1.4. Method 200.7 is applicable to the determination of dissolved, suspended, total recoverable, and total elements in water, waste water, and solid wastes. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples if the criteria in Section 11.1 are met. Silver concentrations must be below 0.1 mg/L in aqueous samples and 50 mg/kg in solid matrix samples.

1.5. State-specific requirements may take precedence over this SOP for drinking water sample analyses.

1.6. The applicable LIMS method codes are QO (6010B), QM (6010B Trace), AS (200.7), JI (200.7 Trace).

2. SUMMARY OF METHOD

2.1. This method describes a technique for the determination of multi elements in solution using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate actions taken. Alternatively, multivariate calibration methods may be chosen for which point selection for background correction is superfluous since whole spectral regions are processed.

2.2. Refer to the appropriate SOPs for details on sample preparation methods.

3. DEFINITIONS

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.

3.4. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

4. INTERFERENCES

4.1. Spectral, physical and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.
- Stray light from the line emission of high concentration elements.

4.1.1. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.1.2. Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections IECs must be applied to the analyte to remove the effects of these unwanted emissions.

4.1.3. Physical interferences are generally considered to be effects associated with sample transport, nebulization and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, dilution of the sample, use of a peristaltic pump, mass flow controller, use of an internal standard and/or use of a high solids nebulizer can reduce the effect.

4.1.4. Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not significant with the ICP technique but if observed can be minimized by buffering the sample, matrix matching or standard addition procedures.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL North Canton associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.3.1. The following materials are known to be **corrosive**:

sulfuric acid, hydrochloric acid, nitric acid and hydrofluoric acid. (NOTE: sulfuric and hydrofluoric acids are used in cleaning the ICP torch and hydrofluoric acid is also commonly used in air toxics preparations.)

5.3.2. The following materials are known to be **oxidizing agents**:

nitric acid and hydrogen peroxide.

5.3.3. The plasma emits strong UV light and is harmful to vision. **NOTE: AVOID looking directly at the plasma.**

5.3.4. The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Metals digestates can be processed outside of a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported **immediately** to a laboratory supervisor.

5.6. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S Manager and laboratory supervisor for guidance.

6. EQUIPMENT AND SUPPLIES

6.1. Inductively Coupled Plasma Atomic Emission Spectrometer equipped with autosampler and background correction.

6.2. Radio Frequency Generator.

6.3. Argon gas supply, welding grade or equivalent.

6.4. Coolflow or appropriate water cooling device.

6.5. Peristaltic Pump.

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.7. Class A volumetric flasks.

6.8. Autosampler tubes.

7. REAGENTS AND STANDARDS

7.1. Intermediate standards are purchased as custom STL North Canton multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles. Intermediate standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Expiration dates can be extended provided that the acceptance criteria described in laboratory-specific SOPs are met.

7.2. Working calibration and calibration verification solutions may be used for up to 3 months and must be replaced sooner if verification from an independent source indicates a problem. Standards should be prepared in a matrix of 5% hydrochloric and 5% nitric acids. An exception to this is in the event the Trace ICP is utilized without the internal standard. In this case, the standard acid matrix must be matched to the final preparation matrix as listed in Section 11.9.

7.3. Refer to Tables III, IV, IVA, V and VI (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification, interference correction and spiking solutions.

7.4. Concentrated nitric acid (HNO_3), trace metal grade or better.

7.5. Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.6. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding times for metals are six months from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. Soil samples do not require preservation but must be stored at 4°C ± 2° until the time of preparation .

9. QUALITY CONTROL

Table VII (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using either Method 200.7 or Method 6010B, the following requirements must be met.

Instrument Detection Limit (IDL) - The IDL for each analyte must be determined for each analyte wavelength used least each instrument. The IDL must be determined annually. If the instrument is adjusted in anyway that may affect the IDL, the IDL for that instrument must be redetermined. The IDL shall be determined by multiplying by 3, the standard deviation obtained from the analysis of a standard solution (each analyte in reagent water) at a concentration 3x - 5x the previously determined IDL, with seven consecutive measurements. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure performed between the analysis of separate samples). The result of the IDL determination must be below the STL North Canton reporting limit. The CLP IDL procedure can be used for this method.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte prior to the analysis of any client samples. Refer to STL North Canton SOP NC-QA-0021 for details on MDL analysis and criteria.

9.1.2. Linear Range Verification (LR) - The linear range must be determined on at least an annual basis for each analyte wavelength used on each instrument. The linear range is the concentration above which results cannot be reported without dilution of the sample. The standards used to verify the linear range limit must be analyzed during a routine analytical run and must read within 5% of the expected value.

For the **initial** determination of the upper limit of the linear dynamic range (LDR) for each wavelength, determine the signal responses from a minimum of three to five different concentration standards across the estimated range. One standard should be near the upper limit of the estimated range. The concentration measured at the LDR must be no more than 10% less than the expected level extrapolated from lower standards. If the instrument is adjusted in any way that may affect the LRs, new dynamic ranges must be determined. The LR data must be documented and kept on file.

9.1.3. Background Correction Points - To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on

either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the facility-specific instrument operation SOP and ICP instrument manual for specific procedures to be used in setting background correction points.

9.1.4. Inter-element Corrections (IECs) - ICP interelement correction factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be redetermined. When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC then the possibility of contamination should be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., GFAA or ICP-MS). Published wavelength tables (e.g. MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs. Refer to the facility specific instrument operation SOP and instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which results in a false analyte signal greater than $\pm$ the RL as defined in Tables I, IA or II. To determine IECs, run a single element standard at the established linear range. To calculate an IEC, divide the observed concentration of the analyte by the actual concentration of the "interfering element."

Note: Trace ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the Trace as reflected by the ICSA response. Additional spectral interference is present from easily ionizable elements such as potassium and sodium in axial viewing instruments.

9.1.5. Rinse Time Determination - Rinse times must be determined upon initial set-up of an ICP instrument. To determine the appropriate rinse time for a particular ICP system, the linear range verification standard (see 9.1.3) should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in

environmental samples at that level and an excessive rinse time would be required at the linear range level). Until the required rinse time is established, the method recommends a rinse period of at least 60 seconds between samples and standards. If a memory effect is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented and kept on file, if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

9.2. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20x higher than the blank contamination level).

- If the analyte is a common laboratory contaminant (copper, iron, lead (Trace only) or zinc) the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

- For dissolved metals samples which have not been digested, a CCB result is reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.

9.3. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. Aqueous LCS spike levels are provided in Table III (Appendix A). The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

- If any analyte is outside established control limits the system is out of control and corrective action must occur. Unless in-house control limits are established, a control limit of 80 - 120% recovery must be applied.
- In the event that an MS/MSD analysis is not possible a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.
- In the instance where the LCS recovery is greater than 120% and the sample results are < RL, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the report narrative.**
- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.
- For dissolved metals samples which have not been digested, a CCV result is reported as the LCS. The CCV run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCV.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Tables III and VI (Appendix A).

- If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. For both methods 200.7 and 6010B, control limits of 75-125% recovery and 20% RPD or historical acceptance criteria must be

applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported as NC, MSB (i.e., not calculated). If the reporting software does not have the ability to report NC, MSB then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level." Two other narrative notes for metals analyses: Matrix spike/spike duplicate spike recovery/recoveries was/were outside the acceptance limits of some analytes. The acceptable LCS analysis data indicated that the analytical system was operating within control and this condition is most likely due to matrix interference. See the Matrix Spike Report for the affected analytes which will be flagged with N. Matrix spike/spike duplicate relative percent difference (RPD) exceeded the acceptance limits for some analytes. The imprecision may be attributed to sample heterogeneity. See the Matrix Spike Report for the affected analytes, which will be flagged with *.

- If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- For dissolved metals samples which have not been digested, a MS/MSD must be performed per batch of up to 20 samples by spiking two aliquots of the sample at the levels specified in Table III (Appendix A).

9.5. Dilution test – A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. One sample per preparation batch must be processed as a dilution test. The test is performed by running a sample at a 5x (1:4) dilution. Samples identified as field blanks cannot be used for dilution tests. The results of the diluted sample, after correction for dilution, should agree within 10% of the original sample determination when the original sample concentration is greater than 50x the IDL. If the results are not within 10%, the possibility of chemical or physical interference exists.

9.6. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). For analyses conducted under Method 200.7, the ICV result must fall within 5% of the true value for that solution with relative standard deviation <3% from replicate (minimum of two) exposures. For Method 6010B, the ICV must fall

within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the RL from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (See Section 11.10 or 11.13 for required run sequence).

9.7. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV is be a mid-range standard made from a dilution of the calibration standard. The CCV for both methods must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. A CCB is analyzed immediately following each CCV. (See Section 11.10 or 11.13 for required run sequence.) The CCB result must fall within +/- RL from zero. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within 10% of the action limit, reanalysis and recalibration are not required before continuation of the run. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCV or CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. (Refer to Section 11.13 for an illustration of the appropriate rerun sequence).

9.8. Interference Check Analysis (ICSA/ICSAB) - The validity of the interelement correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Table V (Appendix A) for the details of ICSA and ICSAB composition. Custom STL North Canton multielement ICS solutions must be used. All analytes should be spiked into the ICSAB solution, therefore, if a non-routine analyte is required then it should be manually spiked into the ICSAB using a certified ultra high purity single element solution or custom lab-specific mix. If the ICP will display overcorrection as a negative number then the non-routine elements can be controlled from the ICSA as described in section 9.8.3. Elements known to be interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICP runs.

9.8.1. The ICSA and ICSAB solutions must be run at the beginning of the run. (See Section 11.10 or 11.13 for required run sequence.)

9.8.2. The ICSAB results for the interferents must fall within 80 - 120% of the true value. If any ICSAB interferent result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the samples rerun.

9.8.3. ICSA results for the non-interfering elements with reporting limits ≤ 10 ug/L must fall within the STL North Canton guidelines of ± 2x RL from zero. ICSA results for the non-interfering elements with RLs> 10 µg/L must fall within the STL North Canton guidelines of ± 1x RL from zero. If the ICSA results for the non-interfering elements do not fall within +/- 2x RL (RL ≤10) or ± 1xRL (RL>10) from zero the field sample data must be evaluated as follows:

- If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.
- If the affected element was not required then the sample data can be accepted.
- If the interfering elements are not present in the field sample at a concentration which would result in a false positive or negative result greater than +/- 2x RL from zero then the field sample data can be accepted.
- If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than ± 2x RL from zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10x the analyte signal in the ICSA.
- If the data does not meet the above conditions then the IECs must be re-evaluated and corrected if necessary and the affected samples reanalyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually the calculations must be clearly documented on the raw data.

9.9. CRI - To verify linearity near the CRDL for ICP analysis, a CRI standard is run at the beginning of each sample analysis run but not before the ICV. (See Section 11.10 or 11.13 for required run sequence).

Note: The custom STL North Canton CRI mix contains most analytes at a level two times the standard lab reporting limit.

9.10. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Section 11.17 for additional information on when MSA is required as well as Appendix D for specific MSA requirements.

9.11. Quality Assurance/Project Summaries - Certain clients may require project- or program-specific QC which may supersede this SOP requirements. Quality Assurance Summaries (QASs) or equivalent documents providing project-specific requirements should be developed so that project staff clearly understand the special project requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.2. Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures. Flush the system with the calibration blank between each standard or as the manufacturer recommends. The calibration curve must consist of a minimum of a blank and a standard. Refer to the facility-specific instrument SOP or ICP instrument manual for a detailed set up and operation protocols.

10.3. Calibration must be performed daily and each time the instrument is set up. Instrument runs may be continued over periods exceeding 24 hours as long as all calibration verification (CCV) and interference check QC criteria are met. The instrument standardization date and time must be included in the raw data.

10.4. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corresponding corrective actions.

11. PROCEDURE

11.1. For 200.7 analyses, dissolved (preserved) samples must be digested unless it can be documented that the sample meets all of the following criteria:

A. Visibly transparent with a turbidity measurement of 1 NTU or less.

B. Is of one liquid phase and free of particulate or suspended matter following acidification.

C. Is NOT being analyzed for silver.

11.2. A minimum of two exposures for each standard, field sample and QC sample is required. The average of the exposures is reported. For Trace ICP analyses, the results of the sum channel must be used for reporting.

11.3. Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in 9.1.6 it can be demonstrated that a shorter rinse time may be used. Triton-X can be added to the rinse solution to facilitate the rinse process.

11.4. The use of an autosampler for all runs is strongly recommended.

11.5. The use of automated QC checks through the instrument software is highly recommended for all calibration verification samples (ICV,CCV), blanks (ICB,CCB,PB), interference checks (ICSA,ICSAB) and field samples (linear range) to improve the data review process.

11.6. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.7. To facilitate the data review and reporting processes it is strongly recommended that all necessary dilutions be performed before closing out the instrument run.

11.8. The use of an internal standard is recommended on the conventional, non-Trace ICPs as an alternative to using the method of standard additions. This technique is useful in overcoming matrix interferences especially in high solids matrices. However, for conventional ICP techniques, internal standards may not be necessary provided that one of the following is performed to minimize physical interferences: (1) peristaltic pump is used, (2) high solids nebulizer is used, or (3) high solids samples are diluted and reanalyzed.

11.9. The use of an internal standard is **required** on the Trace ICP unless the calibration and QC standards are matrix matched to each digestion procedure used as follows:

Preparation Method	% HNO_3	% HCl
CLP Aqueous	1	5
CLP Soil	5	2.5
SW846 3050	10	10
SW846 3005	2	5
SW846 3010	6	5

The following procedural guidelines must be followed when using an internal standard:

11.9.1. Typically used internal standards are: yttrium or scandium. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)

11.9.2. The internal standard (IS) must be added to every sample and standard at the same concentration. It is recommended that the IS be added to each analytical sample automatically through use of a third pump channel and mixing coil. Internal standards should be added to blanks, samples and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

11.9.3. The concentration of the internal standard should be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.

11.9.4. The internal standard raw intensity counts must be printed on the raw data.

11.9.5. The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte). The instrument automatically adjusts sample results based on comparison of the internal standard intensity in the sample to the internal standard intensity at calibration.

11.9.5.1. If the internal standard counts fall within ±30% of the counts observed in the ICB then the data is acceptable.

11.9.5.2. If the internal standard counts in the field samples are more than ±30% higher than the expected level, the field samples must then be:

(1) Diluted and reanalyzed;

(2) The IS concentrations must be raised; or

(3) A different internal standard must be used.

11.10. The following analytical sequence must be used for Methods 6010B and 200.7:

Instrument Calibration
ICV
ICB
CRI
ICSA
ICSAB
7 samples
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table VII (Appendix A) for Method 6010B and 200.7 quality control criteria.

11.11. Additional quality control analyses are necessary for analysis under the Contract Laboratory Program (CLP). If these are included then CLP, 6010 and 200.7 samples can be included in the same sequence. Refer to CORP-MT-0002NC for details.

11.12. Full method required QC must be available for each wavelength used in determining reported analyte results.

11.13. The following run sequence provides an illustration of a mid-run CCV or CCB failure and the appropriate corrective action run sequence as described in Section 9.7:

Original Run: Instrument Calibration

ICV

ICB

CRI

ICSA

ICSAB

7 samples

CCV1

CCB1

10 samples

CCV2

CCB2

10 samples **

CCV3 *

CCB3 *

10 samples **

CCV4

CCB4

10 samples

CCV5

CCB5

* Failure occurs at CCV3/CCB3

**Samples requiring rerun for affected analytes

Reanalysis:	Recalibrate
	ICV
	ICB
	CRI
	ICSA
	ICSAB
	CCV2
	CCB2
	10 samples
	CCV3
	CCB3
	10 samples
	CCV4
	CCB4

Notes:

Samples between CCV4 and CCV5 do not require reanalysis as they were bracketed by compliant QC samples.

See CORP-MT-0002NC for the appropriate reanalysis sequence if CLP requirements must also be met.

11.14. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.15. All measurements must fall within the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that

exceed the linear range or use an alternate wavelength for which QC data are established. If an interelement correction exists for an analyte which exceeds the linear range, the IEC may be inaccurately applied. Therefore, even if an overrange analyte may not be required to be reported for a sample, if that analyte is a interferent for any requested analyte in that sample, the sample must be diluted. Acid strength must be maintained in the dilution of samples.

11.16. For TCLP samples, full four-point MSA will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

The reporting and regulatory limits for TCLP analyses as well as matrix spike levels are detailed in Table VI (Appendix A). Appendix E provides guidance on performing MSA analyses.

11.17. Any variation in procedure shall be completely documented using instrument run logs, maintenance logs, report narratives, a Nonconformance Memo, or an anomaly report and is approved by a Supervisor/Group Leader and QA Manager. If contractually required, the client shall be notified by the Project Manager.

11.18. Nonconformance documentation shall be filed in the project file.

11.19. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%\,R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.6. The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg/Kg, dry\ weight = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. The dilution test percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where:
I = Sample result (Instrument reading)
S = Dilution test result (Instrument reading × 5)

12.9. Appropriate factors must be applied to sample values if dilutions are performed.

12.10. Sample results should be reported with up to three significant figures in accordance with the STL North Canton significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Refer to Tables I, IA & II in Appendix A for the list of Method 6010B and 200.7 analytes as well as additional analytes that may be analyzed using this SOP.

13.3. Method performance is determined by the analysis of MS and MSD samples as well as method blanks and laboratory control samples. The MS or MSD recovery should fall within +/- 25 % and the MS/MSD should compare within 20% RPD or within the laboratory's historical acceptance limits. These criteria apply to analyte concentrations

greater than or equal to 10xIDL. Method blanks must meet the criteria specified in Section 9.2. The laboratory control samples should recover within 20% of the true value or within the laboratory's historical acceptance limits.

13.4. Training Qualification:

The group/team leader or the supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed of according to the facility hazardous waste procedures and per the local, state, and federal regulations. The Environmental Health and Safety Director should be contacted, if additional information is required.

15.2. Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

16. REFERENCES

16.1. 40 CFR Part 136, Appendix B, 7-5-95, Determination of Method Detection Limits.

16.2. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996. Method 6010B.

16.3. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4, May 1994. Method 200.7.

16.4. CORP-MT-0002NC, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Method 200.7 & CLP-M, SOW ILMO3.0 and ILMO4.0).

16.5. QA-003, STL North Canton QC Program.

16.6. QA-004, Rounding and Significant Figures.

16.7. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference method

17.1.1. Modifications/interpretations from both Methods 6010B and 200.7.

17.1.1.1. STL North Canton laboratories use mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in house as recommended by the subject methods.

17.1.1.2. Methods 200.7 and 6010B state that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. In determining IECs, because of lack of definition clarification for "concentration range around the calibration blank," STL North Canton has adopted the procedure in EPA CLP ILMO4.0.

17.1.1.3. Section 8.5 of Method 6010B and Section 9.5 of Method 200.7 recommend that whenever a new or unusual matrix is encountered, a series of tests be performed prior to reporting concentration data for that analyte. The dilution test helps determine if a chemical or physical interference exists. Because STL North Canton laboratories receive no prior information from clients regarding when to expect a new or unusual matrix, STL North Canton may select to perform a dilution test on one sample in each prep batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. At STL North Canton labs, matrix interference is determined by evaluating data for the LCS and MS/MSD. STL North Canton requires documented, clear guidance when a new or unusual matrix will be received for a project and a request to perform the dilution test or PDS on a client-identified sample.

17.1.2. Modifications from Method 200.7.

17.1.2.1. Method 200.7 defines the IDL as the concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of the calibration blank signal at the

same wavelength. STL North Canton labs utilize the CLP IDL definition as defined in Section 9.1.1 of this SOP.

17.1.2.2. The calibration blank is prepared in an acid matrix of 5% HNO_3/5% HCl instead of the specified 2% HNO_3/10% HCl matrix as the former matrix provides for improved performance relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied.

17.1.2.3. Method section 9.3.4 specifies that "Analysis of the IPC (ICSA/AB) solution immediately following calibration must verify that the instrument is within ± 5% of calibration with a relative standard deviation <3% from replicate integrations ≥ 4." STL North Canton uses a minimum of two exposures.

17.1.2.4. Section 7.12 of 200.7 indicates that the QCS (ICV) should be prepared at a concentration near 1 ppm. The ICV specified in this SOP accommodates the 1 ppm criteria for the majority of analytes. For the remaining analytes, this SOP specifies ICV concentrations which are appropriate to the range of calibration. The intent of the ICV, verification of calibration standard accuracy, is independent of the ICV concentration used.

17.1.2.5. The ICS criteria applied by this SOP differ from those stated in the method. Method 200.7 section 10.4 states that results should fall within the established control limits of 3 times the standard deviation of the calibration blank for that analyte. The control limits listed in this SOP are those applicable to the EPA designed solution.

17.1.2.6. Method 200.7 section 9.3.4 states the CCB should be less than the IDL, but > the lower 3-sigma control limit of the calibration blank. The intent of this requirement is to ensure that the calibration is not drifting at the low end. STL North Canton has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. SOP section 9.7 provides the detailed corrective action criteria that must be followed.

17.1.3. Modifications from Method 6010B.

17.1.3.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the

reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

17.1.3.2. Method 6010B section 8.6.1.3 states that the results of the calibration blank are to agree within 3x the IDL. If not, repeat the analysis two or more times and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. The intent of this requirement is to ensure that the calibration is not drifting at the low end. STL North Canton has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. See SOP Section 9.7 for a detailed description of the required corrective action procedures.

17.2. Modifications from previous SOP

Refer to revision 2 of this SOP.

17.3. Facility-Specific SOPs

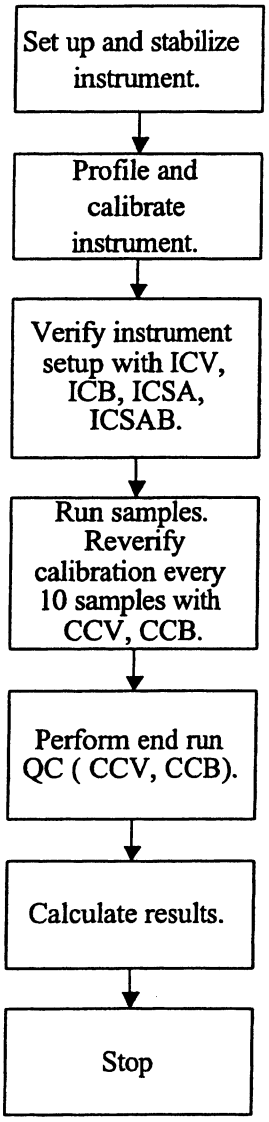
Each facility shall review and revise as appropriate this SOP to reflect any facility-specific requirements. If no facility-specific amendments are required, the SOP can be adopted as is.

17.4. Documentation and Record Management

The following documentation comprises a complete ICP raw data package:

- Raw data (direct instrument printout).
- Relevant sample preparation benchsheets.
- Run log printout from instrument software where this option is available (TJA) or manually generated run log (i.e., Ward WSL printout).
- Data review checklist - See Appendix B.
- Standards documentation (including prep and expiration dates, source, and lot #).
- Nonconformance/anomaly documentation (if applicable).

17.5. Flow Diagram



APPENDIX A

TABLES

TABLE I. Method 200.7 and 6010B Target Analyte List

ELEMENT	Symbol	CAS #	6010B analyte	200.7 analyte	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Aluminum	Al	7429-90-5	X	X	200	20
Antimony	Sb	7440-36-0	X	X	60	6
Arsenic	As	7440-38-2	X	X	300	30
Barium	Ba	7440-39-3	X	X	200	20
Beryllium	Be	7440-41-7	X	X	5.0	0.5
Boron	B	7440-42-8		X	200	20
Cadmium	Cd	7440-43-9	X	X	5.0	0.5
Calcium	Ca	7440-70-2	X	X	5000	500
Chromium	Cr	7440-47-3	X	X	10	1
Cobalt	Co	7440-48-4	X	X	50	5
Copper	Cu	7440-50-8	X	X	25	2.5
Iron	Fe	7439-89-6	X	X	100	10
Lead	Pb	7439-92-1	X	X	100	10
Magnesium	Mg	7439-95-4	X	X	5000	500
Manganese	Mn	7439-96-5	X	X	15	1.5
Molybdenum	Mo	7439-98-7	X	X	40	4
Nickel	Ni	7440-02-0	X	X	40	4
Potassium	K	7440-09-7	X	X	5000	500
Selenium	Se	7782-49-2	X	X	250	25
Silver	Ag	7440-22-4	X	X	10	1
Sodium	Na	7440-23-5	X	X	5000	500
Thallium	Tl	7440-28-0	X	X	2000	200
Vanadium	V	7440-62-2	X	X	50	5
Zinc	Zn	7440-66-6	X	X	20	2

TABLE IA. Method 200.7 and 6010B Trace ICP Target Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Arsenic	As	7440-38-2	10	1.0
Lead	Pb	7439-92-1	3.0	0.3
Selenium	Se	7782-49-2	5.0	0.5
Thallium	Tl	7440-28-0	10	1.0
Antimony	Sb	7440-36-0	10	1.0
Cadmium	Cd	7440-43-9	2.0	0.2
Silver	Ag	7440-22-4	5.0	0.5
Chromium	Cr	7440-47-3	5.0	0.5

TABLE II. Non-Routine Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Tin	Sn	7440-31-5	100	10
Titanium	Ti	7440-32-6	50	5

TABLE III. Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	LCS Level (ug/L)	Matrix Spike Level (ug/L)
Aluminum	2000	2000
Antimony	500	500
Arsenic	2000	2000
Barium	2000	2000
Beryllium	50	50
Cadmium	50	50
Calcium	50000	50000
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1000	1000
Lead	500	500
Magnesium	50000	50000
Manganese	500	500
Molybdenum	1000	1000
Nickel	500	500
Potassium	50000	50000
Selenium	2000	2000
Silver	50	50
Sodium	50000	50000
Thallium	2000	2000
Vanadium	500	500
Zinc	500	500
Boron	1000	1000
Tin	2000	2000
Titanium	1000	1000

TABLE IV. ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	100000	200	25000	50000
Antimony	10000	60	1000	5000
Arsenic	10000	300	1000	5000
Barium	10000	200	1000	5000
Beryllium	10000	5	1000	5000
Cadmium	10000	5	1000	5000
Calcium	100000	5000	25000	50000
Chromium	10000	10	1000	5000
Cobalt	10000	50	1000	5000
Copper	10000	25	1000	5000
Iron	100000	100	25000	50000
Lead	10000	100	1000	5000
Magnesium	100000	5000	25000	50000
Manganese	10000	15	1000	5000
Molybdenum	10000	40	1000	5000
Nickel	10000	40	1000	5000
Potassium	100000	5000	25000	50000
Selenium	10000	250	1000	5000
Silver	2000	10	500	1000
Sodium	100000	5000	25000	50000
Thallium	20000	2000	5000	10000
Vanadium	10000	50	1000	5000
Zinc	10000	20	1000	5000
Boron	10000	200	1000	5000
Tin	10000	100	1000	5000
Titanium	10000	50	1000	5000

TABLE IVA. Trace ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	50000	200	12500	25000
Antimony	1000	10	250	500
Arsenic	1000	10	250	500
Barium	4000	10	1000	2000
Beryllium	4000	5	1000	2000
Cadmium	1000	2	250	500
Calcium	100000	5000	25000	50000
Chromium	4000	5	1000	2000
Cobalt	4000	50	1000	2000
Copper	4000	25	1000	2000
Iron	50000	100	12500	25000
Lead	1000	3	250	500
Magnesium	100000	5000	25000	50000
Manganese	4000	15	1000	2000
Molybdenum	4000	40	1000	2000
Nickel	4000	40	1000	2000
Potassium	100000	5000	25000	50000
Selenium	1000	5	250	500
Silver	2000	5	500	1000
Sodium	100000	5000	25000	50000
Thallium	2000	10	500	1000
Vanadium	4000	50	1000	2000
Zinc	4000	20	1000	2000

TABLE V. Interference Check Sample Concentrations*

Element	ICSA (ug/L)	ICSAB (ug/L)
Aluminum	500000	500000
Antimony	-	1000
Arsenic	-	1000
Barium	-	500
Beryllium	-	500
Cadmium	-	1000
Calcium	500000	500000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200000	200000
Lead	-	1000
Magnesium	500000	500000
Manganese	-	500
Molybdenum	-	1000
Nickel	-	1000
Potassium	-	10000
Selenium	-	1000
Silver	-	1000
Sodium	-	10000
Thallium	-	10000**
Vanadium	-	500
Zinc	-	1000
Tin	-	1000
Boron		1000
Titanium		1000

* Custom STL North Canton solutions contain analytes common to all STL North Canton facilities. Non-routine elements not listed above should be spiked into the ICSAB at 1000 ug/L.

** Thallium level for Trace ICP should be at 1000 ug/L.

TABLE VI. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	Reporting Level (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

TABLE VII. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Two-point Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met		Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
ICV	Beginning of every analytical run.	Method 200.7: 95 - 105 % recovery. RSD dupl. exp < 3% Method 6010B: 90 - 110 % recovery. RSD dupl. exp < 5%	Terminate analysis; Correct the problem; Recalibrate.
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate.
CCV	Every 10 samples and at the end of the run.	Method 200.7 & 6010B: 90 - 110 % recovery. RSD dupl. exp < 5%	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
ICSA	Beginning of every run	See Section 9.8.3	See Section 9.8.3
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.8.2.

* See Sections 11.10 and 11.13 for exact run sequence to be followed.

TABLE VII. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Dilution Test	One per prep batch.	For samples > 50x IDL, dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference per client request.
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Common lab contaminants may be accepted up to 2x the RL after consultation with the client (See 9.2). Sample results greater than 10x the blank concentration are acceptable. Samples for which the contaminant is < RL may not require redigestion or reanalysis (see Section 9.2).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.2 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits. Samples for which the contaminant is < RL and the LCS results are > 120% may not require redigestion or reanalysis (see Section 9.3)	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.

TABLE VII. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Matrix Spike	One per sample preparation batch of up to 20 samples.	75 - 125 % recovery or in-house control limits. For TCLP See Section 11.17.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. For TCLP see Section 11.17.
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery; RPD ≤ 20% .	See Corrective Action for Matrix Spike.

APPENDIX B

STL NORTH CANTON ICP DATA REVIEW CHECKLIST

(EXAMPLE)

SCAL STD_____ ICSA STD_____ SCAL2 STD_____ ICSAB STD_____

CRI STD_____ ICV STD_____ CLP CRI STD_____ CCV STD_____

(Example)

STL North Canton ICP Data Review Checklist

Run/Project Information:

Run Date: _____ **Analyst:**_____ **Instrument:**_____

Prep Batches Run:_____

Cu/MnRatio:_____

Circle Methods used: **6010B / 200.7: CORP-MT-0001 Rev 2 CLP ILM03.0: CORP-MT-0002 Rev 1**

6010A/200.7: CORP-MT-0001NC, Rev 2

Review Items

A. Calibration/Instrument Run QC	**Yes**	**No**	**N/A**	**2nd Level**
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits ? (*ICV:* 6010B, CLP = 90 - 110%, 200.7 = 95 -105%) *(CCV: 90 - 110%)*				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP) ?				
4. High Std. (HCAL) reanalyzed before samples and recovered within QC limits ? (6010A/200.7 95-105%, CLP- N/A)				
5. CRI run and recovered within QC limits ? (+/- 50% for non-CLP - 6010A only)				
6. ICSA/ICSAB run at required frequency and within SOP limits ?				
B. Sample Results				
1. Were samples with concentrations > the linear range for any parameter diluted and reanalyzed ?				
2. All reported results bracketed by in control QC ?				
3. Sample analyses done within holding time ?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				

3. MS/MSD run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits ?				
5. Serial dilution done per prep batch (or per SDG for CLP) ?				
6. Post digest spike analyzed if required (CLP only) ?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/LR/IEC data on file ?				
3. Calculations checked for error ?				
4. Transcriptions checked for error ?				
5. All client/project specific requirements met ?				
6. Date/time of analysis verified as correct ?				

Level I Analyst:_____ **Date:**_____ **Time:**_____

Comments:_____

Level II Reviewer:_____ **Date:**_____ **Time:**_____

APPENDIX C

CROSS REFERENCE OF TERMS USED IN METHODS 6010B, 200.7, AND BY STL NORTH CANTON

CROSS REFERENCE OF TERMS COMMONLY USED IN METHODS EPA 200.7, SW6010B, AND STL NORTH CANTON INC. SOP

EPA 200.7	SW6010B	STL North Canton Inc. SOP
Calibration blank (CB)	Calibration blank	Initial and continuing calibration blanks (ICB/CCB)
Dilution test	Dilution test	Dilution Test
Instrument detection limit (IDL)	Instrument detection limit (IDL)	Instrument detection limit (IDL)
Instrument performance check (IPC)	Continuing calibration verification (CCV)	Continuing calibration verification (CCV)
Internal standard	Internal standard	Internal standard (IS)
Laboratory duplicates	n/a	n/a
Laboratory fortified blank (LFB)	n/a	Laboratory control sample (LCS)
Laboratory fortified sample matrix (LFM)	Matrix spike and matrix spike duplicate (MS/MSD)	Matrix spike and matrix spike duplicate (MS/MSD)
Laboratory reagent blank (LRB)	Method blank	Method or Prep blank (MB)
Linear dynamic range (LDR)	Linear dynamic range (LDR)	Linear dynamic range (LDR)
Method detection limit (MDL)	Method detection limit (MDL)	Method detection limit (MDL)
Quality control sample (QCS)	Check standard or Initial calibration verification (ICV)	Initial calibration verification (ICV)
Spectral interference check solution (SIC)	Interference check solution (ICS)	Interference check solution (ICSA/ICSAB)

APPENDIX D

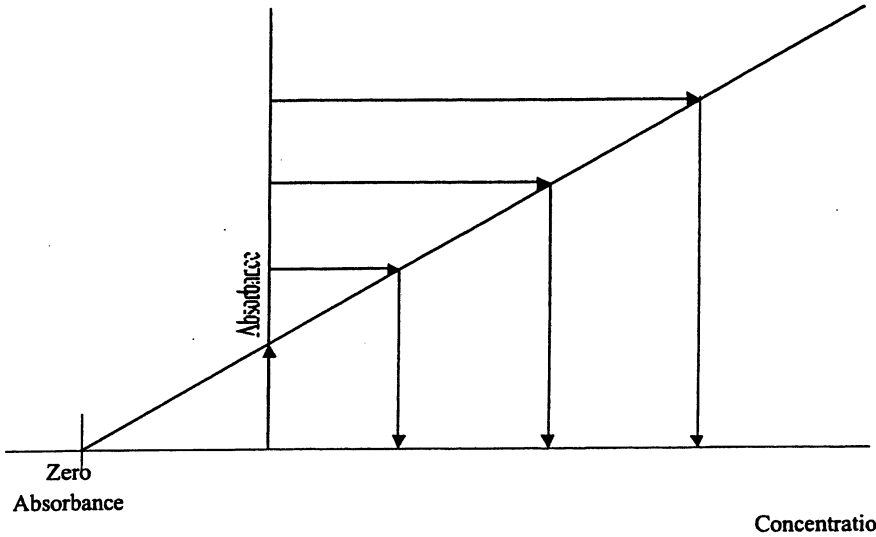
MSA GUIDANCE

Appendix D. MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown.



Conc. of Sample	Addn 0 No Addn	Addn 1 Addn of 50% of Expected Amount	Addn 2 Addn of 100% of Expected Amount	Addn 3 Addn of 150% of Expected Amount

For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.

- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX E

TROUBLESHOOTING GUIDE

APPENDIX E. TROUBLESHOOTING GUIDE

Problem	Possible Cause/ Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer Clean or replace mixing chamber Lower Torch
Instrument Drift	RF not cooling properly Vacuum level is too low Replace torch (Crack) Clean or replace nebulizer (blockage) Check room temperature (changing) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Adjust sample carrier gas Reprofile Horizontal Mirror Replace PA tube
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage(back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Cu/Mn Ratio Outside Limits or Low Sensitivity	Plasma conditions changed Clean nebulizer, torch or spray chamber Replace tubing (clogged) Realign torch Check IECs
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

APPENDIX F

CONTAMINATION CONTROL GUIDELINES

APPENDIX F. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipet tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Latex gloves contain over 500 ppb of zinc.

APPENDIX G

PREVENTIVE MAINTENANCE

APPENDIX G. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Daily	Change sample pump tubing and pump windings Check argon gas supply level Check rinse solution and fill if needed Check waste containers and empty if needed Check sample capillary tubing is clean and in good condition Check droplet size to verify nebulizer is not clogged. Check sample flow for cross flow nebulizer Check Cu/Mn ratio-should be 30% of value at date that IECs were performed Check pressure for vacuum systems
As Needed	Clean plasma torch assembly to remove accumulated deposits Clean nebulizer and drain chamber; keep free-flowing to maintain optimum performance Replace peristaltic pump tubing, sample capillary tubing and autosampler sipper probe
Weekly	Apply silicon spray on autosampler tracks Check water level in coolflow
Monthly	Clean air filters on back of power unit to remove dust Check D mirror for air instruments
Bi-yearly	Change oil for vacuum systems Replace coolant water filter (may require more or less frequently depending on quality of cooling water)

APPENDIX H

ICP OPERATING INSTRUCTIONS

ICP Analysis (TJA 61E)

(Example)

1. SETUP

a. Plasma Control Panel (enter)

b. (F1)-Startup

c. (F9)-Continue

d. (F2)-Levels

1. Change auxiliary gas to low – use space bar to toggle
2. Change nebulizer gas flow to 0.5 L/min.
3. Change pump rate to 130
4. Esc
5. Allow instrument to warm up approximately 30 minutes.

2. DEVELOPMENT

a. Methods (enter)

b. Enter method name

c. (F3)-Method Info.

d. Change file name

e. (F9)-Done

f. (F9)-Done/Keep

3. OPERATION

a. Analysis (enter)

b. (F5)-Profile

1. (F3)-Automatic

2. (F1)-Run

3. If peak position is greater than +/- .05 units from the center peak position, you must adjust the profile. If it is within +/- .05 units, press (F9)-done.

4. To adjust select (F1)-CalcSS and enter current vernier position. (enter)

5. Adjust to new vernier position(F9)-done

6. Rerun profile until peak position is +/- .05 units.

7. (F9)-Done

c. Autosampler (F9)

1. Enter method name (enter)

2. Enter autosampler table name (enter)

3. (F1)-Run

STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS SAMPLES BY COLD VAPOR ATOMIC ABSORPTION, SW846 7470A AND MCAWW 245.1

(SUPERSEDES: REVISION 2.2, DATED 02/05/01)

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7470A and MCAWW Method 245.1.

1.2. The associated LIMs method codes are BL (Method 245.1) and O8 (Method 7470A).

1.3. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants, potassium permanganate and potassium persulfate, has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation and volume of sample used.

1.4. Method 7470A is applicable to the preparation and analysis of mercury in ground water, aqueous samples, TCLP, and other leachates/extracts. Certain solid and sludge type wastes may also be analyzed, however Method 7471A (see CORP-MT-0007NC) is usually the method of choice. All matrices require sample preparation prior to analysis.

1.5. Method 245.1 is applicable to the determination of mercury in drinking, surface and saline waters and domestic and industrial wastes. All matrices require sample preparation prior to analysis.

1.6. The STL North Canton reporting limit for mercury in aqueous matrices is 0.0002 mg/L except for TCLP or SPLP leachates for which the reporting limit is 0.002 mg/L.

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the

sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3. DEFINITIONS

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following digestion.

4. INTERFERENCES

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1. Potassium permanganate, which is used to breakdown organic mercury compounds also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.2. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.3. Chlorides can cause a positive interference. Sea waters, brines and industrial effluents high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample headspace before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.4. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This

is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.5. Samples containing high concentrations of oxidizing organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

4.6. The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL North Canton associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. The following materials are known to be **corrosive:**

hydrochloric acid, nitric acid and sulfuric acid.

5.3.2. The following materials are known to be **oxidizing agents**:

nitric acid, potassium permanganate, potassium persulfate and magnesium perchlorate.

5.3.3. Mercury is a highly toxic element that must be handled with care. The analyst must be aware of the handling and clean up techniques before working with mercury.

Since mercury vapor is toxic, precaution must be taken to avoid its inhalation, ingestion or absorption through skin. All lines should be checked for leakage and the mercury vapor must be vented into a hood or passed through a mercury absorbing media such as:

5.3.3.1. Equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4, or

5.3.3.2. Iodine, 0.25%, in a 3% KI solution.

5.3.4. Magnesium sulfate is known to be a reproductive toxin (mutagen).

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported **immediately** to a laboratory supervisor.

5.6. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.

5.7. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders are located outside the laboratory and the gas led to the instrument through approved lines.

5.8. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

6. EQUIPMENT AND SUPPLIES

6.1. Temperature controlled water bath (capable of maintaining a temperature of 90-95 °C) or autoclave that is able to obtain conditions of 15 lbs., 120 °C for 15 minutes.

6.2. Atomic Absorption Spectrophotometer equipped with:

6.2.1. Absorption Cell with quartz end windows perpendicular to the longitudinal axis. Dimensions of the cell must result in sufficient sensitivity to meet the SOP defined reporting limit. The quartz windows must be maintained to provide accurate

measurements. Any scratches or fingerprints can alter the absorption of UV radiation.

6.2.2. Mercury specific hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL).

6.2.3. Peristaltic pump, which can deliver 1 L/min, air.

6.2.4. Flowmeter capable of measuring an airflow of 1 L/min.

6.2.5. Recorder or Printer.

6.2.6. Aeration Tubing: A straight glass frit having a course porosity and Tygon tubing is used for the transfer of mercury vapor from the sample bottle to the absorption cell and return.

6.2.7. Drying device (a drying tube containing magnesium perchlorate or magnesium sulfate and/or a lamp with a 60 W bulb) to prevent condensation in cell. The lamp is positioned to shine on the absorption cell maintaining the air temperature in the cell about 10 °C above room temperature. Other drying devices that achieve the same purpose are also acceptable (i.e., Gortex filter).

NOTE: Instruments designed specifically for the measurement of mercury using the cold vapor technique may be substituted for the atomic absorption spectrophotometer.

6.3. BOD bottles or equivalent.

6.4. Nitrogen or argon gas supply, welding grade, or equivalent.

6.5. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.6. Class A volumetric flasks.

6.7. Thermometer (capable of accurate readings at 95 °C).

6.8. Disposable cups or tubes.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Stock (10 ppm) mercury standards (in 10% HNO_3) are purchased as custom STL North Canton solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Working mercury standard (0.1 ppm): Take 1 mL of the stock mercury standard (7.2) and dilute to 100 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO_3. This acid (150 uL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.4. The calibration standards listed in Table I must be prepared fresh daily from the working standard (7.3) by transferring 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working mercury standard into 100 mL flasks and diluting to volume with reagent water.

Note: Alternate approaches to standard preparation may be taken and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained. For example, automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

7.5. The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.

7.6. Refer to Table I (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All standards must be processed through the entire analytical procedure including sample preparation.

7.7. Nitric acid (HNO_3), concentrated, trace metal grade or better.

Note: If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

7.8. Sulfuric acid (H_2SO_4), concentrated, trace metal grade or better.

7.9. Stannous sulfate solution: Add 25 g of stannous sulfate to 250 mL of 0.5 N sulfuric acid. This mixture is a suspension and should appear cloudy. This solution should be made daily and should be stirred continuously during use.

Note: Stannous chloride may be used in place of stannous sulfate. Prepare the stannous chloride solution according to the recommendations provided by the instrument manufacturer.

7.10. Sodium chloride-hydroxylamine hydrochloride solution: Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride to every 100 mL of reagent water.

Note: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.11. Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate for every 100 mL of reagent water.

7.12. Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate for every 100 mL of reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for mercury is 28 days from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. Refrigeration is not required. Preservation must be verified prior to analysis.

9. QUALITY CONTROL

Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to the analysis of any analyte using 7470A or the 245.1, the following requirements must be met.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined

on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the STL North Canton reporting limit.

9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (Method Blanks, LCS, and MS/MSDs) are not included in the sample count for determining the size of a preparation batch.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit or at or above 10% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20 times higher than the blank contamination level).

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

9.5. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. On going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The LCS must be carried through the entire analytical procedure. If the LCS is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, a control limit of 80 - 120% recovery must be applied.

- In the instance where the LCS recovery is > 120% and the sample results are < RL, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the case narrative.

- In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table I (Appendix A).

- If analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 75 - 125 % recovery and 20% RPD must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation

and reanalysis of the batch. MS/MSD results, which fall outside the control limits, must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.7. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV result must fall within 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. (See Section 11.2.11 and Section 11.2.12 for required run sequence). If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include repreparation of the ICV, ICB, CRA, CCV, and CCB with the calibration curve.

9.8. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV must be a mid-range standard at a concentration other than that of the ICV. The CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. (See Section 11.2.11 and 11.2.12 for required run sequence.) The CCB result must fall within +/- RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs.

9.9. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the sample prior to preparation. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences, which cause a baseline shift. Refer to Section 11.2.13 for additional information on when full 4 point MSA is required as well as Appendix C for specific MSA requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Calibration standards must be processed through the preparation procedure as described in Section 11.1.

10.2. Due to the differences in preparation protocols separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.

10.3. Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.4. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility specific instrument SOP and CVAA instrument manual for detailed setup and operation protocols.

10.5. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at the STL North Canton reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7.5 and Table I for additional information on preparing calibration standards and calibration levels.

10.6. The calibration curve must have a correlation coefficient of ≥ 0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results can not be reported from a curve with an unacceptable correlation coefficient.

10.7. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corrective actions.

11. PROCEDURE

11.1. Sample Preparation:

11.1.1. All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed through the digestion procedure as well as the field samples. Transfer 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working standard (7.3) into a series of 100 ml class A volumetrics, then dilute to volume . For the ICV, use a 2.5 ml aliquot of the working standard. The ICV working standard must be made from a source other than that used for the calibration standards.

11.1.2. Transfer 100 mL of well-mixed sample or standard to a clean sample digestion bottle.

Note: Reduced sample volumes can be used as long as a representative sample can be obtained and the reagent levels are adjusted to maintain the same sample to reagent ratio. All samples and standards must be processed similarly.

Note: Spiking is done before the addition of acids or reagents.

11.1.3. Add 5 mL of concentrated H_2SO_4 and 2.5 mL of concentrated HNO_3 mixing after each addition.

11.1.4. Add 15 mL of potassium permanganate solution. For samples high in organic materials or chlorides, additional permanganate may be added. Shake and add additional portions of permanganate solution until a purple color persists for at least 15 minutes. If after the addition of up to 25-mL additional permanganate the color does not persist, sample dilution prior to reanalysis may be required.

Note: When performing analyses using automated vs. manual techniques the sample dilution resultant from the addition of more than the original aliquot of permanganate solution must be compensated for by the addition of the same volume of permanganate to all other associated samples and standards in the run. In instances, where this is not feasible, the addition of excess reagent can be addressed through mathematical correction of the results to account for the resultant dilution effect.

11.1.5. Add 8 mL of potassium persulfate solution and heat for two hours in a water bath at 90 - 95 °C.

NOTE: Alternatively, for RCRA analyses using 7470A, samples may be digested using an autoclave for 15 minutes at 120 °C and 15 lbs.

11.1.6. Cool samples.

11.2. Sample Analysis:

11.2.1. Because of differences between various makes and models of CVAA instrumentation, no detailed operating instructions can be provided. Refer to the facility specific instrument operating SOP and the CVAA instrument manual for detailed setup and operation protocols.

11.2.2. All labs are required to detail the conditions/programs utilized for each instrument within the facility specific instrument operation SOP.

11.2.3. When ready to begin analysis, add 6 mL of sodium chloride-hydroxylamine hydrochloride solution to the samples to reduce the excess permanganate (the permanganate has been reduced when no purple color remains). Add this solution in 6-mL increments until the permanganate is completely reduced.

11.2.4. Manual determination:

11.2.4.1. Treating each sample individually, purge the headspace of the sample bottle for at least one minute.

11.2.4.2. Add 5 mL of stannous chloride solution and immediately attach the bottle to the aeration apparatus.

11.2.4.3. Allow the sample to stand quietly without manual agitation while the sample is aerated (1 L/min flow). Monitor the sample absorbance during aeration. When the absorbance reaches a maximum and the signal levels off, open the bypass valve and continue aeration until the absorbance returns to its baseline level. Close the bypass valve and remove the aeration device.

11.2.4.4. Place the aeration device into 100 mL of 1% HNO_3 and allow to bubble rinse until the next sample is analyzed.

11.2.5. Automated determination: Follow instructions provided by instrument manufacturer.

11.2.6. Perform a linear regression analysis of the calibration standards by plotting maximum response of the standards vs. concentration of mercury. Determine the mercury concentration in the samples from the linear regression fit of the calibration curve. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.

11.2.7. All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard.

11.2.8. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.

11.2.9. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

11.2.10. Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

11.2.11. The following analytical sequence must be used with 7470A and 245.1:

Instrument Calibration
ICV
ICB
Maximum 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table II (Appendix A) for quality control criteria to apply to Methods 7470A and 245.1.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions.

11.2.12. The following run sequence is consistent with 7470A, CLP and 245.1 and may be used as an alternate to the sequence in 11.2.11. This run sequence is recommended if multiple method requirements must be accommodated in one analytical run:

Instrument Calibration
ICV
ICB
CRA*
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run.
CCV

CCB

Refer to the appropriate CLP SOP (CORP-MT-0006) for quality control requirements for QC samples.

* Refer to the CLP SOP for information on the CRA.

11.2.13. For TCLP samples, full four point MSA will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

The reporting and matrix spike levels for TCLP analyses are detailed in Table I (Appendix A). Appendix E provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sample preparation.

11.3. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data are reviewed periodically throughout the run.

11.4. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.5. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.6. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix spike recoveries are calculated according to the following equation:

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:

DU1 = Sample result
DU2 = Sample duplicate result

12.5. The final concentration for an aqueous sample is calculated as follows:

$$mg/L = C \times D$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor

12.6. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.7. Appropriate factors must be applied to sample values if dilutions are performed.

12.8. Sample results should be reported with up to three significant figures in accordance with the STL North Canton significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Method performance is determined by the analysis of method blanks, laboratory control samples, matrix spike and matrix spike duplicate samples. The matrix spike recovery should fall within +/- 25 % and the matrix spike duplicates should compare within 20% RPD. The method blanks must meet the criteria in Section 9.3. The laboratory control sample should recover within 20% of the true value until in house limits are established.

13.3. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury).

16.2. "Methods for the Chemical Analysis of Water and Wastes", EPA-600/4-79-020, U.S.EPA, August 1983, Method 245.1.

16.3. U.S.EPA Statement of Work for Inorganics Analysis, ILMO3.0 and ILMO4.0.

16.4. QA-003, STL North Canton QC Program.

16.5. QA-004, Rounding and Significant Figures.

16.6. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference method.

17.1.1. Modifications from both 7470A and 245.1.

17.1.1.1. The 200 series methods and Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.1.2. This SOP allows for the use of reduced sample volumes to decrease waste generation. Reagent levels are adjusted to maintain the same ratios as stated in the source methods. According to a letter from Robert Booth of EPA EMSL-Cinn to David Payne of EPA Region V, "Reduction in sample size and appropriate corresponding reduction in

sample volume is not considered a significant change in the methodology."

17.1.1.3. The alternate run sequence presented in Section 11.2.12 is consistent with method requirements. An additional QC analysis (CRA) was added to accommodate the CLP protocol requirements.

17.1.2. Modifications from Method 7470A

17.1.2.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.1.2.2. Documentation is on file from EPA's Office of Solid Waste (Olliver Fordham 11/28/95) regarding the acceptance of the autoclave as an equivalent heating device to the water bath. In his letter, Mr. Fordham cited the CLP water protocol 245.1 CLP-M and therefore the operating parameters from that method were adopted for 7470A (15 minutes at 120 °C and 15 lbs.).

17.1.3. Modifications from 245.1

17.1.3.1. Method 245.1 Section 9.3 states concentrations should be reported as follows: Between 1 and 10 ug/L, one decimal; above 10 ug/L, to the nearest whole number. STL North Canton reports all Hg results under this SOP to two significant figures.

17.2. Modifications from previous SOP

17.2.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.2.2. Documentation is on file from EPA's Office of Solid Waste (Olliver Fordham 11/28/95) regarding the acceptance of the autoclave as an equivalent heating device to the water bath. In his letter, Mr. Fordham cited the CLP water protocol 245.1 CLP-M and therefore the operating parameters from the method were adopted for 7470A (15 minutes at 120° C and 15 lbs.).

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none. Refer to the Appendices for any facility specific information required to support this SOP.

17.4. Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

- Raw data (direct instrument printout)
- Run log printout from instrument software where this option is available or manually generated run log. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence).
- Data review checklist - See Appendix B
- Standards Documentation (source, lot, date).
- Copy of digestion log.
- Non-conformance summary (if applicable).

Figure 1. Aqueous Sample Preparation - Mercury

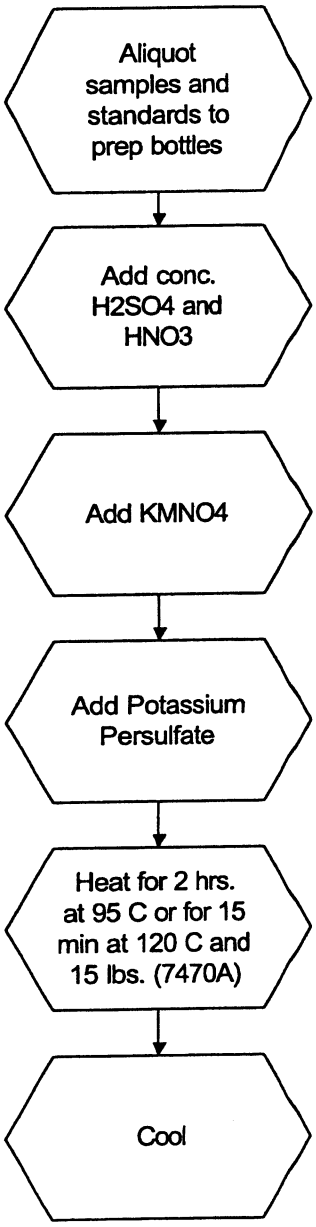
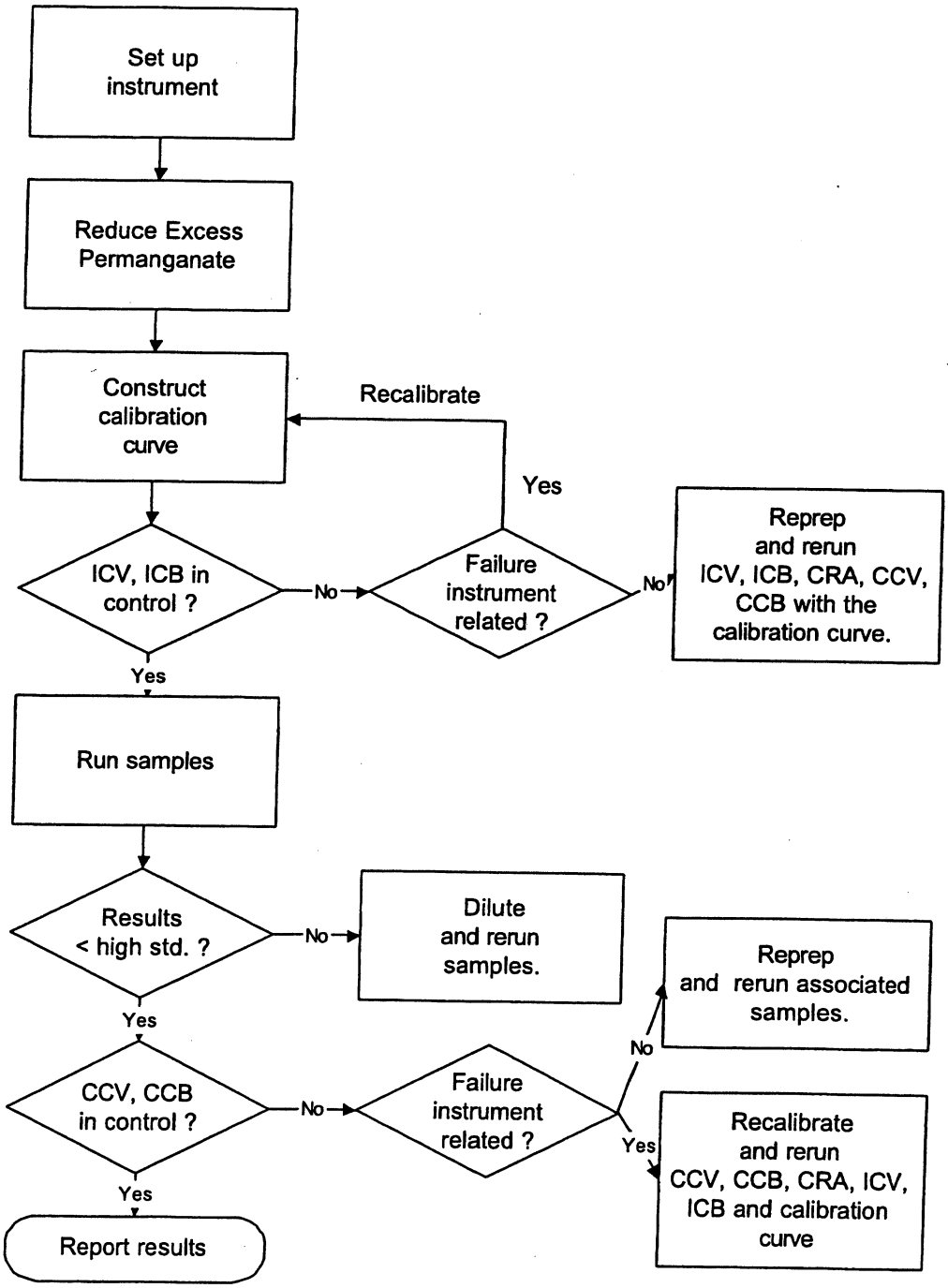


Figure 2. CVAA Mercury Analysis



APPENDIX A

TABLES

TABLE I. MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC STANDARD AND SPIKING LEVELS (MG/L)

Standard Aqueous RL	0.0002
TCLP RL	0.002
Std 0	0
Std 1	0.0002
Std 2	0.0005
Std 3	0.001
Std 4	0.005
Std 5 **	0.010
ICV	0.001 or 0.0025 ***
LCS/CCV	0.0025 or 0.005 ***
Aqueous MS	0.001
TCLP MS	0.005

* **SOP specified calibration levels must be used unless prevented by the instrument configuration or client specific requirements. Deviations from specified calibration levels must be documented in the facility specific instrument operation SOP and must be approved by the facility technical manager and Quality Assurance Manager.**

** **Optional standard which may be used to extend the calibration range as allowed by the instrument configuration. If the instrument configuration prevents the use of 6 standards, the 2-ppb standard may be eliminated in favor of the 10 ppb standard.**

*** **Concentration level dependent on high calibration standard used. CCV must be 50% of high standard concentration and ICV must be 20-25% of high standard concentration.**

TABLE II. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run.	90-110 % recovery.	Terminate analysis; Correct the problem; Recalibrate or reprep with calibration curve. (See Section 9.7).
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate or reprep with calibration curve. (See Section 9.7).
CCV	Every 10 samples and at the end of the run.	80 - 120 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep with calibration curve. (See Section 9.8).
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB or reprep with calibration curve. (See Section 9.8).
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL do not require redigestion (See Section 9.4).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.4 for additional requirements.

*See Sections 11.2.11 and 11.2.12 for exact run sequence to be followed.

TABLE II. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits.	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS (see Section 9.5).
Matrix Spike	One per sample preparation batch of up to 20 samples.	75 - 125 % recovery or in-house control limits. If the MS/MSD is out for an analyte, it must be in control in the LCS.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. (see Section 9.6) For TCLP see Section 11.2.13
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery or in-house control limits; RPD ≤ 20%. (See MS)	See Corrective Action for Matrix Spike.

APPENDIX B
EXAMPLE
STL NORTH CANTON Hg DATA REVIEW CHECKLIST

Example
STL North Canton Hg Data Review Checklist

Run/Project Information

Run Date: _____ **Analyst:** _____ **Instrument:** _____
Prep Batches Run: _____

Circle Methods used: **7470A / 245.1 :** CORP-MT-0005 Rev 1 **7471 / 245.5 :** CORP-MT-0007 Rev 1
CLP - AQ : CORP-MT-0006 Rev 0 **CLP - SOL :** CORP-MT-0008 Rev 0

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2ndLevel
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels?				
2. ICV/CCV analyzed at appropriate frequency and within control limits?				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?				
4. CRA run (CLP only)?				
B. Sample Results				
1. Were samples with concentrations > the high calibration standard diluted and reanalyzed?				
2. All reported results bracketed by in control QC?				
3. Sample analyses done within holding time?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits?				
2. Method blank done per prep batch and < RL or CRDL (CLP)?				
3. MS run at required frequency and within limits?				
4. MSD or DU run at required frequency and RPD within SOP limits?				
D. Other				
1. Are all nonconformances documented appropriately?				
2. Current IDL/MDL data on file?				
3. Calculations and Transcriptions checked for error?				
4. All client/ project specific requirements met?				
5. Date of analysis verified as correct?				

Analyst: _____ **Date:** _____
Comments:
_____
_____
_____

2nd Level Reviewer : _____ **Date:** _____
_____
_____
_____

APPENDIX C

MSA GUIDANCE

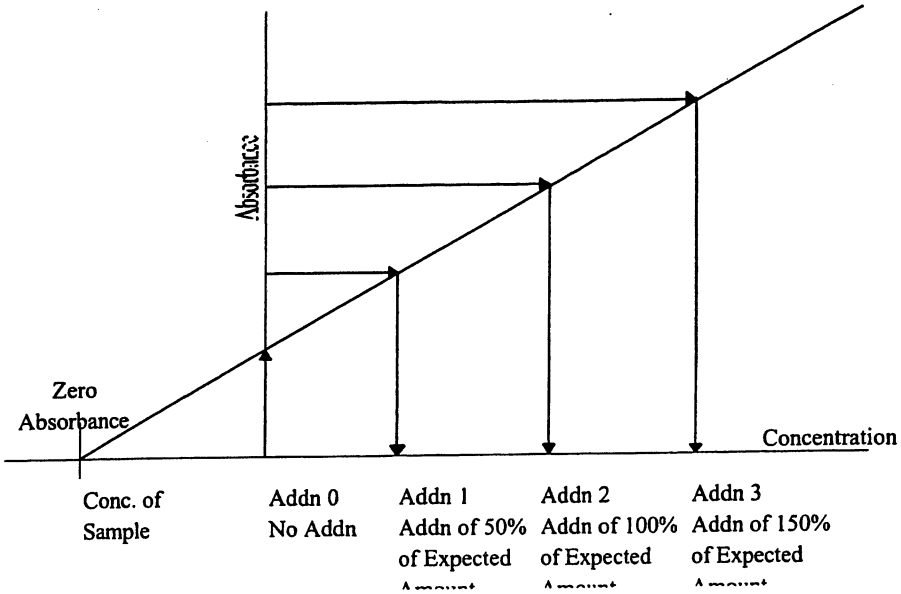
APPENDIX C. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.
- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX D

TROUBLESHOOTING GUIDE

APPENDIX D. TROUBLESHOOTING GUIDE

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Wrong lamp Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak EDL power supply set on "Continuous"
Erratic Readings	Source lamp not aligned properly Lamp not prewarmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
EDL Won't Light	Lamp cable not plugged in Lamp power set at 0 Lamp is dead Power supply fuse is blown Short in cord
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell
Background Correction Light Blinking	Background screen or attenuator faulty

APPENDIX E

CONTAMINATION CONTROL GUIDELINES

APPENDIX E. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

APPENDIX F

PREVENTIVE MAINTENANCE

APPENDIX F. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption (Leeman PS 200) [1]

Daily	Semi-annually	Annually
Clean lens.	Check Hg lamp intensity.	Change Hg lamp.
Check aperture.		Check liquid/gas separator.
Check argon flow.		
Check tubing.		
Check drain.		
Replace drying tube.		

Cold Vapor Atomic Absorption (PE 5000) [1]

Daily	Monthly
Clean aspirator by flushing with DI water.	Clean cell in aqua regia.
Check tubing and replace if needed.	Clean aspirator in aqua regia.
Clean windows with methanol.	
Change silica gel in drying tube.	
Check argon gas supply.	
Adjust lamp.	

APPENDIX G

INSTRUMENT SET UP

Hg Analysis (Leeman PS200II)

SYSTEM INITIALIZATION AND WARM UP

1. F1 Menu
2. Instrument

a. TASKMASTER

b. #4 Wake System Up Enter

The warming up period takes approximately 10 minutes.

TO SET UP INSTRUMENT FOR ANALYSIS

1. F1 Menu
2. Autosampler

   A. Rack Entry

   B. Edit (ex. Rack 1), Enter

   C. Cup ID - Enter (clears sample #'s)

   D. Extended ID- type in matrix of sample (water or solid), Enter.

   E. Press Insert Key and move cursor with arrows to cup ID and begin typing labels.

   F. F3 Print Screen

3. Press F2 Macro key and type in analyst's first name - Enter

   A. Enter folder name - ex. HG0306, Enter. If folder does not exist, type Y - Enter.

B. Type in - "Rack 1", "Rack 2" etc. , Enter.

C. Type in : FROM CUP TO CUP

Ex. = 1 30

Do the same for position 2 if needed. If not needed, you must press Enter 3 times to begin analysis.

STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: ACID DIGESTION OF SOILS, SW846 METHOD 3050B

(SUPERSEDES: REVISION 2.1 DATE 02/11/00)

Prepared by:	Patrick O'Mara	9/26/01 Date
Reviewed by:	Lisa McGaul Technology Specialist	9.24.1 Date
Reviewed by:	Gail Lin Cahn Quality Assurance Manager	9/25/01 Date
Reviewed by:	Environmental Health and Safety	9-27-01 Date
Reviewed by:	Cindy McQuanolo Laboratory Director	9-29-01 Date
Reviewed by:	Corporate Technology	Date

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of soil samples for the analysis of certain metals by Graphite Furnace Atomic Absorption (GFAA), Flame Atomic Absorption (FLAA), Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP), and Inductively Coupled Plasma-Mass Spectrometry (ICP/MS) as specified in SW846 Method 3050B.

1.2. Samples prepared by the protocols detailed in this SOP may be analyzed by ICP, ICP/MS, FLAA or GFAA for the elements listed in Table I (Appendix A). Other elements and matrices may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.

1.3. This method is not a total digestion, but will dissolve almost all metals that could become "environmentally available". By design, metals bound in silicate structures are not dissolved by this procedure, as they are not usually mobile in the environment. This SOP can be applied to metals in solids, sludges, wastes and sediments.

1.4. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

A representative 1 gram (wet weight) portion of sample is digested in nitric acid and hydrogen peroxide. The digestate is refluxed with hydrochloric acid for ICP, FLAA or antimony by GFAA analysis. The digestates are then filtered and diluted to 100 mL/100 g.

3. DEFINITIONS

3.1. Refer to the glossary in the Laboratory Quality Manual (LQM), latest version.

3.2. Total Metals: The concentration determined on an unfiltered sample following digestion. Note that this method is designed to determine the total *environmentally available* metals.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include: metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2. The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination.

4.3. Boron and silica from the glassware will grow into the sample solution during and following sample processing. For critical low level determinations of boron and silica, only quartz and/or plastic labware should be used.

4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents and other matrices may not be digested using these methods if they are not soluble with acids. If physical interferences are present, they should be documented.

4.5. Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.6. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs the sample must be reprepared. Antimony is easily lost by volatilization from hydrochloric media.

4.7. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL North Canton associates.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. The following materials are known to be **corrosive:**

Hydrochloric acid and nitric acid.

5.3.2. The following materials are known to be **oxidizing agents**:

Nitric acid and hydrogen peroxide.

5.3.3. All heating of samples must be carried out in a fume hood.

5.4. The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood. The analyst should also be aware of the potential for a vigorous reaction.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit or under other means of mechanical ventilation.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported **immediately** to a laboratory supervisor.

5.8. Always carry bulk concentrated acid bottles in appropriate impact proof containers.

5.9. Acid/peroxide spills must be neutralized immediately, flushed with water and cleaned up using appropriate spill kits.

5.10. Discard chipped or broken beakers to prevent injury. Chipped glassware may be fire polished as an alternative to disposal.

5.11. Any and all accidents and spills must be reported to the lab supervisor or EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

6.1. Hot plate, digestion block, steam bath or other heating source capable of maintaining a temperature of 90-99°C.

6.2. Calibrated thermometer that covers a temperature range of 0-200°C.

6.3. Griffin beakers of assorted sizes or equivalent.

6.4. Vapor recovery device (Watch glasses, ribbed or other device).

6.5. Whatman No. 41 filter paper or equivalent.

6.6. Funnels or equivalent filtration apparatus.

6.7. Centrifugation equipment (if desired method of removing particulates is centrifugation).

6.8. Graduated cylinder or equivalent capable of measuring 100 mL within 3% accuracy.

6.9. Analytical balance capable of accurately weighing to the nearest 0.01 grams.

6.10. Repippetors or suitable reagent dispensers.

6.11. Calibrated automatic pipettes with corresponding pipette tips or Class A glass volumetric pipettes.

6.12. Class A volumetric flasks.

6.13. pH indicator strips (pH range 0 - 6).

6.14. Plastic bottles.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative SOPs.

7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom STL North Canton solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Working ICP LCS/MS spike solution: Prepare the ICP LCS/MS working spike solutions from custom stock standards to the final concentration listed in Table II. The working spike must be prepared in a matrix of 5% HNO_3. This acid (5 mL of concentrated HNO_3 per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working ICP LCS solution must be made fresh every three months.

7.4. Working GFAA LCS/MS spike solution: Prepare the GFAA working LCS/MS spike solutions by diluting the custom stock solution (7.2) 200x. The working spike solution must be prepared in a matrix of 5% HNO_3. This acid (5 mL of concentrated HNO_3 per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working GFAA LCS/MS solution must be made fresh every three months.

7.5. The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom STL North Canton solution, the individual facility must purchase a solution from the designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.6. Aqueous laboratory control samples (LCSW) and matrix spike samples are prepared as described in Sections 9.5 and 9.6. Refer to Tables II and III (Appendix A) for details regarding the stock, working standard and final digestate spike concentrations for ICP and GFAA LCS and matrix spike preparations.

7.7. Nitric acid (HNO_3), concentrated, trace metal grade or better.

7.8. Nitric acid, 1:1 - dilute concentrated HNO_3 with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

7.9. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.10. Hydrochloric acid, 1:1 - dilute concentrated HCl with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

7.11. 30% Hydrogen peroxide (H_2O_2), reagent grade.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2. Soil samples do not require preservation but must be stored at 4 °C ± 2 °C until the time of analysis.

9. QUALITY CONTROL

Table IV (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using Method 3050B the following requirements must be met.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements or verified as detailed in STL North Canton QA Policy QA-005. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the STL North Canton reporting limit.

9.1.2. Initial Demonstration Study- this requires the analysis of four QC check samples. The QC check sample is a well characterized laboratory generated sample used to monitor method performance, which should contain all the analytes of interest. The results of the initial demonstration study must be acceptable before analysis of samples may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.1.2.2. Calculations and acceptance criteria for QC check samples are given in the determinative SOPs (CORP-MT-0001, CORP-MT-0003).

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not counted towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Criteria for the acceptance of blanks are contained within the individual analytical method SOP's. If the method blank does not meet the criteria contained within the analytical method SOPs, the blank and all associated samples in the batch must be redigested.

9.5. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOP's. Corrective action when LCS results fail to meet control limits will be repreparation and reanalysis of the batch. Tables II and III provide the details regarding the stock, working standards and final spike concentrations for ICP and GFAA. Refer to Section 7.3 or 7.4 for instructions on preparation of the aqueous LCS.

9.5.1. The LCS is prepared by spiking a 1mL or 1 g aliquot of reagent water with 2 mL of the working LCS/MS spike solution (7.3 or 7.4). The LCS is then processed as described in either Section 11.10.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis. If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. Corrective action when MS results fail to meet control limits

does not include repreparation of samples unless the results indicate that a spiking error may have occurred. Tables II and III provide the details regarding the stock, working standards and final matrix spike concentrations for ICP and GFAA. Refer to Sections 7.3 and 7.4 for instructions on preparation of the working matrix spike solutions.

9.6.1. The soil matrix spike sample is prepared by spiking a 1 g aliquot of a sample with 2mL of the working LCS/MS spike solution (7.3 or 7.4). The matrix spike sample is then processed as described in either Section 11.10.

9.7. Quality Assurance Summaries - Certain clients may require specific project or program QC which may supersede the SOP requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Hotplate or block temperature must be verified daily for each unit used and must be recorded on either the metals preparation log or in a hotplate temperature logbook. The hotplate temperature should be verified by measuring the temperature of a beaker of reagent water placed on each hotplate. For block digestors, use a tube containing water.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. The heating procedures are carried out in a properly functioning hood.

11.4. All samples are to be checked out of sample control with the chain of custody documentation filled out completely.

11.5. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample. The use of automatic label printing programs is recommended to reduce transcription errors (QuantIMS option).

11.6. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating prep, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge like, organic liquid, lots of sediment etc.) contact the lab supervisor or project administrator for further instructions. In some cases it may be more appropriate to process these samples as solids.

11.7. If possible prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and reporting group.

11.8. In some cases, both AA and ICP digests are required on each sample. It is recommended that both aliquots be weighed out and processed at the same time.

11.9. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

11.10. Preparation of Soils, Sediments and Sludges for Analysis by GFAA, ICP, ICP/MS and FLAA.

11.10.1. Mix sample thoroughly by stirring with a clean plastic or wooden spoon or spatula.

11.10.2. For each digestion procedure required (i.e., ICP or GFAA), weigh a 1.0 portion of solid and record the exact weight to the nearest 0.01 g. A 2 g sample size may also be used if needed to meet the reporting limits.

11.10.3. Measure additional aliquots of the designated samples for the MS and MSD analyses.

11.10.4. Spike each of the MS and MSD aliquots with 2 mL of the working LCS/MS spiking solution (7.3 or 7.4).

11.10.5. Prepare a beaker for the method blank.

11.10.6. Prepare a beaker for the LCS. Add 2 mL of the working LCS/MS spiking solution (7.3 or 7.5).

11.10.7. Add 10 mL of 1:1 HNO_3 and mix the sample.

11.10.8. Heat sample to 95°±4° C and reflux for 10 minutes without boiling, using a vapor recovery device.

Note: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY during any part of the digestion. Doing so will result in the loss of analyte and the sample must be reprepared.

11.10.9. Allow sample to cool, if necessary.

11.10.10. Add 5 mL of concentrated HNO_3 and replace vapor recovery device.

11.10.11. Reflux at 95°±4° C for 30 minutes. (Add reagent water as needed to ensure that the volume of solution is not reduced to less than 5 mL.)

11.10.12. If brown fumes are observed, repeat step 11.10.10 until no more fumes are evolved.

11.10.13. Allow the samples to cool, if necessary.

11.10.14. Add approximately 2 mL of reagent water and 1 mL of 30 % H_2O_2. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

11.10.15. Replace the vapor recovery device and heat sample until effervescence subsides.

11.10.16. Allow the sample to cool, if necessary.

11.10.17. Continue adding 30% H_2O_2 in 1 mL aliquots with warming until effervescence is minimal or sample appearance is unchanged.

Note: Do not add more than a total of 10 mL of 30 % H_2O_2.

11.10.18. Continue heating at 95°±4° C for 75 minutes until the volume is reduced to approximately 5-10 mL. Alternatively the sample may be heated for 2 hours.

11.10.19. If the sample is being prepared for ICP or FLAA analyses add 10 mL of concentrated HCl and reflux for an additional 10 minutes without boiling. This step is omitted for analysis by ICP-MS and GFAA

Note: Antimony and silver have poor solubility in dilute nitric acid solution. Therefore it is strongly recommended that these elements are determined by the ICP procedure that includes HCl as the final digestion acid.

11.10.20. Allow the sample to cool.

11.10.21. Wash down beaker walls and vapor recovery device with reagent water.

11.10.22. Filter sample through Whatman 41 filter paper or equivalent into a graduated cylinder or equivalent or a pre-weighed bottle. Other measuring bottles (for example, Corning Snap Seals™) may be used if their accuracy is documented and is better than $\pm$ 2%. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material

11.10.23. Dilute sample to 100 mL or 100g with reagent water. The sample is now ready for analysis.

Note: This SOP allows for samples to be weighed instead of measured volumetrically. This assumes the density of the diluted sample is close to 1.0 g/mL (See Section 17.1.2).

12. DATA ANALYSIS AND CALCULATIONS

Not Applicable.

13. METHOD PERFORMANCE

13.1. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. In general, the matrix spike recovery should fall within +/- 20 % and the matrix spike duplicates should compare within 20% RPD. Method blanks must meet the criteria specified in the determinative SOPs. The laboratory control samples should recover within 20% of the true value until in house control limits are established. Acceptance criteria are given in the determinative SOPs.

13.2. The initial demonstration study as detailed in Section 9.1.2 must be acceptable before the analysis of field samples under this SOP may begin. The results of the initial demonstration

study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

13.3. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

15.2. Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996. Method 3050B.

16.2. CORP-MT-0001, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis of Water and Wastes, Method 6010B and Method 200.7.

16.3. CORP-MT-0003, Graphite Furnace Atomic Absorption Spectroscopy, SW846 Method 7000A and MCAWW 200 Series Methods.

16.4. NC-MT-0002, Inductively Coupled Plasma-Mass Spectrometry, EPA Methods 6020 and 200.8.

16.5. QA-003, STL North Canton QC Program.

16.6. QA-004, Rounding and Significant Figures.

16.7. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference method.

17.1.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants, as defined in the determinative SOPs, are allowed up to two times the reporting limit in the blank following consultation with the client.

17.1.2. This SOP allows for aqueous samples to be weighed instead of measured volumetrically. This assumes the density of the sample is close to 1.0 g/mL. Samples with large amounts of sediment or suspended solids, sludges, non-aqueous liquids must be processed volumetrically. Weighing samples directly into the digestion vessel minimizes the potential for cross contamination, offers improved accuracy over the use of graduated cylinders (comparable to volumetric flask accuracy), uses less glassware and is more efficient.

17.2. Modifications from previous SOP

17.2.1. ICP/MS has been added as an appropriate determinative technique.

17.2.2. The approved analyte list may be modified provided that additional elements meet the requirements in section 13.

17.2.3. Directions for digestion for set time periods rather than reduction to set volumes have been added.

17.2.4. The order of two steps in the digestion has been changed. (See section 11.10.20)

17.2.5. Definition of the method as determining total environmentally available metals has been added.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none. Refer to the Appendices for any facility specific information required supporting this SOP.

17.4. Documentation and Record Management

The preparation benchsheet should, at a minimum, include the following information:

- Preparation date, analyst initials, matrix, prep type (ICP or GFAA), SOP reference.
- Sample ID; initial weight/volume and final weight/volume.
- Standards Documentation (source, lot, prep date, volume added).
- Analyst Signature.
- Reviewer's Signature and date.

Figure 1. Soil Sample Preparation (Section 11.10)

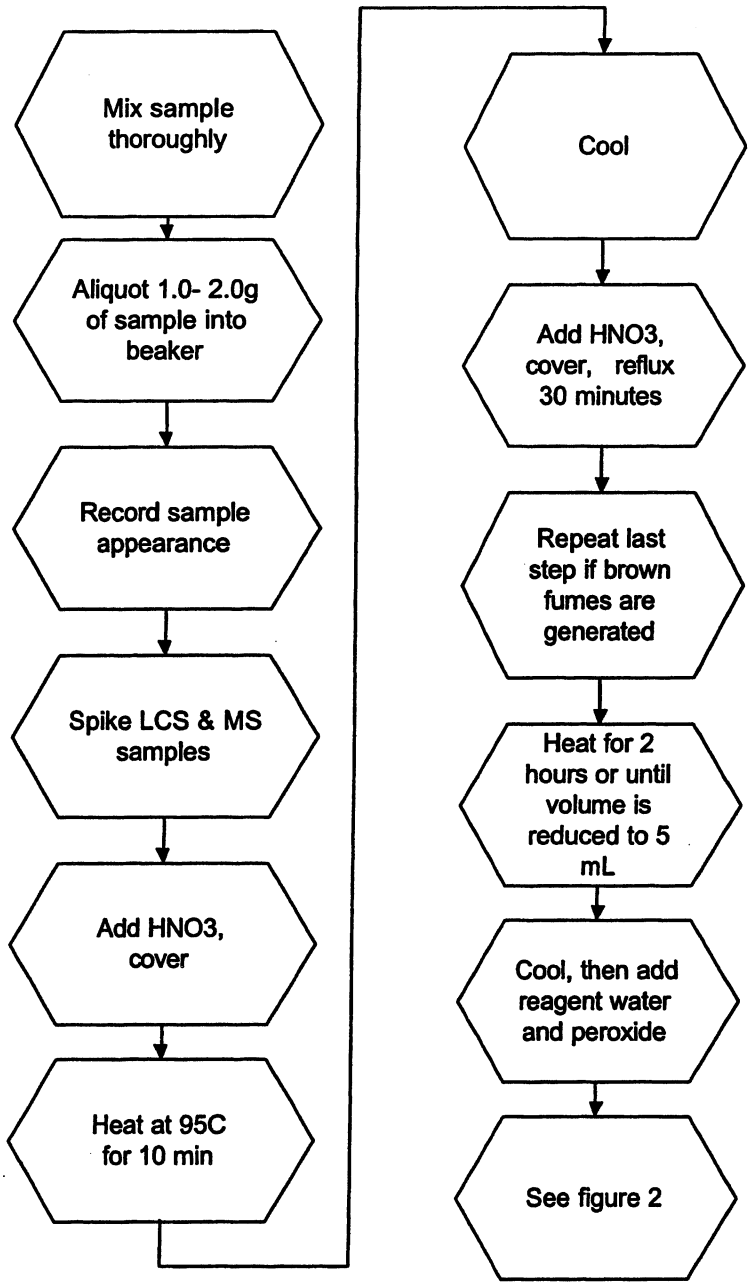
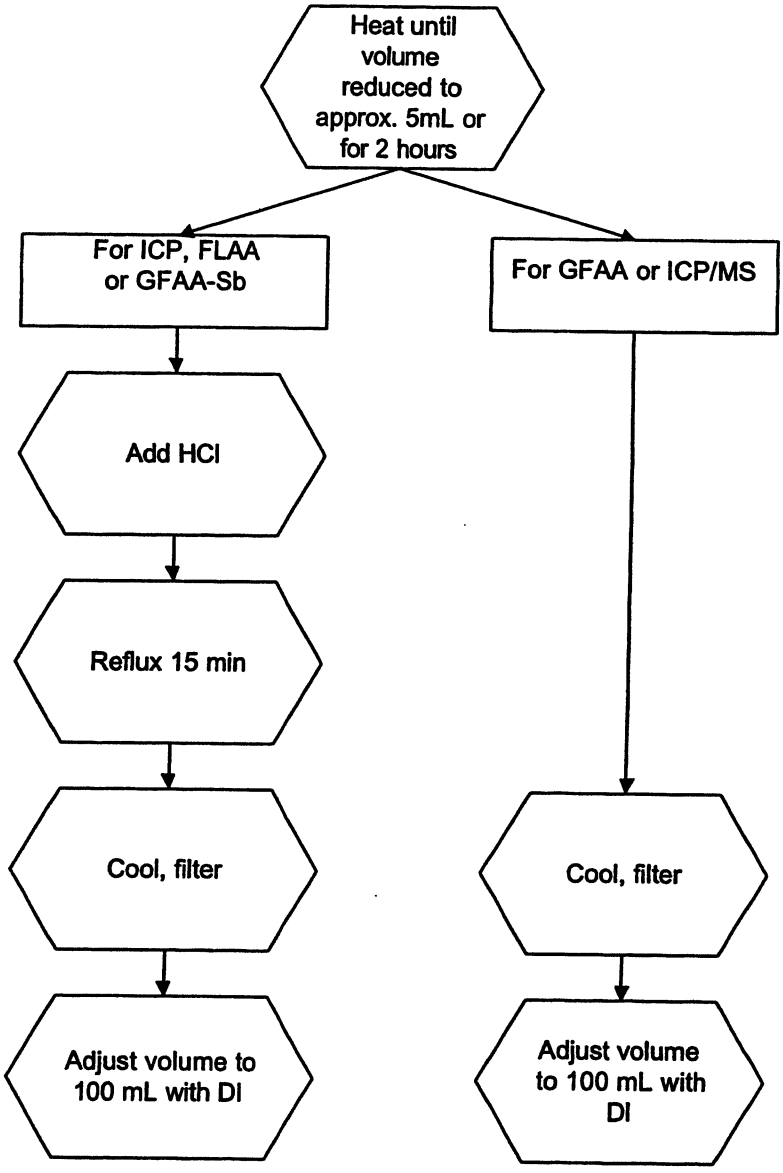


Figure 2. Soil Sample Preparation (continued)



APPENDIX A

TABLES

TABLE I. Method 3050A Approved Analyte List

ELEMENT	Symbol	CAS Number
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Thallium	Tl	7440-28-0
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

TABLE II. ICP and FLAA Soil Matrix Spike and Aqueous LCS Levels

ELEMENT	Working LCS/MS Standard (mg/L)	Aqueous LCS/MS Level* (ug/L)	Soil MS Level ** (mg/Kg)
Aluminum	100	2000	200
Antimony	25	500	50
Arsenic	100	2000	200
Barium	100	2000	200
Beryllium	2.5	50	5
Cadmium	2.5	50	5
Calcium	2500	50000	5000
Chromium	10	200	20
Cobalt	25	500	50
Copper	12.5	250	25
Iron	50	1000	100
Lead	25	500	50
Lithium	50	1000	100
Magnesium	2500	50000	5000
Manganese	25	500	50
Molybdenum	50	1000	100
Nickel	25	500	50
Potassium	2500	50000	5000
Selenium	100	2000	200
Silver	2.5	50	5
Sodium	2500	50000	5000
Strontium	50	1000	100
Thallium	100	2000	200
Vanadium	25	500	50
Zinc	25	500	50
Boron	50	1000	100
Tin	100	2000	200
Titanium	50	1000	100

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 2.0 mL working spike (7.3) to 100 mL of sample.

** Final soil spike concentration based on the addition of 2.0 mL working spike (7.3) to 1.0 g of sample/100 mL final volume (assumes 100% solids).

TABLE III. ICPMS Soil Matrix Spike and Aqueous LCS Levels

ELEMENT	Working LCS/MS Standard (mg/L)	Aqueous LCS/MS Level* (ug/L)	Soil MS Level ** (mg/Kg)
Aluminum	10	1000	100
Antimony	10	100	10
Arsenic	10	100	10
Barium	10	100	10
Beryllium	10	100	10
Cadmium	10	100	10
Calcium	100	1000	100
Chromium	10	100	10
Cobalt	10	100	10
Copper	10	100	10
Iron	100	1000	100
Lead	10	100	10
Magnesium	100	1000	100
Manganese	10	100	10
Molybdenum	10	100	10
Nickel	10	100	10
Potassium	100	1000	100
Selenium	10	100	10
Silver	10	100	10
Sodium	100	1000	100
Strontium	10	100	10
Thallium	10	100	10
Vanadium	10	100	10
Zinc	10	100	10
Boron	10	100	10
Tin	10	100	10
Titanium	10	100	10
Zirconium	10	100	10

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 1.0 mL working spike (7.4) to 100 mL of sample.

** Final soil spike concentration based on the addition of 1.0 mL working spike (7.4) to 1.0 g of sample/100 mL final volume (assumes 100% solids).

TABLE IV. GFAA Soil Matrix Spike and Aqueous LCS Spike Levels

ELEMENT	Stock LCS/MS Standard (mg/L)	Working LCS/MS Standard (ug/L)	Aqueous LCS/ MS Level * (ug/L)	Soil MS Level** (mg/Kg)
Arsenic	400	2000	40	4
Selenium	400	2000	40	4
Lead	400	2000	40	4
Thallium	400	2000	40	4
Antimony	400	2000	40	4
Cadmium	40	200	4	0.4
Chromium	100	500	10	1
Silver	50	250	5	0.5

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 1.0 mL working spike (7.5) to 50 mL of sample.

** Final soil spike concentration based on the addition of 2.0 mL working spike (7.5) to 1.0 g of sample/100 mL final volume (assumes 100% solids).

TABLE V. Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: CORP-MT-0001 CORP-MT-0003	Redigest and reanalyze samples.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: CORP-MT-0001 CORP-MT-0003	Redigest and reanalyze all samples associated with the LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: CORP-MT-0001 CORP-MT-0003	Reprep not required unless preparation error suspected.
Matrix Spike Duplicate	See Matrix Spike	Refer to determinative SOPs: CORP-MT-0001 CORP-MT-0003	See Corrective Action for Matrix Spike.

APPENDIX B

CONTAMINATION CONTROL GUIDELINES

APPENDIX B. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or latex gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

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STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: PREPARATION AND ANALYSIS OF MERCURY IN SOLID SAMPLES BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY, SW846 7471A AND MCAWW 245.5

(SUPERSEDES: REVISION 2.2, DATED 02/06/01)

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7471A and MCAWW Method 245.5.

1.2. The associated LIMs method code is O9.

1.3. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants, potassium permanganate and potassium persulfate, has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation and volume of sample used.

1.4. Methods 7471A and 245.5 are applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits, wastes, wipes and sludge-type materials. All matrices require sample preparation prior to analysis.

1.5. The STL North Canton reporting limit for mercury in solid matrices is 0.033 mg/kg based on a 0.6 g sample aliquot (wet weight).

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in hydrochloric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3. DEFINITIONS

3.1. Total Metals: The concentration determined on an unfiltered sample following digestion.

4. INTERFERENCES

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1. Potassium permanganate which is used to breakdown organic mercury compounds also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.2. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.3. Chlorides can cause a positive interference. Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample headspace before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.4. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.5. Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

4.6. The most common interference is laboratory contamination which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL North Canton associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. The following materials are known to be **corrosive:**

hydrochloric acid, nitric acid and sulfuric acid.

5.3.2. The following materials are known to be **oxidizing agents**:

nitric acid, potassium permanganate, potassium persulfate and magnesium perchlorate.

5.3.3. Mercury is a highly toxic element that must be handled with care. The analyst must be aware of the handling and clean-up techniques before working with mercury. Since mercury vapor is toxic, precaution must be taken to avoid its inhalation, ingestion or absorption through skin. All lines should be checked for leakage and the mercury vapor must be vented into a hood or passed through a mercury absorbing media such as:

5.3.3.1. Equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4, or

5.3.3.2. Iodine, 0.25%, in a 3% KI solution.

5.3.4. Magnesium sulfate is known to be a reproductive toxin (mutagen).

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported **immediately** to a laboratory supervisor.

5.6. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.

5.7. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders be located outside the laboratory and the gas led to the instrument through approved lines.

5.8. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

6. EQUIPMENT AND SUPPLIES

6.1. Temperature controlled water bath (capable of maintaining temperature of 90- 95 °C).

6.2. Atomic Absorption Spectrophotometer equipped with:

6.2.1. Absorption Cell with quartz end windows perpendicular to the longitudinal axis. Dimensions of the cell must result in sufficient sensitivity to meet the SOP defined reporting limit. The quartz windows must be maintained to provide accurate measurements. Any scratches or fingerprints can alter the absorption of UV radiation.

6.2.2. Mercury specific hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL).

6.2.3. Peristaltic pump which can deliver 1 L/min air.

6.2.4. Flowmeter capable of measuring an airflow of 1 L/min.

6.2.5. Recorder or Printer.

6.2.6. Aeration Tubing: A straight glass frit having a course porosity and Tygon tubing is used for the transfer of mercury vapor from the sample bottle to the absorption cell and return.

6.2.7. Drying device (a drying tube containing magnesium perchlorate or magnesium sulfate and/or a lamp with a 60 W bulb) to prevent condensation in cell. The lamp is positioned to shine on the absorption cell maintaining the air temperature in the cell about 10 °C above room temperature. Other drying devices that acheive the same purpose are also acceptable (i.e., Gortex filter).

Note: Instruments designed specifically for the measurement of mercury using the cold vapor technique may be substituted for the atomic absorption spectrophotometer.

6.3. BOD bottles or equivalent.

6.4. Nitrogen or argon gas supply, welding grade or equivalent.

6.5. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.6. Class A volumetric flasks.

6.7. Top-loading balance, capable of reading up to two decimal places.

6.8. Thermometer (capable of accurate readings at 95 °C).

6.9. Disposable cups or tubes.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Stock (10 ppm) mercury standards (in 10% HNO_3) are purchased as custom STL North Canton solutions. All standards must be stored in FEP fluorocarbon or previously unused

polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Working mercury standard (0.1 ppm): Take 1 mL of the stock mercury standard (7.2) and dilute to 100 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO_3. This acid (150 uL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.4. The calibration standards must be prepared fresh daily from the working standard (7.3) by transferring 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working mercury standard into sample preparation bottles and proceeding as specified in Section 11.1

Note: Alternate approaches to standard preparation may be taken and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained. For example, automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

7.5. The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.

7.6. Refer to Table I (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All standards must be processed through the entire analytical procedure including sample preparation.

7.7. Nitric acid (HNO_3), concentrated, trace metal grade or better.

Note: If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

7.8. Sulfuric acid (H_2SO_4), concentrated, trace metal grade or better.

7.9. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.10. Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3.

7.11. Stannous sulfate solution: Add 25 g of stannous sulfate to 250 mL of 0.5 N sulfuric acid. This mixture is a suspension and should appear cloudy. This solution should be made daily and should be stirred continuously during use.

Note: Stannous chloride may be used in place of stannous sulfate. Prepare the stannous chloride solution according to the recommendations provided by the instrument manufacturer.

7.12. Sodium chloride-hydroxylamine hydrochloride solution: Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride to every 100 mL of reagent water.

Note: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.13. Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate for every 100 mL of reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for mercury is 28 days from time of collection to the time of sample analysis.

8.2. Soil samples do not require preservation but must be stored at 4° C ± 2° C until the time of analysis.

9. QUALITY CONTROL

Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to the analysis of any analyte using 7471A or the 245.5, the following requirements must be met.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the STL North Canton reporting limit.

9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well characterized laboratory generated sample used to

monitor method performance. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count for determining the size of a preparation batch.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20 times higher than the blank contamination level).

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

9.5. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the

method within acceptable accuracy and precision guidelines. The LCS must be carried through the entire analytical procedure. If the LCS is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, a control limit of 70-130% recovery must be applied.

- In the instance where the LCS recovery is > 130% and the sample results are < RL, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the case narrative."

- In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table I (Appendix A).

- If analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 70 - 130 % recovery and 20% RPD must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not

necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.7. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV result must fall within 10% of the true value for that solution . An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. (See Section 11.2.10 and Section 11.2.11 for required run sequence). If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include repreparation of the ICV, ICB, CRA, CCV and CCB with the calibration curve.

9.8. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV must be a mid-range standard at a concentration other than that of the ICV. The CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. (See Section 11.2.10 and 11.2.11 for required run sequence.) The CCB result must fall within +/- RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs.

9.9. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the sample prior to preparation. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Section 11.2.12 for additional information on when full 4 point MSA is required as well as Appendix C for specific MSA requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Calibration standards must be processed through the preparation procedure as described in Section 11.1.

10.2. Due to the differences in preparation protocols separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.

10.3. Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.4. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility specific instrument SOP and CVAA instrument manual for detailed setup and operation protocols.

10.5. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at the STL North Canton reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7.5 and Table I for additional information on preparing calibration standards and calibration levels.

10.6. The calibration curve must have a correlation coefficient of ≥0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results can not be reported from a curve with an unacceptable correlation coefficient.

10.7. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corrective actions.

11. PROCEDURE

11.1. Standard and Sample Preparation:

11.1.1. All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed through the digestion procedure as well as the field samples.

11.1.2. Transfer 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working standard (7.3) into a series of sample digestion bottles. For the ICV, transfer a 2.5 ml aliquot of the working standard. The ICV working standard must be made from a source other than that used for the calibration standards.

Note: Alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained.

11.1.3. Add reagent water to each standard bottle to make a total volume of 10 mL. Continue preparation as described under 11.1.5 below.

11.1.4. Transfer triplicate 0.2 g portions of a well mixed sample into a clean sample digestion bottle. Add 10mL of reagent water to each standard bottle. Continue

preparation as described under 11.1.5.

11.1.5. Water Bath protocol:

11.1.5.1. To each **standard** bottle: Add 5 mL of aqua regia.
To each **sample** bottle: Add *10* mL of reagent water and 5 mL of aqua regia.

11.1.5.2. Heat for 2 minutes in a water bath at 90 - 95 ° C.

11.1.5.3. Add *40* mL of distilled water.

11.1.5.4. Add 15 mL of potassium permanganate solution.

11.1.5.5. Heat for 30 minutes in the water bath at 90 - 95 °C.

11.1.5.6. Cool.

11.1.5.7. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.

11.1.5.8. To each **standard** bottle: Add 50 mL of reagent water.
To each **sample** bottle: Add *50* mL of reagent water.

11.1.5.9. Continue as described under Section 11.2.

11.2. Sample Analysis:

11.2.1. Because of differences between various makes and models of CVAA instrumentation, no detailed operating instructions can be provided. Refer to the facility specific instrument operating SOP and the CVAA instrument manual for detailed setup and operation protocols.

11.2.2. All labs are required to detail the conditions/programs utilized for each instrument within the facility specific instrument operation SOP.

11.2.3. Manual determination:

11.2.3.1. Treating each sample individually, purge the head space of the sample bottle for at least one minute.

11.2.3.2. Add 5 mL of stannous chloride solution and immediately attach the bottle to the aeration apparatus.

11.2.3.3. Allow the sample to stand quietly without manual agitation while the sample is aerated (1 L/min flow). Monitor the sample absorbance during aeration. When the absorbance reaches a maximum and the signal levels off, open the bypass valve and continue aeration until the absorbance returns to its baseline level. Close the bypass valve and remove the aeration device.

11.2.3.4. Place the aeration device into 100 mLs of 1% HNO_3 and allow to bubble rinse until the next sample is analyzed.

11.2.4. Automated determination: Follow instructions provided by instrument manufacturer.

11.2.5. Perform a linear regression analysis of the calibration standards by plotting maximum response of the standards vs. ug of mercury. Determine the mercury concentration in the samples from the linear regression fit of the calibration curve. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.

11.2.6. All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard.

11.2.7. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.

11.2.8. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

11.2.9. Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

11.2.10. The following analytical sequence must be used with 7471A and 245.5:

Instrument Calibration
ICV
ICB

Maximum 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table II (Appendix A) for quality control criteria to apply to Methods 7471A and 245.5.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions.

11.2.11. The following run sequence is consistent with 7471A, CLP and 245.5 and may be used as an alternate to the sequence in 11.2.10. This run sequence is recommended if multiple method requirements must be accommodated in one analytical run:

Instrument Calibration
ICV
ICB
CRA*
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run.
CCV
CCB

Refer to the appropriate CLP SOP (CORP-MT-0008) for quality control requirements for QC samples.

* Refer to the CLP SOP for information on the CRA.

11.2.12. For TCLP samples, full four point MSA will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

Appendix E provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sample preparation.

11.3. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.4. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.5. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.6. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix spike recoveries are calculated according to the following equation:

$$\%\,R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:

DU1 = Sample result
DU2 = Sample duplicate result

12.5. For automated determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$mg/kg,\ dry\ weight = (C \times V \times D)/(W \times S)$$

Where:

C = Concentration (ug/L) from instrument readout
V = Volume of digestate (L)
D = Instrument dilution factor
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation.

12.6. For manual (total) determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$mg/kg,\ dry\ weight = (C)/(W \times S)$$

Where:
C = Concentration (ug) from instrument readout
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. Sample results should be reported with up to three significant figures in accordance with the STL North Canton significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.1.

13.2. Method performance is determined by the analysis of method blank, laboratory control sample, matrix spike and matrix spike duplicate samples. The matrix spike recovery should fall within +/- 30 % and the matrix spike duplicates should compare within 20% RPD. The method blanks must meet the criteria in Section 9.4. The laboratory control sample should recover within 20% of the true value until in house limits are established.

13.3. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A (Mercury).

16.2. "Methods for the Chemical Analysis of Water and Wastes", EPA-600/4-79-020, U.S.EPA, August 1983, Method 245.5.

16.3. U.S.EPA Statement of Work for Inorganics Analysis, ILMO3.0.

16.4. QA-003, STL North Canton QC Program.

16.5. QA-004, Rounding and Significant Figures.

16.6. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference method.

17.1.1. Modifications from both 7471A and 245.5.

17.1.1.1. A potassium persulfate oxidation step has been included to facilitate the breakdown of organic mercurials which are not completely oxidized by potassium permanganate. Use of potassium persulfate in combination with the permanganate improves the recovery of mercury from organo-mercury compounds. The use of persulfate has been incorporated in several recent EPA mercury protocols.

17.1.1.2. The alternate run sequence presented in Section 11.2.11 is consistent with method requirements. An additional QC analysis (CRA) was added to accommodate the CLP protocol requirements.

17.1.2. Modifications from Method 7471A

17.1.2.1. Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.2.2. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.1.3. Modifications from 245.5

17.1.3.1. Method 245.5 Section 9.3 states concentrations should be reported as follows: Between 0.1 and 1 ug/g, to the nearest 0.01 ug; between 1 and 10 ug/g, to the nearest 0.1ug; above 10 ug/g, to the nearest ug. STL North Canton reports all Hg results under this SOP to two significant figures.

17.2. Modifications from previous SOP

17.2.1. Section 1.4 reporting limit changed form 0.1 mg/kg based on a 0.2 g to 0.3 mg/kg based on a 0.6 g sample aliquot

17.2.2. Section 9.3 added MS and MSDs as not counted in determination of preparation batches.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none. Refer to the Appendices for any facility specific information required to support this SOP.

17.4. Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

- Raw data (direct instrument printout)
- Run log printout from instrument software where this option is available or manually generated run log. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence).
- Data review checklist - See Appendix B
- Standards Documentation (source, lot, date).
- Copy of digestion log.

Non-conformance summary (if applicable).

Figure 1. Solid Sample Preparation for Mercury - Water Bath Procedure

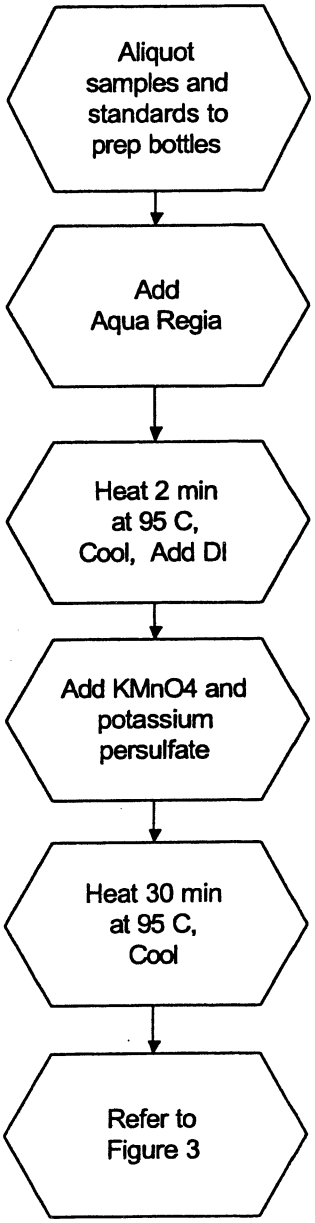
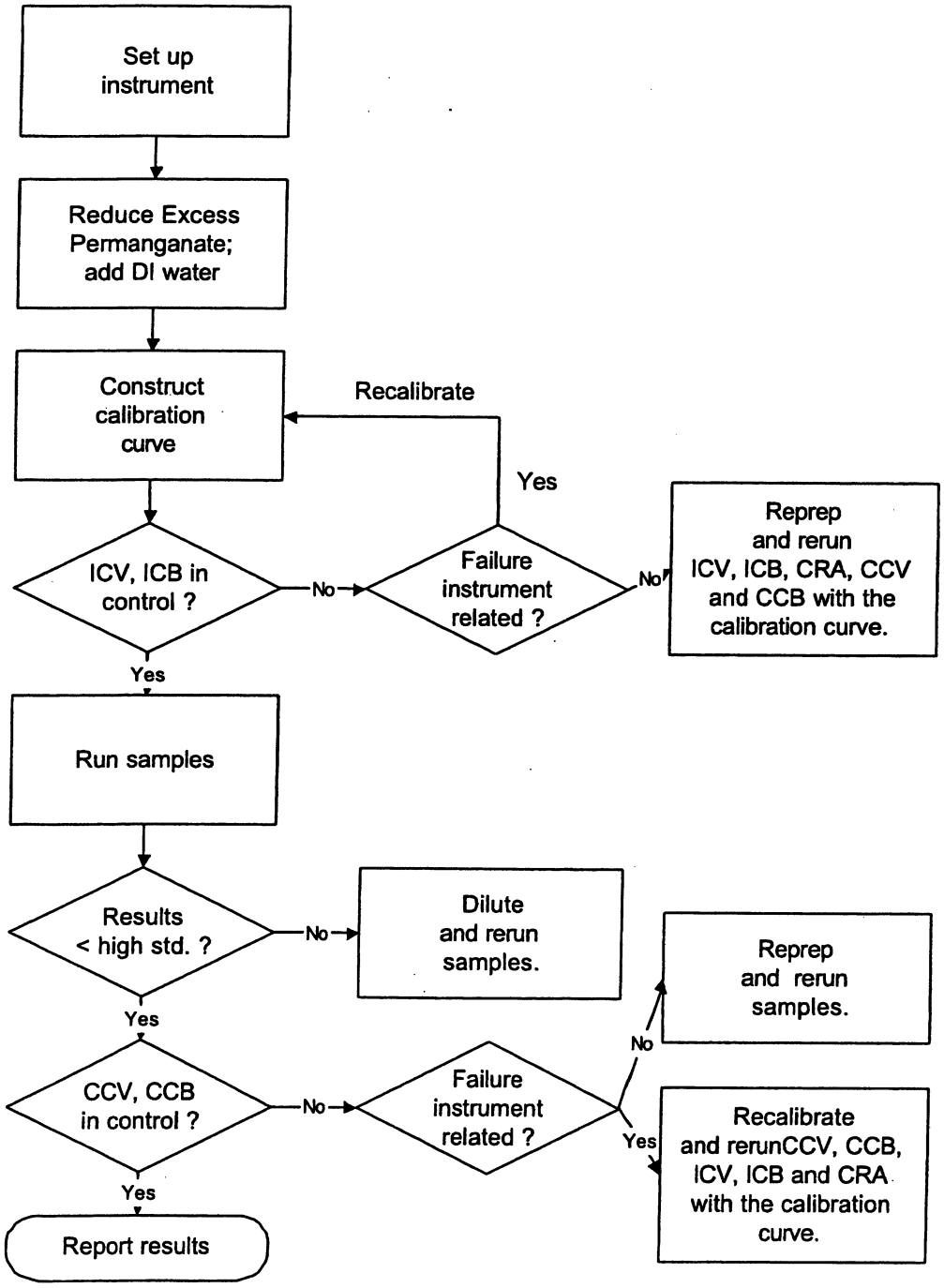


Figure 2. CVAA Mercury Analysis



APPENDIX A

TABLES

TABLE I. MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC STANDARD AND SPIKING LEVELS

Soil RL (mg/kg)	0.1
Std 0 (mg/L)	0
Std 1 (mg/L)	0.0002
Std 2 (mg/L)	0.0005
Std 3 (mg/L)	0.001
Std 4 (mg/L)	0.005
Std 5 (mg/L) **	0.010
ICV (mg/L)	0.001 or 0.0025 ***
CCV/LCS (mg/L)	0.0025 or 0.005 ***
MS (mg/L)	0.001

* **SOP specified calibration levels must be used unless prevented by the instrument configuration or client specific requirements. Deviations from specified calibration levels must be documented in the facility specific instrument operation SOP and must be approved by the facility technical manager and Quality Assurance Manager.**

** **Optional standard which may be used to extend the calibration range as allowed by the instrument configuration. If the instrument configuration prevents the use of 6 standards, the 2 ppb standard may be eliminated in favor of the 10 ppb standard.**

*** **Concentration level dependent on high calibration standard used. CCV must be 50% of the high standard concentration and the ICV must be 20-25% of the high standard concentration.**

TABLE II. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run.	90-110 % recovery.	Terminate analysis; Correct the problem; Recalibrate or reprep with calibration curve (see Section 9.7).
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate or reprep with calibration curve (see Section 9.7).
CCV	Every 10 samples and at the end of the run.	80 - 120 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep with calibration curve (see Section 9.8).
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB or reprep with calibration curve (see Section 9.8).
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL do not require redigestion (See Section 9.4)	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.4 for additional requirements.

*See Sections 11.2.10 and 11.2.11 for exact run sequence to be followed.

TABLE II. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits.	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS (see Section 9.5).
Matrix Spike	One per sample preparation batch of up to 20 samples.	75 - 125 % recovery or in-house control limits. If the MS/MSD is out for an analyte, it must be in control in the LCS.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. (see Section 9.6) For TCLP see Section 11.3.12
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery or in-house control limits; RPD ≤ 20%. (See MS)	See Corrective Action for Matrix Spike.

APPENDIX B

EXAMPLE
STL NORTH CANTON Hg DATA REVIEW CHECKLIST

Example
STL North Canton Hg Data Review Checklist

Run/Project Information

Run Date: _____ **Analyst:**_____ **Instrument:**_____
Prep Batches Run:_____

Circle Methods used: **7470A / 245.1 :** CORP-MT-0005 Rev 1 **7471 / 245.5 :** CORP-MT-0007 Rev 1
CLP - AQ : CORP-MT-0006 Rev 0 **CLP - SOL :** CORP-MT-0008 Rev 0

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2ndLevel
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits?				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?				
4. CRA run (CLP only)?				
B. Sample Results				
1. Were samples with concentrations > the high calibration standard diluted and reanalyzed?				
2. All reported results bracketed by in control QC ?				
3. Sample analyses done within holding time?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/MDL data on file?				
3. Calculations and Transcriptions checked for error ?				
4. All client/ project specific requirements met?				
5. Date of analysis verified as correct ?				

Analyst: _____ **Date:**_____
Comments:
_____
_____
_____

2nd Level Reviewer : _____ **Date:**_____
_____
_____
_____

APPENDIX C

MSA GUIDANCE

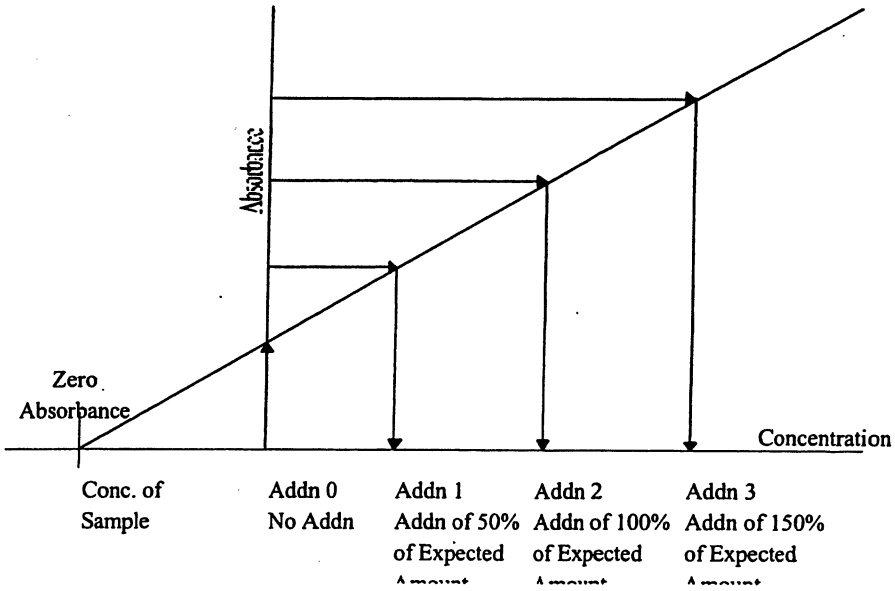
APPENDIX C. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.
- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX D

TROUBLESHOOTING GUIDE

APPENDIX D. TROUBLESHOOTING GUIDE

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Wrong lamp Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak EDL power supply set on "Continuous"
Erratic Readings	Source lamp not aligned properly Lamp not prewarmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
EDL Won't Light	Lamp cable not plugged in Lamp power set at 0 Lamp is dead Power supply fuse is blown Short in cord
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell
Background Correction Light Blinking	Background screen or attenuator faulty

APPENDIX E

CONTAMINATION CONTROL GUIDELINES

APPENDIX E. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

APPENDIX F

PREVENTIVE MAINTENANCE

APPENDIX F. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption (Leeman PS 200) [1]

Daily	Semi-annually	Annually
Clean lens.	Check Hg lamp intensity.	Change Hg lamp.
Check aperture.		Check liquid/gas separator.
Check argon flow.		
Check tubing.		
Check drain.		
Replace drying tube.		

Cold Vapor Atomic Absorption (PE 5000) [1]

Daily	Monthly
Clean aspirator by flushing with DI water.	Clean cell in aqua regia.
Check tubing and replace if needed.	Clean aspirator in aqua regia.
Clean windows with methanol.	
Change silica gel in drying tube.	
Check argon gas supply.	
Adjust lamp.	

APPENDIX G

INSTRUMENT SET UP

Hg Analysis (Leeman PS200II)

SYSTEM INITIALIZATION AND WARM UP

1. F1 Menu
2. Instrument

a. Taskmaster

b. #4 Wake System Up Enter

The warming up period takes approximately 10 minutes.

TO SET UP INSTRUMENT FOR ANALYSIS

1. F1 Menu
2. Autosampler
   A. Rack Entry
   B. Edit (ex. Rack 1), Enter
   C. Cup ID - Enter (clears sample #'s)
   D. Extended ID- type in matrix of sample (water or solid), Enter.
   E. Press Insert Key and move cursor with arrows to cup ID and begin typing labels.
   F. F3 Print Screen
3. Press F2 Macro key and type in analyst's first name - Enter
   A. Enter folder name - ex. HG0306, Enter. If folder does not exist, type Y - Enter.
   B. Type in - "Rack 1", "Rack 2" etc. , Enter.

D. Type in : FROM CUP TO CUP

Ex. = 1 30

Do the same for position 2 if needed. If not needed, you must press Enter 3 times to begin analysis.

LABORATORY-SPECIFIC

QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: TOTAL ORGANIC CARBON (TOC) AND TOTAL INORGANIC CARBON (TIC)

(SUPERSEDES: REVISION 1, DATED 11/20/96)

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Approved by: [signature] 6-22-99
Laboratory Director Date

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Total Organic Carbon (TOC) or Total Inorganic Carbon (TIC) in waters and similar matrices. It is based on SW846 Method 9060 and EPA Method 415.1. The working linear range is instrument dependent at 1 mg/L to 50 mg/L with a reporting limit of 1 mg/L.

1.2. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

1.3. QuantIMS method codes are I8 (415.1 TIC), AY (9060 TIC), DA (415.1 – TOC) FM (9060 – TOC).

2. SUMMARY OF METHOD

2.1. Organic carbon is converted to carbon dioxide (CO_2) using chemical oxidation. The CO_2 is then measured by an infrared detector.

3. DEFINITIONS

3.1. Refer to the glossary in the Quality Assurance Management Plan (QAMP), current version.

4. INTERFERENCES

4.1. Contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts may cause Method interferences. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Carbonate and bicarbonate interfere but are eliminated by the acidification and purging step of the instrument.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfies ANSI Z87.1 (per the Chemical Hygiene Plan), Laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazard is known:

5.3.1. The following materials are known to be **corrosive:** Phosphoric **Acid, Sulfuric Acid.**

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable,** therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in/a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. O•I Corporation Model 1010 TOC Analyzer with 1051 vial multisampler and Printer

6.2. Nitrogen Gas and Regulator

6.3. Volumetric flasks: Various sizes

6.4. Volumetric pipettes: Various sizes

6.5. Vials: 40 mL glass

6.6. Graduated cylinders: Various sizes

6.7. pH Strips

6.8. Whatman filter #4

6.9. Top loading balance: capable of accurately weighing ± 0.01 g

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Sodium Persulfate: Reagent Grade

7.1.2. Sodium Persulfate Solution: Add 200 g sodium persulfate ($Na_2S_2O_8$) to a 1 liter volumetric flask and dilute to volume with reagent water.

7.1.3. Phosphoric Acid, concentrated: Reagent Grade

7.1.4. Phosphoric Acid Solution: Carefully add 59 mL concentrated phosphoric acid (H_2PO_4) to 900 mL of reagent water in a 1 liter volumetric flask. Dilute to volume with reagent water.

7.1.5. Sulfuric Acid, concentrated: Reagent Grade

7.2. Standards

7.2.1. All standards should be prepared in volumetric flasks, using volumetric pipettes, and diluted to volume with reagent water.

7.2.2. TOC Stock Standard

7.2.2.1. Primary and secondary sources are needed.

7.2.2.2. TOC 1000 mg/L

7.2.2.2.1. Dilute 1.06 g KHP (potassium acid phthalate) to volume in a 500 mL volumetric flask.

7.2.2.3. Prepare every six months.

7.2.3. TOC Calibration Standards

7.2.3.1. Prepare the following standards from the primary stock standard described in Section 7.2.2.2.1.

Concentration (mg/L)	Volume (mL)	Stock Concentration (mg/L)	Final Volume (mL)
50	5	1000	100
25	2.5	1000	100
10	1	1000	100
1	0.1	1000	100

7.2.4. TOC Verification Standard (LCS, MS/MSD and CCV)

7.2.4.1. Prepare the following standard from the secondary stock standard described in Section 7.2.2.2.1.

Concentration (mg/L)	Volume (mL)	Stock Concentration (mg/L)	Final Volume (mL)
25	12.5	1000	500

7.2.4.2. Prepare when setting up run.

7.2.5. TIC Stock Standard 1000 mg/L

7.2.5.1. Add 3.5 g Na_2HCO_3 (sodium bicarbonate) and 4.418 g Na_2CO_3 (sodium carbonate) to a 1000 mL flask. Dilute to volume with reagent water.

7.2.5.2. Prepare every 3 months.

7.2.6. TIC Calibration Standard

7.2.6.1. Prepare in the same manner as the TOC calibration standard (Section 7.2.3.1), using the TIC stock standard described in Section 7.2.5.1.

7.2.6.2. Prepare weekly.

7.2.7. TIC Verification Standard (LCS, MS/MSD and CCV)

7.2.7.1. Prepare in the same manner as the TOC verification standard (Section 7.2.4.1), using the TIC stock standard described in Section 7.2.5.1.

7.2.7.2. Prepare daily.

8. SAMPLE PRESERVATION AND STORAGE

8.1. Samples are preserved to a pH < 2 with sulfuric acid (H_2SO_4) or hydrochloric acid (HCl) and stored in plastic or glass containers at 4°C ± 2° C.

8.2. The holding time is twenty-eight days from sampling to analysis.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples, excluding QC samples (LCS, MS, MSD, and Method Blanks), which are processed similarly with respect to the procedure. All samples within the batch must be treated with the same lots of reagents and the same processes. All samples within the batch must be treated with the same processes.

9.2. Method Blank (MB)

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination

of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.2. A method blank consisting of 40 mL of reagent water and all reagents added to the samples must be prepared and analyzed with each batch of samples. The method blank is used to identify any background interference or contamination of the analytical system, which may lead to the reporting of elevated concentration levels or false positive data.

9.2.3. Corrective Action for Blanks

9.2.3.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be taken in consultation with the client and must be addressed in the project narrative.**

9.2.3.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

9.3. Laboratory Control Sample

9.3.1. Laboratory Control Samples are well characterized; laboratory generated samples used to monitor the laboratory's day to day performance of routine analytical methods. The LCS is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.3.2. A 25 mg/L LCS (TOC or TIC) must be analyzed with each batch of samples.

9.3.3. Corrective Action for LCS

9.3.3.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.3.1.1. The only exception to the requirement established in section 9.3.3.1 occurs if the LCS recovery is biased high and the associated sample(s) are ND (non detect). Under this condition, the batch is acceptable.

9.3.3.2. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. An MS/MSD consisting of 20 mL of sample and 20 mL of the 25 mg/L standard will be analyzed with each analytical batch of samples.

9.4.3. Corrective action for MS/MSDs

9.4.3.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

9.4.3.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported as DIL (diluted out).

9.4.3.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.5. QC Acceptance Criteria

9.5.1. Control limits are established by the laboratory as described in NC-QA-0018.

10. CALIBRATION AND BLANKING

10.1. Recommended Initial Setup

Contant Settings		
STD Mass	=	6.76 ug C
Sample Vol	=	2.0 mL
Acid Vol	=	4 x 100 uL
Oxidant Vol	=	10 x 100 uL

10.1.1. Adjust the nitrogen to 30 psi using the flow valve on the tank. The gauge should always be set at 30 psi when the instrument is not in use.

10.1.1.1. If the main valve reads < 200 psi, the tank should be changed. **Do not** allow the tank to go empty.

10.1.2. Remove the reagent bottles and fill with appropriate reagents (phosphoric acid solution and sodium persulfate). Do not fill bottles completely full; leave a small amount of air space. Loosely reconnect caps (and tubing), and replace the bottles into the instrument.

10.1.3. Blank and calibrate the instrument when CCVs and/or CCBs fail to meet acceptance criteria or when other problems are encountered.

10.1.3.1. Choose "sequences" from the "databases" menu option, open up the calibration template.

10.1.3.2. Confirm all information. If blanking is required, it is best if done at this step. Enter the desired number of blanks (no less than five) in the "reagent blanks before" field.

10.1.3.3. Analyze an ICV/ICB

10.1.3.4. Save the file using the current date as the filename.

10.1.3.5. Update data file information:

10.1.3.5.1. Choose the setup menu option, go into win TOC output, change the log fill name and prefix counter.

10.1.3.6. When analysis is complete, print the run from "/utilities/view run log"

10.1.3.7. Evaluate the data. The correlation coefficient of the original curve must be ≥0.995 or recalibration is required.

10.2. Continuing Calibration

10.2.1. The run is checked at the beginning, after every ten samples, and at the end of the run of the same species (TOC or TIC) using a midrange CCV made from a secondary source (Section 7.2.4 or 7.2.7) to verify continued linearity. A CCV cannot vary from the original curve by more than ± 10%, or recalibration is required.

10.2.2. System cleanliness is checked every ten samples and at the end of the run using a Continuing Calibration Blank (CCB). A CCB cannot contain the analyte of interest above the reporting limit, or recalibration is required.

10.3. Analytical Documentation

10.3.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

10.3.2. All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. The supervisor or designee reviews logbooks.

10.3.3. Documentation, such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs, is available for each data file.

10.3.4. Sample results and associated QC are entered into the Laboratory Information Management System (LIMS) after final technical review.

11. PROCEDURE

11.1. Sample Preparation Procedure

11.1.1. Check the pH of the sample. If the pH > 2, add concentrated H_2SO_4 drop by drop to the sample (in the TOC vial) until the pH is < 2.

11.1.2. If excess particulate matter exists, filter an aliquot of sample through a Whatman #4 filter into a TOC vial.

11.2. Preparation Documentation

11.2.1. Record any sample preparation on the analytical logsheet.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Sample Analysis Procedure

12.1.1. Type a run protocol sequence into the computer using the run template, if desired. Update the data file information in the setup/winTOC output.

12.1.1.1. For method 9060, quadruplicate analysis is required. If quadruplicate reporting is requested each of four results are reported. Replicate analysis should be taken from separate vials, if available. If only one reportable result is requested per sample, the four results should be taken from one vial, and the average of the four results are reported.

12.1.1.2. For method 415.1 only one analysis is required. The single analysis result is reported directly from the instrument printout.

12.1.2. All samples and standards should be poured into 40 mL vials. Samples received in vials can be run in those containers, provided there is not an excess of solids.

12.1.3. Be sure the samples are loaded on the sampler, the first one positioned under the needle.

12.1.4. Click on the "Start" button.

12.1.5. Samples that fall outside the linear range (> 100 mg/L) of the instrument must be diluted and reanalyzed.

12.1.5.1. Samples following a high sample should be re-analyzed if carryover is a concern.

12.1.6. Print the run from "utilities/view run log."

12.1.7. When analysis is complete, properly dispose of or put away samples and standards.

12.2. Calculations

12.2.1. ***Total Organic Carbon, mg/L = Average Instrument Value x Dilution***

Where:

TOC, mg/L = average of the 4 instrument values x dilution, calculated (without dilution) by the instrument.*

12.2.2. ***LCS % Recovery =*** $\dfrac{\dfrac{\textit{Average of 4 instrument values*}}{25} \textit{ x 100}}{}$

12.2.3. ***MS/MSD % recovery***

$$\frac{\dfrac{\textit{(Avg of 4 instrument values* MS or MSD) - (Avg sample instrument value* ÷ 2)}}{\textit{12.5}}}{100} \text{ x}$$

**One of the values may be judged erroneous and disregarded if three of the four are consistent. If no consistency can be found in the four values, the sample must be rerun.*

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. The group/team leader has the responsibility to ensure that an associate who has been properly trained in its use and has the required experience performs this procedure.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Acid waste must be collected in clearly labeled acid water containers.

15.2. Solid materials (gloves, soiled paper products) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.3. Refer to the Laboratory Sample and Waste Disposal plan.

15.4. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of Quanterra. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by annual refresher training.

16. REFERENCES

16.1. References

16.1.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Total Organic Carbon, Method 9060

16.1.2. EPA 600, Methods for Chemical Analysis of Water and Wastes, Organic Carbon, Method 415.1

16.1.3. Quanterra® Quality Assurance Management Plan (QAMP), current version.

16.2. Associated SOP

16.2.1. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Reporting limits

17.1.1. The lower reporting limit is 1 mg/L.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Troubleshooting guide

17.2.1. See the manufacturer's instructions for an instrument troubleshooting guide and maintenance requirements.

17.3. Deviations from the Method

17.3.1. A blender is not used to homogenize samples.

STL STANDARD OPERATING PROCEDURE

TITLE: CYANIDE PREPARATION METHOD

(SUPERSEDES: REVISION 8.1, DATED 03/20/00)

Prepared by: _____ 6/5/01 Date

Reviewed by: _____ 6/5/01 Date
Technology Specialist

Approved by: _____ 6/5/01 Date
Quality Assurance Manager

Approved by: _____ 6/5/01 Date
Environmental Health and Safety Coordinator

Approved by: _____ 6-5-01 Date
Laboratory Director

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Total, Amenable, and Free Cyanide in solids, liquids, and waters. It is based on CLP ILM03.3, CLP ILM04.0, SW 846 Method 9012A, EPA 335.1, 335.2, 335.4, Standard Method 4500-CN-I and ASTM D 4282-83. The working linear range is 0.005 - 0.1 mg/L for waters and 0.25 to 5 mg/kg for solids.

1.2. The associated method codes are CLP ILM03.0 (DV), CLP ILM04.0 (OU), SW846 Method 9012A (QP), ASTM D4282-83, EPA 335.1 and 335.2 (CG), EPA 335.4 (O2), and Standard Method 4500-CN-E (A4).

1.3. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

2. SUMMARY OF METHOD

2.1. The Cyanide, as HCN, is released by distilling/refluxing the sample with strong acid and is trapped in a sodium hydroxide solution.

3. DEFINITIONS

3.1. Refer to the glossary in the Laboratory Quality Manual (LQM), latest version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Oxyidizing agents such as chlorine will decompose most cyanides. Sulfide will distill over with the cyanide and could affect colorimetric, titrimetirc, and electrode procedures. Refer to the preparation section on how to screen and treat samples appropriately.

4.3. Aldehydes convert cyanide to cyanohydrin which could result in the lost of cyanide. If the presence of aldehydes are suspected, stabilize the sample with NaOH at the time of collection and add 2 mL 3.5% ethylenediamine solution per 100 mL of sample.

4.4. Refer to Standard Methods for further information on possible interferences and associated treatments.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

5.2. Eye protection that satisfies ANSI Z87.1 (per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. The following are known to be **corrosive: sulfamic acid, sodium hydroxide, sulfuric acid, acetic acid.**

5.3.2. Cyanide is a highly toxic compound that must be handled with care. The analyst must be aware of the handling and clean-up techniques before using this material.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable,** therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Cyanide Distillation Apparatus

6.2. Analytical balance, capable of accurately weighing ± 0.0001 g

6.3. Vacuum pump

6.4. Graduated cylinders: various

6.5. Volumetric flasks: various

6.6. Volumetric pipets: various

6.7. Balance: Top loading, capable of accurately weighing ± 0.01 g

6.8. Lead Acetate Indicator Paper

6.9. Potassium Iodide (KI) Indicator Paper

6.10. Erlenmeyer flasks: various

6.11. Buret: Class A 10 mL

6.12. pH strips

6.13. Boiling stones or chips

6.14. Beakers: various

6.15. Snap seal containers: 120 mL

6.16. Plastic bottles with lids: 250mL or 500mL

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Sulfamic Acid: reagent grade (not used for CLP ILM03.0)

7.1.2. Sulfamic Acid Solution: Add 100 g of sulfamic acid to 800 mL reagent water and dilute to 1 liter with reagent water (not used for CLP ILM03.0)

7.1.3. Ascorbic Acid: reagent grade

7.1.4. Sodium Hydroxide: (NaOH), high purity grade.

7.1.5. Sodium Hydroxide, 1.25 N: Add 50 g of NaOH to 900 mL and dilute to 1 liter with reagent water.

7.1.6. Sodium Hydroxide, 0.25 N: Add 10 g of to 900 mL reagent water and dilute to 1 liter with reagent water.

7.1.7. Sulfuric Acid: (H_2SO_4), concentrated

7.1.8. Acetic Acid Solution: Add 10 mL of glacial acetic acid to 90 mL of reagent water.

7.1.9. Magnesium Chloride: ($MgCl_2 \bullet 6H_2O$), reagent grade

7.1.10. Magnesium Chloride Solution: Add 510 g $MgCl_2 \bullet 6H_2O$ to 500 mL reagent water and dilute to 1 liter with reagent water.

7.1.11. Calcium Hypochlorite: [Ca $(OC1)_2$], reagent grade

7.1.12. Calcium Hypochlorite Solution: Add 5 g of $Ca(OC1)_2$ to 100 mL of reagent water.

7.1.13. Methyl Red Indicator: Add 0.05 g of methyl red to 50 mL of glacial acetic acid and dilute to 100 mL with reagent water.

7.1.14. Methyl red reagent grade glacial

7.1.15. Acetic Acid: (CH_3COOH), glacial reagent grade

7.1.16. Zinc Acetate: [$Zn(C_2H_3O_2)$], reagent grade

7.1.17. Zinc Acetate Solution: Add 100 g zinc acetate to 800 mL reagent water and dilute to 1 liter with reagent water.

7.1.18. Sodium Acetate: [$NaC_2H_3O_2 \bullet 3H_2O$] reagent grade

7.1.19. Sodium Acetate Buffer: Add 410 g of sodium acetate to 500 mL of reagent water. Adjust the pH to 4.5 using glacial acetic acid and dilute to 1 liter with reagent water.

7.1.20. Rhodanine: reagent grade

7.1.21. 0.0192 N Silver Nitrate: reagent grade

7.1.22. Bismuth nitrate [$Bi(NO_3)_3$]: Dissolve 30 g of $Bi(NO_3)_3$ in 100 mL of reagent water. While stirring, add 250 mL of glacial acetic acid. Stir until dissolved. Dilute to 1 L with reagent water.

7.1.23. Cadmium chloride [$CdCl_2$] – anhydrous : Dissolve 1.0 g $CdCl_2$ in 90 mL of reagent water. Dilute to 100 mL with reagent water to produce a 10 g/L $CdCl_2$ solution.

7.2. Standards

7.2.1. Secondary Source Cyanide Stock Standard, 1000 mg/L: Add 2.51 g of potassium ferricyanide (KCN) and 2.0 g of potassium hydroxide (KOH) to a 1000 mL volumetric flask and dilute to volume with reagent water. Mix well. Prepare fresh every 1 - 3 months.

7.2.2. Note: Cyanide stock standard must be standardized prior to use (See Section 7.3) Working Standards: Add the required amounts of 1.25 N NaOH and cyanide standards to volumetric flasks according to the suggested chart. Dilute to volume using reagent water. See chart below:

Working Standards					
Volume 1.25 N NaOH (mL)	Std Volume (mL)	Std Conc. (mg/L)	Final Volume (mL)	Final Conc. (mg/L)	Expiration Time (Days)
20	10	1000	100	100	14
20	10	100	100	10	7
20	10	10	100	1	1

7.3. Cyanide Standardization

7.3.2. Using a volumetric pipet, aliquot 10.0 mL of the 1000 ppm stock cyanide standard into an Erlenmeyer flask and add 90 mL of reagent water.

7.3.3. Add 10 drops of Rhodanine indicator.

7.3.4. Titrate with 0.0192 N Silver nitrate until the color changes from yellow to pink/orange.

7.3.5. Calculation:

$$\text{Cyanide, mg/L} = \frac{(A)\ (1000)}{\text{mL cyanide solution (10)}}$$

Where: A = *mL Titrant for standard*

7.3.6. If the cyanide concentration is not 1000 ppm, adjust concentration accordingly.

8. SAMPLE PRESERVATION AND STORAGE

8.1. Solid and liquid samples are not chemically preserved. Water samples are preserved with NaOH to a pH>12. All samples are stored at 4° C ± 2°C in plastic or glass containers.

8.2. The holding time is fourteen days from sampling to analysis.

NOTE: CLP samples holding time is twelve days from receipt to analysis.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same process.

9.2. Method Blank (MB)

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.2. A blank consisting of 50 mL reagent water must be prepared and distilled (or diffused, 3 mL) with each analytical batch of samples.

9.2.3. Corrective Action for Blanks

9.2.3.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.3.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. A midrange LCS consisting of 2.0 mL of the 1.0 mg/L standard (secondary source) brought to a final volume of 50 mL must be distilled with each analytical batch of samples.

9.3.3. Corrective Action for Laboratory Control Samples

9.3.3.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.3.2. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. A MS/MSD consisting of 50 mL or 1.0 g of sample brought up to 50 mL with reagent water and 2.0 mL of the 1.0 mg/L standard (either source) must be distilled with each analytical batch of samples.

9.4.2.1. **Note:** MS for CLP samples require 5 mL of 1.0 mg/l standard (true 100ug/L)

9.4.3. Corrective action for MS/MSDs

9.4.3.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

9.4.3.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data reported as DIL (diluted out).

9.4.3.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.5 Control Limits

9.5.1 Control limits are established by the laboratory as described in SOP, NC-QA-0018.

9.5.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are listed in the Laboratory Quality Manual (LQM) and the latest is version easily accessible via the LIMs (QC Browser program).

9.6 Method Detection Limits (MDLs) and MDL Checks

9.6.1 MDLs and MDL Checks are established by the laboratory as described in SOP, NC-QA-0021.

9.6.2 MDLs are listed in the Laboratory Quality Manual (LQM) and the latest version is easily accessible via the LIMs (QC Browser program).

9.7 Nonconformance and Corrective Action

9.7.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager/

10. CALIBRATION AND STANDARDIZATION

10.1. For all non-CLP methods, a low and high standard are distilled from the same source as the calibration curve each day. Prepare the low standard (0.025 mg/L) by diluting 0.125 mL of 10.0 mg/L standard with reagent water to a final volume of 50 mL. Prepare the high standard (0.075 mg/L) by diluting 0.375 mL of a 10.0 mg/L standard with reagent water to a final volume of 50mL. Method 9012A *recommends* that the distilled standards (a high and a low) be compared to the curve with a ±10% agreement with the undistilled standards. The distilled standards are evaluated against all applicable batch QC.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described

11.3 Summary

11.3.1. For all non-CLP methods, the sample is distilled/refluxed under acidic conditions for one hour. For CLP and EPA 335.4 methods, the samples are distilled for one and a half hours. The released HCN is trapped in 0.25 N NaOH solution.

11.4. Sample Preparation Procedure applicable for use with the Midi distillation unit.

11.4.1. Checking for Interferences (For non-CLP samples)

11.4.1.1. Using pH paper strips, check the pH of the sample and record it as >12 or <12 on the analytical logsheet. If pH is <12, the deviation **must be addressed in the project narrative.**

11.4.1.2.For ALL samples (non-CLP and CLP samples): Test each sample for the presence of sulfides using lead acetate paper. If sulfides are present, treat the sample with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution. Avoid a large excess of cadmium carbonate and long contact time in order to minimize loss by complexation or occlusion of cyanide on the precipitated material.

11.4.2. Complex Cyanide Preparation Procedure [THIS PROCESS IS PERFORMED ONLY WHEN REQUESTED BY THE CLIENT]: Add 100 mL of water sample or 2.0 g of solid sample and 100 mL reagent water to a plastic, sealable container. Add 10 g NaOH pellets. Mix or tumble for 12-16 hours. Allow solids to settle out. Transfer 50 mL of the solution to the distillation tube. Distill sample (See section 11.2.4.1).

Note: If the sample requires an MS/MSD or MS/Dup, double or triple the volumes as stated above. A blank and LCS should be prepared and analyzed with samples using 10 g NaOH and 100 mL reagent water.

11.4.3. Amenable Cyanide (Chlorinated Aliquot) [THIS PROCESS IS PERFORMED ONLY WHEN REQUESTED BY THE CLIENT]

11.4.3.1.Place 50 mL (waters) or 1.0 g (solids/liquids) into a beaker. Add 50 mL of reagent water to non-waters. Place the beaker on a stir plate and begin mixing. Test the pH of the solution, if less than 12 add 1.25 N NaOH, drop by drop until pH $\geq$ 12. Drop by drop, add calcium hypochlorite until an excess of chlorine is reached. Test for chlorine excess using KI paper. Allow the sample to chlorinate for one hour. At the end of the chlorination period, add about 0.1 to 0.5 g of ascorbic acid to destroy excess chlorine. Test using KI paper. Keep the addition of ascorbic acid to a minimum. Pour the sample into a distillation flask and follow the total cyanide preparation method (Section 11.2.4). Also set up an unchlorinated aliquot of sample (50 mL or 1.0 g) following the total cyanide method.

11.4.4. Total Cyanide

11.4.4.1. Add 50 mL or 1.0 g of the sample and 2 - 3 boiling chips to the distillation tube. Bring all volumes to 50 mL with reagent water. Add 50 mL of 0.25 N NaOH solution to the absorber tube and assemble the scrubber. Assemble the cyanide distillation apparatus.

NOTE: If solid samples are being prepared under the Michigan program, the initial solid weight is 2.5 g

11.4.4.2. Turn on the cooling water to "60". Turn on the vacuum source and adjust the flow such that even stream of air bubbles are in the scrubber tube approximately 1/4 " of foam or 1-2 bubbles per second. At this time add any spiking solutions to the LCS or MS/MSDs samples directly into the inlet tube.

11.4.4.3. In the order stated, add 2.0 mL sulfamic acid solution (not for CLP ILM03.0 samples), 2.5 mL of concentrated sulfuric acid and 2.0 mL of magnesium chloride solution to the distillation tube. Be sure to rinse the inlet tube sparingly with reagent water between and after reagent additions.

Note: Complex cyanide samples require an additional 2.5 mL of sulfuric acid. Be sure the pH is < 2.

11.4.4.4. Flip the heater switch to "on" and turn the dial to an appropriate setting (according to manufacturer's instructions) to allow the apparatus to warm up. Be sure to adjust the air flow and water as necessary. Heat for one hour. After the heating period, the heater will turn off automatically. Allow to cool for fifteen minutes. Keep the vacuum and cooling water on.

NOTE: For CLP, heat samples for one and a half hours.

11.4.4.5. Disconnect the absorber. Pour solution into a 120 mL snap seal container. Do not rinse the scrubber tube or dilute NaOH in the snap seal. Be sure to properly label the bottles "total" or "amenable" along with sample ID, position and date.

11.4.5. Free Cyanide (Weak and Dissociable) [THIS PROCESS IS PERFORMED ONLY WHEN REQUESTED BY THE CLIENT]

11.4.5.1. Add 50 mL or 1.0 g of sample to the distillation tube. Bring all volumes up to 50 mL with reagent water. Add 50 mL of 0.25 N NaOH to the absorber tube and assemble.

11.4.5.2. Turn on cooling water to "60". Turn on the vacuum source. Add any spiking standards to the appropriate LCS or MS/MSDs at this time. Through the inlet tube, add 1.0 mL of sodium acetate buffer, 1.0 mL of zinc acetate, and 0.25 mL of methyl red indicator. Rinse the inlet tube sparingly with reagent water between and after reagent additions. If the sample is not red, carefully add 1:9 acetic acid dropwise until the sample does turn red.

11.4.5.3. Flip on the heater switch and turn the dial to "10.5" (This allows the apparatus to warm up). Adjust the air flow and water as needed. Heat for 1 hour. After the

heating period, the heater will shut off automatically. Allow the sample to cool for fifteen minutes with the air and water on.

11.4.5.4. Pour the scrubber contents into a 120 mL snap seal bottle. Do not rinse the scrubber. Label the bottle well and be sure to denote that it is a "free" sample distillate.

11.4.6 Cyanide Unit Clean-up

11.4.6.1 Cyanide distillation unit glassware is very fragile and expensive. It must be handled with care at all times.

11.4.6.2 Disassemble each set-up and rinse the sample down the drain with large amounts of water. Be sure to collect the solids in a screen and dispose of properly.

11.4.6.3 Wash each set-up with soap and hot water, and then rinse with 1:1 HCl. Rinse several times with reagent water. **After preparing samples found to be extremely high in cyanide, a subsequent rinse with 1.0N NaOH, followed by further rinsing with reagent water can help rinse away any residual cyanide.

11.4.6.4 Re-assemble the set-up.

11.4.6.5 Be sure to wash each set up as a separate unit and replace in the same position.

11.4.6.6 If a sample of known high cyanide concentration was distilled in a certain position, be sure to change the appropriate tubing on that position.

11.5 Analytical Documentation

11.5.1 Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.5.2 All standards are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3 Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4 Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files

14. POLLUTION PREVENTION

14.1. Using a Midi Cyanide distillation unit saves time and requires less sample and reagents for use.

15. WASTE MANAGEMENT

15.1. Acid waste must be collected in clearly labeled acid waste cans.

15.2. Solid waste materials (soil, gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.3. Refer to the Laboratory Sample and Waste Disposal plan

15.4. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1. References.

16.1.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Total and Amenable Cyanide, Automated UV; Method 9012A.

16.1.2. EPA 600; Cyanide, Total and Cyanide, Amenable to Chlorination; Methods 335.1 and 335.2

16.1.3. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition; Weak and Dissociable Cyanide; Method 4500-CN-I

16.1.4. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition; Complex Cyanide; Method 4500-CN-E

16.1.5. USEPA CLP SOW ILM03.0 Section D-Cyanide Midi Distillation

16.1.6. Corporate Quality Management Plan (QMP), current version

16.1.7. STL Laboratory Quality Manual (LQM), current version

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2 Glassware Washing, NC-QA-0014

16.2.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4 Method Detection Limits and Instrument Detection Limits, NC-QA-0021

16.2.5 Navy/Army SOP, NC-QA-0016

16.2.6 Cyanide, Automated Method NC-WC-0031

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. BASF RFI Requirements

17.1.1. Amendments to this SOP to satisfy BASF RCRA Facility Investigation (RFI) requirements are based on comments from US EPA upon review of site requirements and laboratory procedures.

17.1.2. Amendment to the preparation of **all** samples

17.1.2.1. **Section 7.2.1**: Change 2.51 g of potassium cyanide to 2.11g potassium ferricyanide.

17.1.2.2. **Section 9.4**: The matrix spike/duplicate frequency has been increased from one per batch to one per 7 samples. Consult with the project manger for specific samples to be spiked. The goal is to spike each sampling area at the site at least once.

17.1.3. Amendment to the preparation of **Prussian blue area** samples

17.1.3.1. **Section 7.1.22**: Sodium thiocyanate (reagent grade): Prepare a spiking solution at a concentration 100X greater than the measured cyanide for the appropriate Prussian blue area samples.

17.1.3.2. **Section 9.5**: Three Prussian blue area samples will be spiked with 1 mL of the sodium thiocyanate solution (Section 17.1.3.1) in order to assess the potential impact of thiocyanate in the original samples on the measured cyanide concentration. These matrix spikes do not replace the cyanide matrix spikes mentioned above (Section 17.1.2.2). The increase (if any) in measured cyanide concentration relative to the original sample shall be reported as "percentage increase" using the following equation:

$$\%CN_{increase} = \frac{Conc_{SCN_spike} - Conc_{unspiked}}{Conc_{unspiked}} \times 100$$

where

$Conc_{SCN_spike}$ = *cyanide concentration measured in the thiocyanate spiked sample*

$Conc_{unspiked}$ = *cyanide concentration measured in the unspiked sample*

Note: Section 11.3.2.1 of NC-WC-0031 addresses sulfide testing and precipitation.

17.2. Method Deviation (9012A, 335.1, 335.2)

17.2.1. The reflux distillation apparatus used is the midi distillation.

17.2.2. The volume of sample used is reduced to 50 mL vs. 500 mL using the midi-distillation apparatus.

17.2.3. Method of Standard Addition is not performed for samples with matrix interference (sulfides).

APPENDIX A

Cyanide Distillation Form

(Example)

APPENDIX A

N:\WET-LOG\CYANIDE

QUANTERRA- NORTH CANTON
CYANIDE DISTILLATION LOGSHEET

PAGE 1 OF 2

Batch No. 0040131 waters 0040130 leach
Prep/Anal. Date 000207 000209
Start Time: 2 30 pm

LCS&MS/MSD No. CY0117
LCS Prep Date: 000208
End Time: 3:30 pm

SDG No. —
Analyst: MFG

Pos.	Sample No.	Type T	C	F	Sample Volume	Final Volume(mL)	pH >12	S	Cl2	Comments
1A	BLK	T	C	F	50mL	50mL	—			D8CDF B/C
2	LCS	T	C	F			—			2ml CY0117 1ppm 2°
3	LCSD	T	C	F			—			1 D8CDE 0040130
4	.025	T	C	F			—			.125ml CY0105 10ppm 1°
5	.075	T	C	F			—			.375ml CY0105 10ppm 1°
6	D833X	T	C	F			✓	✓	✓	
7	D833X MS	T	C	F			—			2ml CY0117 1ppm 2°
8	D833X MSD	T	C	F			—			" "
*9	D851G MS	T	C	F			—			" "
*10	D851G MSD	T	C	F			—			" "
*1A	D851G	T	C	F			✓	✓	✓	
*2	D851K	T	C	F			✓	✓	✓	
*3	leach blk	T	C	F			✓	✓	✓	D8CDE 1.01 0040130
4	D81MG	T	C	F			✓	✓	✓	
5	D81MK --	T	C	F			✓	✓	✓	
6	D81ML	T	C	F			✓	✓	✓	
7	D83RM	T	C	F			✓	✓	✓	
8	D833H	T	C	F			✓	✓	✓	
9	D833P	T	C	F			✓	✓	✓	
10	D83RI	T	C	F			✓	✓	✓	

Type: T=Total C= Chlorinated F= Free

S and Cl2 column- a check mark in these columns means the sample was checked for this interference.

If the interference is present, the analyst will address this in the comments section.

* leaches came down on 2.4.00

Level Review X
Calibration
Calculation
Date: 2.8.00

Level Review X
Calibration
Calculation
Date: 2.8.00

Rev 5-16-97

APPENDIX A

N:\WET-LOG\CYANIDE

QUANTERRA- NORTH CANTON
CYANIDE DISTILLATION LOGSHEET

PAGE 2 OF 2

Batch No. 0040131 waters 0040130 leaches
Prep/Anal. Date 000208 / 000209
Start Time: 2:30 pm

LCS&MS/MSD No. CR017
LCS Prep Date: 000208
End Time: 3:30pm

SDG No. —
Analyst: MFG

Pos.	Sample No.	Type T C F	Sample Volume	Final Volume(mL)	pH >12	S	Cl2	Comments
B 1X	D83R6	(T) C F	50 mL	50mL	✓	✓	✓	
2	D83R8	(T) C F			✓	✓	✓	
3	D856F	(T) C F			✓	✓	✓	
4	D856Q	(T) C F			✓	✓	✓	
5		T C F						
6		T C F						
7		T C F						
8		T C F						
9		T C F						
10		T C F						
1B		T C F						
2		T C F						
3		T C F						
4		T C F	MFG 000208					
5		T C F						
6		T C F						
7		T C F						
8		T C F						
9		T C F						
10		T C F						

Type: T=Total C= Chlorinated F= Free
S and Cl2 column- a check mark in these columns means the sample was checked for this interference.
If the interference is present, the analyst will address this in the comments section.

Level Review X
Calibration
Calculation
Date 2-8-00

Level Review X
Calibration ✓
Calculation ✓
Date 2-9-00

Rev. 5-16-97

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Implementation Date: 10/16/00

SOP No: NC-WC-0084
Revision No: 3
Revision Date: 10/03/00

STL STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPHY

(SUPERSEDES: REVISION (2))

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Date

Reviewed by: Kurt C. Lee 10/5/00
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Approved by: Gail Ann Poh 10/5/00
Quality Assurance Manager Date

Approved by: [signature] 10-10-00
Environmental Health and Safety Coordinator Date

Approved by: Christy A Grandi 10/11/00
Laboratory Director Date

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1. SCOPE AND APPLICATION

1.1. This method covers the determination of fluoride, chloride, nitrite, bromide, nitrate, ortho-phosphate and sulfate in drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, solids (after extraction 11.7) and leachates (when no acetic acid is used).

1.2. A listing of associated LIMs method codes is located in Section 8.2.

1.3. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. A 25 uL volume of sample is introduced into the ion chromatograph. The sample is pumped through three different ion exchange columns and into a conductivity detector. The first two columns, a precolumn or guard column and a separator column, are packed with low-capacity, strongly basic anion exchange resin. Ions are separated into discrete bands based on their affinity for the exchange sites of the resin. The last column is a suppresser column that reduces the background conductivity of the eluent to a low or negligible level and converts the anions in the sample to their corresponding acids. The separated anions in their acid form are measured using an electrical conductivity cell. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a calibration curve generated from known standards.

3. DEFINITIONS

3.1. Refer to the glossary in the Laboratory Quality Manual (LQM).

4. INTERFERENCES

4.1. Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.

4.2. The water dip or negative peak that elutes near, and can interfere with, the fluoride peak can usually be eliminated by the addition of concentrated eluent to each standard and sample.

4.3. Method interferences may be caused by contaminants in the reagent water, reagents, glassware and other sample processing apparatus that lead to discrete artifacts or an elevated baseline in the ion chromatograms.

4.4. Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other small organic anions. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant; however, it is the responsibility of the user to generate precision and accuracy information in each sample matrix.

4.5. The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

5.2. Eye protection that satisfied ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore; unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation when possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Balance -- Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.2. Ion Chromatograph -- Analytical system complete with ion chromatograph and all required accessories including analytical columns, compressed gases and detectors.

6.2.1. Anion guard column: A protector of the separator column. If omitted from the system the retention times will be shorter. Usually packed with same substrate as the separator column. 4 x 50 mm, Dionex IonPac AG14 P/N 46134, or equivalent.

6.2.2. Anion separator column: The separation shown in Figure 1 was generated using a Dionex IonPac AS14 column (P/N 46134). Equivalent column may be used if comparable resolution is obtained, and the requirements of Sect. 9.2 can be met.

6.2.3. Anion suppresser device: Dionex anion micro membrane suppresser (P/N 37106) or ASRS-Ultra Self-Regenerating Suppressor (4mm) P/N 53946 or equivalent.

6.2.4. Detector -- Conductivity cell: approximately 1.25 uL internal volume, Dionex, or equivalent.

6.2.5. Dionex –PeakNet 5.1Data Chromatography Software or equivalent.

6.3. Assorted laboratory glassware (pipettes, volumetric flasks, etc.).

7. REAGENTS AND STANDARDS

7.1. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2. Reagent water: Distilled or deionized water, free of the anions of interest. Water should contain particles no larger than 0.20 microns.

7.3. Eluent solution: sodium bicarbonate (CASRN 144-55-8) 1.0 mM, sodium carbonate (CASRN 497-19-8) 3.5 mM. Dissolve 1.680 g sodium bicarbonate ($NaHCO_3$) and 7.417 g of sodium carbonate (Na_2CO_3) in reagent water (7.2) and dilute to 100 mL in a volumetric flask. Take 10 mL of this concentrated eluent solution and dilute to 2 L for use as the working eluent solution or dissolve the entire bicarbonate/carbonate amount in 20 L of reagent water.

7.4. Stock solutions (1,000 mg/L): All stocks may be prepared as described below or purchased from commercial sources. Primary and secondary sources are required for each target analyte.

7.4.1. Fluoride stock solution (1.00 mL = 1.00 mg F^-): In a 1 liter volumetric flask, dissolve 2.2100 g of sodium fluoride (NaF) in reagent water, and dilute to volume with reagent water. Store in chemical- resistant glass or polyethylene.

7.4.2. Chloride stock solution (1.00 mL = 1.00 mg Cl^-): Dry sodium chloride (NaCl) for 12 hours at 105°C, and cool in a desiccator. In a 1 liter volumetric flask, dissolve 1.6485 g of the dry salt in reagent water and dilute to volume with reagent water.

7.4.3. Nitrite stock solution (1.00 mL = 1.00 mg NO_2^- - N): Place approximately 10.0 g of sodium nitrite (KNO_2) in a 125 mL beaker and dry to constant weight (about 24 hours) in a desiccator. In a 1 liter volumetric flask, dissolve 6.0790 g of the dried salt in reagent water and dilute to volume with reagent water. Store in a sterilized glass bottle. Refrigerate and prepare monthly.

- Nitrite is easily oxidized, especially in the presence of moisture, and only fresh reagents are to be used.
- Prepare sterile bottles for storing nitrite solutions by heating for 1 hour at 170°C in an air oven.

7.4.4. Bromide stock solution (1.00 mL = 1.00 mg Br^-): Dry approximately 5.0 g of sodium bromide (NaBr) for 12 hours at 105°C, and cool in a desiccator. In a 1 liter volumetric flask, dissolve 1.2876 g of the dried salt in reagent water and dilute to volume with reagent water.

7.4.5. Nitrate stock solution (1.00 mL = 1.00 mg NO_3^- - N): Dry approximately 10.00 g of sodium nitrate (KNO_3) at 105°C for 24 hours . In a 1 liter volumetric flask, dissolve 7.2200 g of the dried salt in reagent water and dilute to volume with reagent water.

7.4.6. Phosphate stock solution (1.00 mL = 1.00 mg PO_4 - P): Dry approximately 10.00 g of potassium dihydrogen phosphate (KH_2PO_4) for 1 hour at 105°C and cool in a desiccator. In a 1 liter volumetric flask, dissolve 4.3937 g of the dry salt in reagent water and dilute to volume with reagent water.

7.4.7. Sulfate stock solution (1.00 mL = 1.00 mg SO_4^{-2}): Dry approximately 5.00 g of potassium sulfate (K_2SO_4) at 105°C for 1 hour and cool in a desiccator. In a 1 liter volumetric flask, dissolve 1.8141 g of the dried salt in reagent water and dilute to volume with reagent water.

7.4.8. Commercial stock solution A: F^- - 25 mg/L, Cl^- - 500 mg/L, Br^- - 100 mg/L, NO_3^- - N- 25 mg/L, PO_4 – P - 25 mg/L, SO_4^{-2} - 500 mg/L

7.4.9. Commercial stock solution B: NO_2^- - N- 25 mg/L

7.4.10. Commercial IC Spike solution A: : F^- - 125 mg/L, Cl^- - 2500 mg/L, Br^- - 500 mg/L, NO_3^- - N- 125 mg/L, PO_4 – P - 125 mg/L, SO_4^{-2} - 2500 mg/L

7.4.11. Commercial IC Spike solution B: NO_2^- - N- 125 mg/L

7.5. Working standards: Prepare calibration standard #5 in a 10 mL volumetric flask and transfer to a vial. Adjust the amount of stock solution used to prepare the working standards if the stock concentration differs from 1000 mg/L as assumed Alternatively prepare Cal standard #5 by mixing 4.0 mL commercial stock A, 4.0 mL commercial stock B and 2.0 mL of reagent water.

Calibration Standard #5

Analyte	mL of Stock	Final Conc.
Fluoride	0.10mL	10.0 mg/L
Chloride	2.0 mL	200. mg/L
Nitrite	0.10 mL	10.0 mg/L
Bromide	0.40 mL	40.0 mg/L
Nitrate	0.10 mL	10.0 mg/L
Ortho-Phosphate	0.10 mL	10.0 mg/L
Sulfate	2.0 mL	200. mg/L

7.5.1. In 5 mL PolyVials prepare the following calibration standards in reagent grade water. Final concentrations of working standards are shown below.

Calibration Standard #4: take 2.50 mL of calibration standard #5 and add 2.50 mL of reagent water.

Calibration Standard #2: take 250 µL of calibration standard #5 and add 4.75 mL of reagent water.

Calibration Standard #1: take 25.0 µL of calibration standard #5 and add 4.95 mL of reagent water.

Calibration Standard #3: take 1.25 mL of calibration standard #5 and add 3.75 mL of reagent water.

Calibration Standard #1

Analyte	25.0 µL of Cal Std #5	Final Conc
Fluoride		0.05 mg/L
Chloride		1.0 mg/L
Nitrite		0.05 mg/L
Bromide		0.20 mg/L
Nitrate		0.05 mg/L
Ortho-Phosphate		0.05 mg/L
Sulfate		1.0 mg/L

Calibration Standard #2

Analyte	250 µL of Cal Std #5	Final Conc.
Fluoride		0.5 mg/L
Chloride		10. mg/L
Nitrite		0.5 mg/L
Bromide		2.0 mg/L
Nitrate		0.5 mg/L
Ortho-Phosphate		0.5 mg/L
Sulfate		10. mg/L

Calibration Standard #3

Analyte	1.25 mL of Cal Std #5	Final Conc.
Fluoride		2.5 mg/L
Chloride		50. mg/L
Nitrite		2.5 mg/L
Bromide		10. mg/L
Nitrate		2.5 mg/L
Ortho-Phosphate		2.5 mg/L
Sulfate		50. mg/L

Calibration Standard #4

Analyte	2.5 mL of Cal Std #5	Final Conc.
Fluoride		5.0 mg/L
Chloride		100 mg/L
Nitrite		5.0 mg/L
Bromide		40. mg/L
Nitrate		5.0 mg/L
Ortho-Phosphate		5.0 mg/L
Sulfate		100 mg/L

7.5.2. Prepare or purchase a secondary stock standard(s) using a standards source other than that used for the primary standards as described in Section 7.5. Dilute these stock standards to as indicated in the table below to prepare the mixture to be used for the LCS and CCV solution. Alternatively prepare this solution by mixing 0.50 mL commercial stock A, 0.50 mL commercial stock B and 4.0 mL of reagent water.

LCS & Continuing Calibration Verification Solution

Analyte	Final Conc. (V_f=5ml)
Fluoride	2.5 mg/L
Chloride	50. mg/L
Nitrite	2.5 mg/L
Bromide	10. mg/L
Nitrate	2.5 mg/L
Ortho Phosphate	2.5 mg/L
Sulfate	50. mg/L

7.5.3. Prepare or purchase a secondary stock standard(s) using a standards source other than that used for the primary standards as described in Section 7.5. Dilute these stock standards to prepare the mixture to be used for the Matrix Spike solution. Alternatively purchase these mixes (ready to use) from a commercial source. Add 100 uL of each IC Spike solution to 5 mL of sample when preparing the MS/MSD. Dilute as needed after spiking the sample.

Matrix Spike "True" Values

Analyte	Final Conc.
Fluoride	2.5 mg/L
Chloride	50. mg/L
Nitrite	2.5 mg/L
Bromide	10. mg/L
Nitrate	2.5 mg/L
Ortho Phosphate	2.5 mg/L
Sulfate	50. mg/L

NOTE: Stock standards, calibration standard #5 and LCS standard should be stored in the dark at 4° ± 2°C. Replace these standards when instrument response indicates target analyte degradation may have occurred or after the standard has expired (12 months

commercial mix or 6 months in house mix), which ever occurs first. Nitrite and ortho-phosphate are particularly light and oxygen sensitive.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to ensure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

8.2. Sample preservation and holding times for the anions that can be determined by this method for water samples are as follows:

QuanTIMs Method Code		Analyte	Preservation	Holding Time
EPA 300.0A	SW846 9056A	Fluoride	4° ± 2°C	28 days
C8	3C			
CX	3D	Chloride	4° ± 2°C	28 days
GO	3E	Nitrite	4° ± 2°C	48 hours
GM	3F	Bromide	4° ± 2°C	28 days
C9	3G	Nitrate	4° ± 2°C	48 hours
DO	3H	Ortho Phosphate	4° ± 2°C	48 hours
CY	3I	Sulfate	4° ± 2°C	28 days

Note: Soil leachates will follow the same preservation and holding times as the water samples; starting from the time of extraction.

9. QUALITY CONTROL

9.1. The STL QC Program document provides further details of the QC and corrective action guidelines presented in this SOP. Refer to this document if additional guidance is required.

9.2. Table I provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.3. Initial Demonstration of Capability

9.3.1. Prior to the analysis of any samples by Ion Chromatography, the following requirements must be meet:

9.3.1.1. Method Detection Limit (MDL): An MDL must be determined prior to analysis of any samples. The MDL is determined using seven replicates of reagent water spiked with the anions of interest that has been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis. The spike level must be greater than the calculated MDL but less than or equal to 10x the MDL. The result of the MDL determination must be below the STL reporting limit.

9.4. Batch definition: Preparation and QC batch definitions are provided in the STL QC Policy.

9.5. Method Blank (MB): One method blank must be processed with each preparation batch. The method blank consists of reagent grade water that has been taken through the entire preparation and analytical process. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest above the reporting limit.

9.6. Laboratory Control Sample (LCS): One LCS must be processed with each preparation batch and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. If the result is outside established control limits the system is out of control and corrective action must occur. Until in-house limits are established, a control limit of 90 - 110% recovery must be applied. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable. The LCS consists of reagent grade water containing a known amount of target analytes that has been injected into the ion chromatography system. The LCS is prepared from a separate stock standard, or neat material, of a different manufacturer than the stock, or neat material, used to prepare the calibration standard.

9.7. Matrix Spike/Matrix Spike Duplicate (MS/MSD): One MS/MSD pair must be processed for each QC batch. A matrix spike (MS) is a field sample to which a known concentration of target analyte has been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed

along with the sample and matrix spike. Some client specific DQO's may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Spiking levels will be the same as the LCS values.

- If the MS/MSD recovery or RPD falls outside the acceptance range, the recovery of the analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 90-110% recovery and 20% RPD must be applied to the MS/MSD.

- If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported as NC (i.e. not calculated).

- If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted.

- If the recovery of the LCS is outside the limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

- If a MS/MSD is not possible due to limited sample volume then a LCS duplicate must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike limits.

9.8. Continuing Calibration Verification (CCV/CCB): Continuing calibration is verified by analyzing the calibration standard after every ten (10) samples . The CCV must fall within +/- 10% of the true value for each target analyte. A CCB is analyzed immediately following the CCV to monitor low level accuracy and system cleanliness. The CCB result must be below the reporting limit for that analyte. If either the CCV or CCB fail to meet criteria, the analysis must be terminated, the problem corrected and repreparation and analysis of all samples following the last CCV and CCB which were in control.

10. CALIBRATION AND STANDARDIZATION

10.1. Establish ion chromatographic operating parameters equivalent to those indicated in table 2. Refer to Table 3 for typical standard run retention times. Other than the presence of the analytical column the instrument conditions are the same.

10.2. For each analyte of interest, prepare a **minimum** of 3 calibration standards and a blank by adding accurately measured volumes of one or more stock standards to a volumetric flask and dilution to volume with reagent water. If a sample analyte concentration exceeds the calibration range the sample may be diluted to fall within the range. If this is not possible then three new calibration concentrations must be chosen, two of which must bracket the concentration of the sample analyte of interest. Each attenuation range of the instrument used to analyze a sample must be calibrated individually.

10.3. Using an injection volume of 25 uL of each calibration standard, tabulate peak height or area responses against the concentration. The results are used to prepare a calibration curve for each analyte. During this procedure, retention times must be recorded. All analytes will be calibrated using a quadratic regression forced through the origin. Correlation coefficients (R^2) must be 0.995 or better.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Table 2 summarizes the recommended operating conditions for the ion chromatograph. Included in this table are estimated retention times that can be achieved by this method. Other columns, chromatographic conditions, or detectors may be used if the requirements of Sect. 9.2 are met.

11.4. Check system calibration daily as outlined in Table 1 and, if required, recalibrate as described in Sect 10.

11.5. Load and inject a fixed amount (25 uL) of settled & filtered sample. If the sample is cloudy then it should be filtered prior to loading into the autosampler polyvial. Flush injection loop thoroughly, using each new sample. Use the same size loop for standards and samples. Record the resulting peak size in area or peak height units. An automated constant volume injection system may also be used.

11.6. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of various concentration. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms since retention time is concentration dependent for most analytes..

11.7. If the response for the peak exceeds the working range of the system, dilute the sample with an appropriate amount of reagent water and reanalyze.

11.8. If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, fortify the sample with an appropriate amount of standard and reanalyze.

NOTE: Retention time is affected by concentration. Nitrate and sulfate exhibit the greatest amount of change, although all anions are affected to some degree. See Table 3. In some cases this peak migration may produce poor resolution or identification.

11.9. The following extraction should be used for solid materials: Add an amount of reagent water equal to ten times the weight of dry solid material taken as a sample. This slurry is mixed for one hour using a magnetic stirring device or tumbler. Filter the resulting slurry before injecting using a 0.45 um membrane type filter. This can be the type that attaches directly to the end of the syringe

11.10. Should more complete resolution be needed between peaks the eluent (7.3) can be diluted. This will spread out the run but will also cause the later eluting anions to be retained longer. The analyst must determine to what extent the eluent is diluted. This dilution should not be considered a deviation from the method.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Prepare a calibration curve for each analyte by plotting instrument response against standard concentration. Compute sample concentration by comparing sample response with the standard curve. Multiply answer by appropriate dilution factor.

12.2. Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

12.3. Report results in mg/L for aqueous samples and mg/Kg for 1 hour leachates and mg/L for 18 hour leachates of solid samples.

12.4. Report NO_2^- as N

NO_3^- as N

$HPO_4^=$ as P

13. METHOD PERFORMANCE

13.1. The reporting limits for the following analytes are based on a 25 uL injection volume:

Analyte	Water RL	Soil RL
Fluoride	1.0 mg/L	10 mg/kg
Chloride	1.0 mg/L	10 mg/kg
Nitrite	0.5 mg/L	5 mg/kg
Bromide	0.5 mg/L	5 mg/kg
Nitrate	0.05 mg/L	0.5 mg/kg
O-Phosphate	0.5 mg/L	5 mg/kg
Sulfate	1.0 mg/L	10 mg/kg

13.2. The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. The analyst must be given two blind performance samples to analyze or process for analysis. Upon successful completion of the performance evaluation (PE) samples, these analyses will be documented as initial qualification,. Requalification must be performed annually thereafter for this procedure. The group/team leader must document the training and PE performance and submit the results to the QA Manager for inclusion in the associate's training files.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure must be segregated, and disposed of according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. Method 300.0, "Determination of Inorganic Anions by Ion Chromatography", Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio, Revision 2.1, August 1993.

16.2. Method 9056A, "Determination of Inorganic Anions by Ion Chromatography", SW846, Test Methods for Evaluating Solid Waste, Third Edition, Draft Revision 1, September 1999.

16.3. STL North Canton Laboratory Quality Manual (LQM), current version.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Attachment #1, method Flow Chart

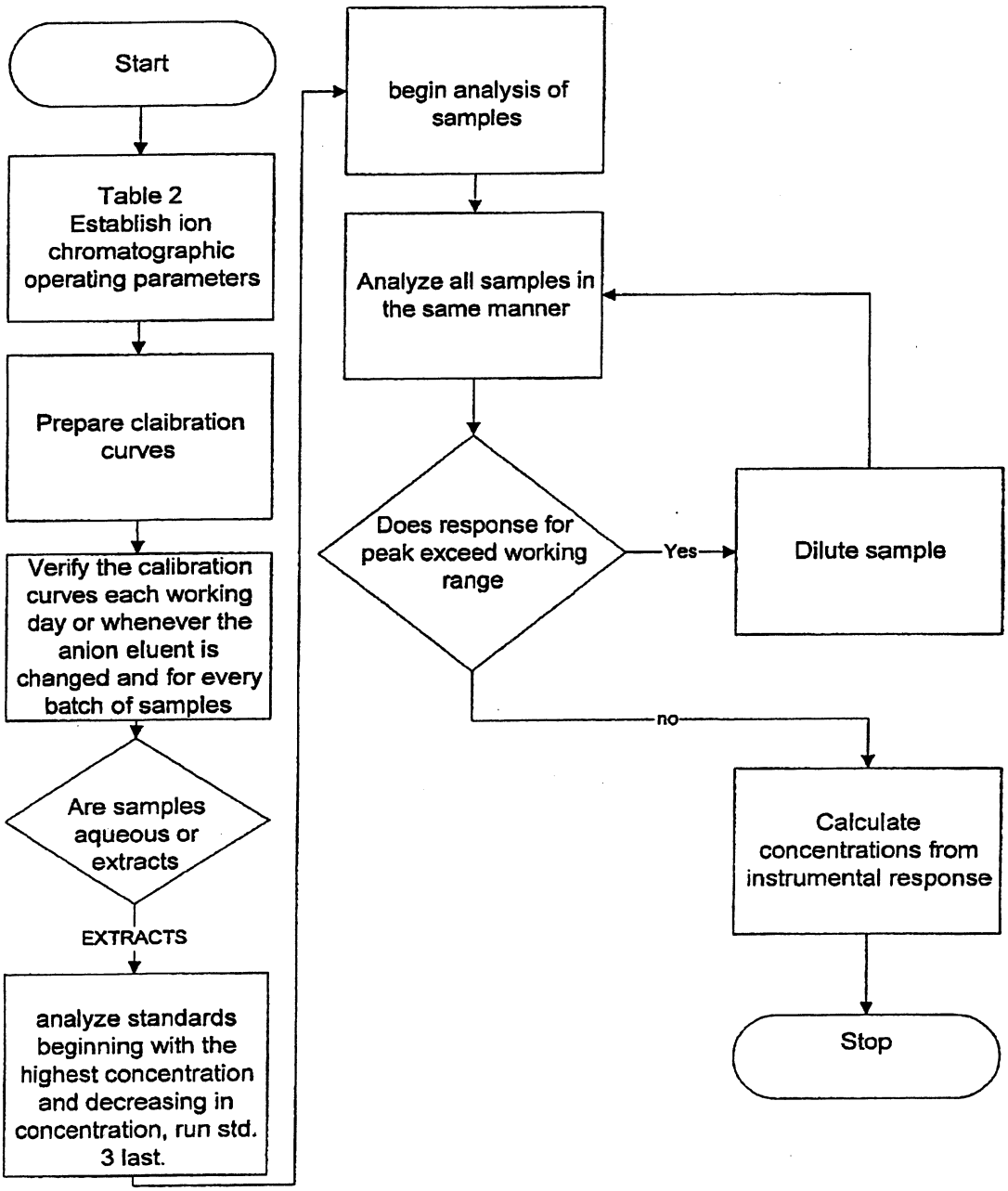
17.2. Table 1, Quality Control Samples

17.3. Table 2, Standard Instrument Operating Parameters

17.4. Table 3, Retention Time Matrix

17.5. Figure 1, Example Chromatogram

Determination Of Inorganic Anions By Ion Chromatography



ATTACHMENT #1

TABLE 1

QUALITY CONTROL SAMPLES

QC Samples	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration Verification (ICV)	At the start of each day following calibrating prior to sample analysis	+/- 10% of true value	Recalibrate and reanalyze
Initial Calibration Blank (ICB)	After Initial Calibration Verification and prior to sample analysis	< the Reporting Limit	Reprepare and reanalyze
Laboratory Control Sample (LCS)	1 per batch of 20 samples	Meets laboratory historical limits	Reanalyze all samples associated with unacceptable LCS
Matrix Spike Sample (MS/MSD)	1 MS/MSD pair per batch or 20 samples	Meets laboratory historical limits	Supervisor's technical judgment
Continuing Calibration Verification (CCV)	Between each group of 10 injections and at the end of the analytical sequence	+/- 10% of true value	Recalibrate and reanalyze all samples since the last acceptable CCV
Continuing Calibration Blank (CCB)	Between each group of 10 injections and at the end of the analytical sequence	< the Reporting Limit	Recalibrate and reanalyze all samples since the last acceptable CCB

TABLE 2

Standard Instrument Operating Parameters

Standard Conditions:

Eluent Pump Rate:	1.20 mL/min (DX-120 and DX-320)
Sample Loop:	25 uL
Eluent:	1.0mM sodium bicarbonate, 3.5mM sodium carbonate
Detector output	Baseline conductivity should be 15 - 20 uS prior to sample analysis.

TABLE 3

Standard Run Retention Time Matrix (minutes)*

Analyte	Concentration (mg/L) 0.05	0.2	0.5	1	2	2.5	5	10	20	40	50	100	200	RT window
F^-	2.75		2.75			2.75	2.75	2.75						
Cl^-				3.97				3.98			4.03	4.08	4.17	
NO_2^-	4.80		4.80			4.78	4.78	4.80						
Br^-		6.15			6.13			6.10	6.08	6.07				
NO_3^-	7.33		7.27			7.17	7.13	7.07						
o-PO_4^{2-}	9.53		9.53			9.52	9.50	9.48						
SO_4^{2-}				11.50				11.48			11.43	11.38	11.27	

* Analyte retention time is concentration dependent for most anions. Retention time increases with increasing concentration for chloride. Retention time decreases with increasing concentration for bromide, nitrate, ortho-phosphate and sulfate.

EXAMPLE ION CHROMATOGRAM

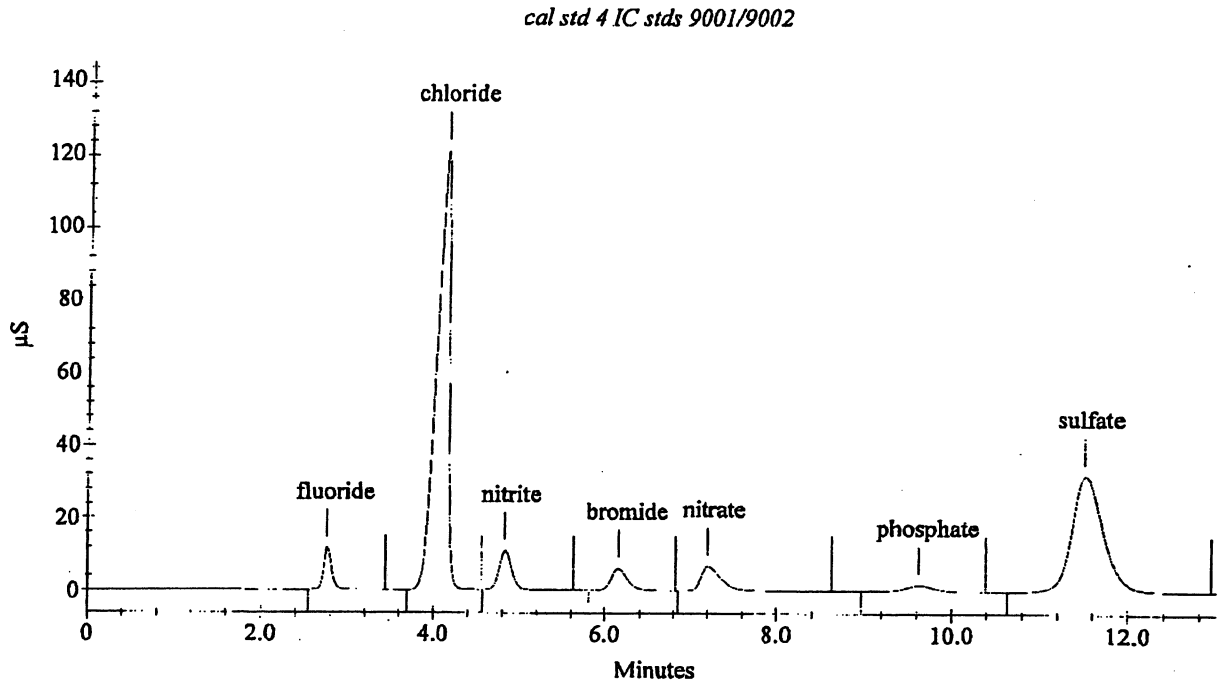


Figure #1

STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: CYANIDE AUTOMATED, PYRIDINE-BARBITURIC ACID METHOD

(SUPERSEDES: REVISION 6 DATED 06/28/99)

Prepared by: _____ 5/31/01
Date

Reviewed by: _____ 5/31/01
Technology Specialist Date

Approved by: _____ 5/31/01
Quality Assurance Manager Date

Approved by: _____ 5-31-01
Environmental Health and Safety Coordinator Date

Approved by: _____ 5-31-01
Laboratory Director Date

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Total, Amenable, and Free Inorganic Cyanide in solids, liquids, and waters. It is based on CLP ILM03.0, CLP ILM04.0, SW846 Method 9012A, 335.2, 335.4 and Standard Method 4500-CN-E. The working linear range is 0.005 - 0.1 mg/L for waters and 0.25 to 5 mg/kg for solids.

1.2. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

1.3. The associated method codes are CLP ILM03.0 (DV), CLP ILM04.0 (OU), SW846 Method 9012A (QP), ASTM D4282-83, EPA 335.1 and 335.2 (CG), EPA 335.4 (O2), and Standard Method 4500-CN-E (A4).

2. SUMMARY OF METHOD

2.1. The Cyanide, as HCN, is released by distilling/refluxing the sample with strong acid and is trapped in a sodium hydroxide solution.

2.2. The sodium hydroxide solution is analyzed colormetrically on an autoanalyzer using the pyridine-barbituric acid method.

3. DEFINITIONS

3.1. Refer to the glossary in the Laboratory Quality Manual (LQM), latest version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Sulfides interfere, but can be eliminated by treating the sample with cadmium carbonate prior to analysis.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. The following materials are known to be **corrosive: Sodium Hydroxide**

5.3.2. Cyanide is a highly toxic compound that must be handled with care. The analyst must be aware of the handling techniques.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents {as well as glassware cleaning procedures that involved solvents such as methylene chloride} should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.6. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Traacs 800 autoanalyzer

6.2. Probe 0.016"

6.3. Flowcell 10nm

6.4. Wavelength cell 570nm

6.5. Manifold-Multitest cartridge with cyanide tubing

6.6. 125 mL Erlenmeyer flasks

6.7. 100 mL, 250 mL, 1000 mL volumetric flasks

6.8. Volumetric pipettes: various

6.9. Top loading balance: capable of accurately weighing ± 0.01g.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Cadmium carbonate: powder

7.1.2. 1.25 N sodium hydroxide: Add 50 g of sodium hydroxide pellets (NaOH) to a 1 liter volumetric flask and dilute to volume with reagent water.

7.1.3. Phosphate buffer: Add 136 g of potassium phosphate - monobasic (KH_2PO_4) and 2.8 g of sodium phosphate – dibasic anhydrous (Na_2HPO_4) to 800 mL of reagent water in a 1 liter volumetric flask. Mix, bring to volume with reagent water. Add 3-5 drops of Brij-35 to 100 mL of Buffer prior to using.

7.1.4. Chloramine-T reagent: Add 1.0 g of chloramine-T to a 250 mL volumetric flask and dilute to volume with reagent water. Prepare fresh daily.

7.1.5. Pyridine reagent: Add 15.0 g of barbituric acid to a 1 liter volumetric flask. Add 75 mL of pyridine and 15 mL of concentrated hydrochloric acid (HCl) and mix. Bring to volume with reagent water and store at 4°C ± 2°C in an amber glass bottle.

7.1.6. 0.25 N sodium hydroxide: Add 200 mL of N NaOH to a 1 liter volumetric flask and dilute to volume with reagent water.

7.1.7. Brij-35

7.1.8. 30% Brij solution: Add 2 mL Brij-35 to 1000 mL reagent water.

7.1.9. Rhodanine indicator, purchased.

7.1.10. 0.0192 N silver nitrate, purchased

7.2. Standards

7.2.1. Primary Source Cyanide Stock Standard, 1000 mg/L: Add 2.51 g of potassium cyanide (KCN) and 2.0 g of potassium hydroxide (KOH) to a 1000 mL volumetric flask and dilute to volume with reagent water. Mix well and store in glass amber container. Stable for 1-3 months.

Note: This stock standard must be standardized prior to use. See Appendix I.

7.2.2. Secondary Source Cyanide Standard, 1000 mg/L: Follow 7.2.1 using an alternate source of Potassium Cyanide (KCN).

Note: This stock standard must be standardized prior to use. See Appendix I.

7.2.3. Calibration Standards (Water and Solid Matrices)

7.2.3.1. Pipet the appropriate amount of cyanide standard into 100 mL volumetric. Add 20 mL 1.25N NaOH to each calibration standard (except the 1.0 mg/L) and bring to volume with reagent water. Prepare weekly.

Concentration CN-	mL CN-	Final Volume
100 mg/L	10 mL of 1000 mg/L	100 mL
10 mg/L	10 mL of 100 mg/L	100 mL
1.0 mg/L	10 mL of 10 mg/L	100 mL
*0.1 mg/L	10 mL of 1 mg/L	100 mL
*0.05 mg/L	5 mL of 1 mg/L	100 mL
*0.025 mg/L	25 mL of 0.1 mg/L	100 mL
*0.01 mg/L	10 mL of 0.1 mg/L	100 mL

*0.005 mg/L	10 mL of 0.05 mg/L	100 mL

*Denotes calibration standards

NOTE: The 1 ppm CN standard is only good for the day it is prepared and is not preserved with NaOH.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Solid and liquid samples are not chemically preserved. Water samples are preserved with NaOH to a pH > 12. All samples are stored at 4°C ± 2°C in plastic or glass containers.

8.2. The holding time is fourteen days from sampling to analysis.

*NOTE: Holding time for CLP is twelve days from receipt to analysis.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.2. CLP

9.2.2.1. Each SDG (sample delivery group) must have one method blank (MB) processed per matrix and per twenty samples. If the SDG is distilled over multiple "racks", the method blanks must be distilled with the samples. If more

than one SDG is on one rack, process one method blank per SDG and per matrix. The solid method blank is called "PBS" (prep blank solid) and the water method blank is called "PBW" (prep blank water).

NOTE: A "rack" is one setup of cyanide samples and QC, and can encompass 1-3 of the midi-distillation units.

9.2.3. A reagent water blank consisting of 50 mL reagent water must be distilled and analyzed with each analytical batch of samples. See SOP NC-WC-0032 for distillation instructions.

9.2.4. Corrective Action for Blanks

9.2.4.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.4.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One LCS from an independent source must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. A midrange LCS consisting of a 0.04 mg/L (2.0 mL of 1.0 mg/L to 50 mL) must be distilled and analyzed with each analytical batch of samples. See SOP NC-WC-0032 for distillation instruction.

9.3.3. The acceptance limits for drinking water samples is 90 – 110%. This is also the same value for CLP.

9.3.4. CLP

9.3.4.1. A LCS (which doubles as an ICV) is processed with each "rack". Even if multiple SDG's are on the "rack", only one LCS (ICV) needs to be distilled.

9.3.4.2. For solids process a solid LCS per SDG. The value for this LCS changes as the solid lot number changes each time a new one is ordered. The value is noted on the product certification. If more than one SDG is processed on a "rack", process a solid LCS for each one. If a SDG has solid sample distilled on a different "rack", then additional solid LCS's will need to be distilled.

9.3.5. Corrective Action for LCS

9.3.5.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.5.2. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. A MS/MSD consisting of 50 mL or 1.0 g sample and 0.04 mg/L spike (2.0 mL of 1.0 mg/L to 50 mL) will be distilled and analyzed with every batch. See SOP NC-WC-0032 for distillation instructions. This is also the same value for CLP.

9.4.3. The acceptance limits for drinking water samples is 90 – 110%.

9.4.4. Corrective action for MS/MSDs

9.4.4.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

9.4.4.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported as DIL (diluted out).

9.4.4.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.4.5. CLP

9.4.5.1. Each SDG must have a sample duplicate and a matrix spike processed per matrix. Sometimes this requirement is waived by the client or PM but, if uncertain, make sure that these are done. Usually , the sample to be spiked is assigned, but not always. If a MS/MSD is created in receiving, either batch the MSD and "N/A" the result or have the MSD deleted. In either case, create a sample duplicate for the sample that is spiked when batching. If processin a SDG over more than one "rack", then there is no need to process additional matrix spikes and sample duplicates from "rack" (unless specifically requested). Typically, only one spiked/duped sample is required per SDG, even if the SDG is distilled over different days.

9.4.5.2. Matrix spike for CLP consists of 50 mL or 1.0 g of sample and 100 µg/L spike (5.0 mL of 1.0 mg/L standard).

9.5. QC Acceptance Criteria

9.5.1. Control limits are established by the laboratory as described in NC-QA-0018.

9.5.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are listed in the Laboratory Quality Manual (LQM) and the latest is version easily accessible via the LIMs (QC Browser program.).

9.6. Method Detection Limits (MDLs) and MDL Checks

9.6.1. MDLs and MDL Checks are established by the laboratory as described in SOP, NC-QA-0021.

9.6.2. MDLs are listed in the Laboratory Quality Manual (LQM) and the latest version is easily accessible via the LIMs (QC Browser program).

9.7. Nonconformance and Corrective Action

9.7.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. The instrument is calibrated using five cyanide standards and a blank at the time of analysis.

10.2. Initial Calibration

10.2.1. The instrument is calibrated at the beginning of each run and is verified at the beginning of the run by using a midrange ICV. The correlation coefficient of the original curve must be $\geq$ 0.995 or recalibration is required.

10.3. Continuing Calibration

10.3.1. The run is checked every ten samples and at the end of the run using a midrange CCV to verify continued linearity. It cannot vary from the original curve by more than $\pm$ 15% or recalibration is required. The previous 10 samples must be reanalyzed and bracketed by a CCV that passes criteria. The CCV is composed of the 0.025 ppm secondary standard.

10.3.2. System cleanliness is checked every ten samples and at the end of the run using a CCB. It cannot contain the analyte of interest above the reporting limit or recalibration is required. The previous 10 samples must be reanalyzed and bracketed by a CCB that passes criteria. The CCB is 0.25N NaOH.

NOTE: Base and gain values may change with new reagents, standards, tubing changes, or board cleaning. These values are for reference only.

10.4. High and Low Standard

10.4.1. The distillation technique is checked by distilling a high and low standard and comparing the values obtained to the standard curve. If the percent recovery is not within ± 10%, the analyst will submit a Nonconformance Memo for the analytical run.

10.4.2. If the percent recovery is outside ±15%, the standard and samples will be redistilled and analyzed.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. See cyanide distillation SOP: NC-WC-0032.

11.3.1.1. The sample is distilled/refluxed under acidic conditions for one hour. The released HCN is trapped in 50 mL of 0.25 N NaOH solution. EPA Method 335.4 says to reflux for one and a half hours.

NOTE: CLP samples are distilled for one and a half hours.

11.3.2. Sample Preparation Procedure

11.3.2.1. All Samples: Test each sample for the presence of sulfides using lead acetate paper. If sulfides are present, treat the sample with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution. Avoid a large excess of

cadmium carbonate and long contact time in order to minimize loss by complexation or occlusion of cyanide on the precipitated material

11.4. Sample Analysis

11.4.1. Summary

11.4.1.1. The sample distillates are analyzed on the autoanalyzer for cyanide using the automated pyridine-barbituric method.

11.4.2. Recommended Instrument Conditions

11.4.2.1. See Manufacturer's information for operation instructions.

11.4.2.2. Start Up Solutions (and Wash Solutions)

11.4.2.2.1. All lines in 0.25N NaOH solution:

11.4.2.2.2. Then all lines in 30% Brij solution.

11.4.2.3. Running Solutions

11.4.2.3.1. Two DI water lines (orange/green) in 30% Brij solution

11.4.2.3.2. Red/Red in pyridine reagent

11.4.2.3.3. Orange/red in chloramine-T reagent

11.4.2.3.4. White/white in phosphate buffer solution

11.4.2.4. Base and Gain

11.4.2.4.1. Performed on the 0.1 mg/L standard

11.4.2.4.2. Approximate values: Base = 75 Gain = 150

*These values are for reference only and may change with new reagent or standard preparations.

11.4.2.5. Trouble shooting for poor Base and Gain or baseline noise

11.4.2.5.1.Place reagent lines 1-3 back in Brig solution and run on high for five minutes. Place all 5 lines (1-5) and probe line in 1.25 N NaOH solution and run on high for five minutes. Place lines back in appropriate "start-up" solutions and run on high for five minutes.

Place lines back in reagents and perform another base and gain when the baseline has stabilized.

11.4.3. Sample Analysis Procedure

11.4.3.1.See Manufacturer's information for operating instructions.

11.4.3.2.A calibration curve is analyzed at the beginning of each run. The correlation coefficient must be > 0.995 to continue.

11.4.3.3.CCV's and CCB's are analyzed at the beginning, end, and between every 10 samples. CCV's must be prepared from a secondary source.

11.4.3.4.Sample distillates higher than the highest calibration standard (0.1 mg/L) must be diluted with 0.25 N NaOH and re-analyzed.

11.4.3.5.Any samples analyzed after a high sample must be re-analyzed if carryover is suspected.

11.4.3.6.CLP

11.4.3.6.1. If more than one SDG is distilled on one rack, they may be analyzed together. Each SDG should have its own method blank(s), its own LCS-S (if samples are solids), and its own sample duplicate and matrix spike. If the SDG has been split over multiple distillation "racks", then you may not have a duped/spiked sample for each batch. You should have method blanks and, if solids, LCS's for each batch. An ICV (LCS) is distilled with each "rack". If more than one SDG is distilled on a "rack", then the ICV (LCS) distilled with that rack must be analyzed with the samples. If the samples from a multiple SDG "rack" are analyzed together, then the ICV needs to be analyzed only once; if they are analyzed on different runs, then the ICV (LCS) needs to be analyzed with each run.

11.4.3.7.Tray Protocol (Run Order)

Normal Tray Protocol

1. Calibration
2. ICV (CCV)
3. ICB (CCB)
4. MD
5. LCS
6. UP to 8 more samples
7. CCV
8. CCB
9. Up to 10 samples
10. CCV
11. CCb

CLP Tray Protocol

12. Calibration
13. Distilled ICV (LCS)
14. ICB
15. CCV
16. CCB
17. PBW

18. PBS (If solids are in SDG)

19. LCS-S (If solids are in SDG)

20. Sample

21. Sample duplicate

22. Sample MS

23. Sample MS @ 2x dilution

24. Sample A-spiked*

25. Up to 2 more samples

26. CCV

27. CCb

28. Up to ten more samples

29. CCV

30. CCb

*A-spike = 2 mL of sample + 2mL of A-spike solution

A-spike solution = 0.8 mL .25N NaOH, +3.2 mL 0.05 ppm 1° standard

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.5.2. All standards are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4. Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. $$\textit{Total Cyanide, mg / L} = \frac{\textit{mg / L } CN^- \textit{ from printout x 50}}{\textit{mL of sample distilled}} \textit{ x D}$$

12.2. $$\textit{Total Cyanide, mg / kg} = \frac{\textit{mg / L } CN^- \textit{ from printout x 50}}{\textit{g of sample distilled.}} \textit{ x D}$$

12.3. $$\textit{Amenable Cyanide, mg / L} = \textit{Total } CN^- \textit{ (mg / L) - Chlorinated } CN^- \textit{ (mg / L)}$$

Where:

mg/L = *can also be mg/kg*

D = *Dilution Factor* = $\frac{\textit{Final Volume of Dilution}}{\textit{Volume of Sample Distillate Used}}$

NOTE: Free cyanide has the same calculations as Total cyanide

12.4. LCS Recovery:

$$\frac{\textit{Instrument Value}}{\textit{0.04 (true)}} \textit{ x 100} = \textit{\% Recovery}$$

12.5. CCV Recovery:

$$\frac{\textit{Instrument Value}}{\textit{0.025 (true)}} \textit{ x 100 = \% Recovery}$$

NOTE: CCV recovery must be between 85 - 115% for data to be acceptable. If CCV recovery is not within these limits, reanalysis is required 335.4 says 10%.

12.6. ***MS/MSD Recovery for Waters and solids:***

$$\frac{\textit{A - B}}{\textit{0.040 (true)}} \textit{ x 100 = \% Recovery}$$

Where:

A = ***Instrument value MS/MSD***

B = ***Sample instrument value***

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All distillates can be rinsed down the drain with large amounts of water unless they contain high levels of Cyanide. If they are highly concentrated, they must be disposed of in the liquid waste container.

15.2. Solvent waste must be disposed of in clearly labeled waste cans.

15.3. Acid waste must be collected in clearly labeled acid waste containers.

15.4. Solid materials (gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.5. Refer to the Laboratory Sample and Waste Disposal plan.

15.6. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1. References

16.1.1. SW846, Test Methods for Evaluating Solid Waste Method 9012 A, and its updates

16.1.2. EPA 600; Cyanide, Total and Cyanide, Amenable to Chlorination; Methods 335.2, March 1983

16.1.3. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition: Weak and Dissociable Cyanide; Method 4500-CN-E

16.1.4. USEPA CLP SOW ILM03.0 and ILM04.0, Section D - Cyanide Midi Distillation

16.1.5. Corporate Quality Management Plan (QMP), current version.

16.1.6. STL Laboratory Quality Manual (LQM), current version

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-0014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-0021

16.2.5. Navy/Army SOP, NC-QA-0016

16.2.6. NC-WC-0032, Cyanide Preparation Method

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Reporting limits

17.1.1. The reporting limit (RL) is 0.01 mg/L for waters (50 mL used) and 0.50 mg/kg for solids (1.0 g used). The reporting limit for solid samples performed only under the Michigan program is 200 µg/kg for solids (2.5 g used). The lowest level of the calibration curve can be used as the reporting limit upon request.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Troubleshooting guide for poor Base and Gain or baseline noise

17.2.1. Place reagent lines 1-3 back in Brig solution and run on high for five minutes. Place all 5 lines (1-5) and probe line in 1.25 N NaOH solution and run on high for five minutes. Place lines back in appropriate "start-up" solutions and run on high for five minutes.

Place lines back in reagents and perform another base and gain when the baseline has been stabilized.

17.3. Method Deviation (9012A335.2)

17.3.1. Method of Standard Addition is not performed for samples with matrix interference (sulfides).

Appendix I - Cyanide Standardization

1. Pipet 10.0 mL of the 1000 ppm stock cyanide standard into a 250 mL Erlenmeyer flask and add 90 mL of reagent water.
2. Add 0.5 mL (10 drops) of Rhodanine indicator.
3. Titrate with 0.0192 N silver nitrate (using a micro burette) until the color changes from yellow to pink/orange.

4. Titrate a blank (100 mL reagent water) following steps 2 and 3.

5. Calculation:

$$\textbf{Cyanide, mg / L} = \frac{\textbf{(A - B) (1000)}}{\textbf{mL Cyanide Solution (10)}}$$

Where:

A = mL titrant for standard

B = mL titrant for blank

6. If the cyanide concentration is not 1000 ppm, adjust concentration accordingly.

ATTACHMENT 3

LABORATORY REFERENCE DATA

STL Reference Data Summary

Structured Analysis Code: I-25-QK-01-01

Target Analyte List: NC: OLM03.1/4.2 Volatile List

Matrix: WATER
Extraction: PURGE AND TRAP - 25 mL purge (Waters)
Method: Volatile Organics, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 4121			Detection Limits				Check List 1630							Spike List 1631						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
11	Acetone	10	ug/L	0.51	ug/L	20010221		Y	20	ug/L	22	200	95		Y	20	ug/L	45	128	30
196	Benzene	1	ug/L	0.16	ug/L	20010215	C	Y	20	ug/L	80	116	20	C	Y	20	ug/L	78	118	20
323	Bromodichloromethane	1	ug/L	0.17	ug/L	20010215		Y	20	ug/L	87	130	30		Y	20	ug/L	80	146	30
340	Bromoform	1	ug/L	0.18	ug/L	20010215		Y	20	ug/L	76	150	30		Y	20	ug/L	58	176	30
343	Bromomethane	1	ug/L	0.2	ug/L	20010223		Y	20	ug/L	64	129	30		Y	20	ug/L	55	145	30
372	2-Butanone	10	ug/L	0.41	ug/L	20010215		Y	20	ug/L	28	237	65		Y	20	ug/L	71	123	30
459	Carbon disulfide	1	ug/L	0.2	ug/L	20010215		Y	20	ug/L	73	139	30		Y	20	ug/L	69	138	41
463	Carbon tetrachloride	1	ug/L	0.12	ug/L	20010221		Y	20	ug/L	75	149	30		Y	20	ug/L	63	176	30
521	Chlorobenzene	1	ug/L	0.14	ug/L	20010221	C	Y	20	ug/L	76	117	20	C	Y	20	ug/L	76	117	20
535	Dibromochloromethane	1	ug/L	0.26	ug/L	20010215		Y	20	ug/L	81	138	30		Y	20	ug/L	71	158	30
550	Chloroethane	1	ug/L	0.26	ug/L	20010215		Y	20	ug/L	66	126	30		Y	20	ug/L	59	142	30
569	Chloroform	1	ug/L	0.14	ug/L	20010221		Y	20	ug/L	84	128	30		Y	20	ug/L	83	141	30
574	Chloromethane	1	ug/L	0.13	ug/L	20010215		Y	20	ug/L	48	123	30		Y	20	ug/L	40	137	39
669	Cyclohexane	1	ug/L	0.13	ug/L	20010221														
539	1,2-Dibromo-3-chloropropane	2	ug/L	0.62	ug/L	20010112														
870	1,2-Dibromoethane	1	ug/L	0.22	ug/L	20010215														
904	1,2-Dichlorobenzene	1	ug/L	0.11	ug/L	20010221														
907	1,3-Dichlorobenzene	1	ug/L	0.15	ug/L	20010215														
910	1,4-Dichlorobenzene	1	ug/L	0.17	ug/L	20010215														
924	Dichlorodifluoromethane	1	ug/L	0.26	ug/L	20010215														
933	1,1-Dichloroethane	1	ug/L	0.16	ug/L	20010215		Y	20	ug/L	86	123	30		Y	20	ug/L	88	127	30
936	1,2-Dichloroethane	1	ug/L	0.21	ug/L	20010215		Y	20	ug/L	79	136	30		Y	20	ug/L	71	160	30
948	cis-1,2-Dichloroethene	0.5	ug/L	0.22	ug/L	20010215		Y	20	ug/L	85	113	30		Y	20	ug/L	87	114	30
950	trans-1,2-Dichloroethene	0.5	ug/L	0.15	ug/L	20010215		Y	20	ug/L	79	120	30		Y	20	ug/L	85	116	30
943	1,1-Dichloroethene	1	ug/L	0.24	ug/L	20010215	C	Y	20	ug/L	63	130	20	C	Y	20	ug/L	62	130	20
986	1,2-Dichloropropane	1	ug/L	0.17	ug/L	20010215		Y	20	ug/L	82	115	30		Y	20	ug/L	87	114	30
998	cis-1,3-Dichloropropene	1	ug/L	0.12	ug/L	20010215		Y	20	ug/L	84	130	30		Y	20	ug/L	82	130	30
1000	trans-1,3-Dichloropropene	1	ug/L	0.27	ug/L	20010215		Y	20	ug/L	84	130	30		Y	20	ug/L	73	147	30
1332	Ethylbenzene	1	ug/L	0.12	ug/L	20010221		Y	20	ug/L	86	116	30		Y	20	ug/L	86	132	30
1515	2-Hexanone	10	ug/L	0.46	ug/L	20010215		Y	20	ug/L	35	200	52		Y	20	ug/L	81	128	30
1578	Isopropylbenzene	1	ug/L	0.12	ug/L	20010215														
1774	Methyl acetate	10	ug/L	0.41	ug/L	20010215														
1799	Methylcyclohexane	1	ug/L	0.15	ug/L	20010221														
1811	Methylene chloride	1	ug/L	0.34	ug/L	20010215		Y	20	ug/L	78	118	30		Y	20	ug/L	82	115	30
1845	4-Methyl-2-pentanone	10	ug/L	0.23	ug/L	20010215		Y	20	ug/L	78	141	32		Y	20	ug/L	82	135	30
2772	Methyl tert-butyl ether	5	ug/L	0.13	ug/L	20010215														

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Structured Analysis Code: I-25-QK-01-01

Target Analyte List: NC: OLM03.1/4.2 Volatile List

Matrix: WATER
Extraction: PURGE AND TRAP - 25 mL purge (Waters)
Method: Volatile Organics, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 4121			Detection Limits			Check List 1630							Spike List 1631						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2355	Styrene	1	ug/L	0.15	ug/L	20010215		Y	20	ug/L	85	117	30		Y	20	ug/L	83	120	30
2439	1,1,2,2-Tetrachloroethane	1	ug/L	0.25	ug/L	20010215		Y	20	ug/L	85	118	30		Y	20	ug/L	88	116	30
2445	Tetrachloroethene	1	ug/L	0.23	ug/L	20010215		Y	20	ug/L	88	113	30		Y	20	ug/L	85	121	30
2489	Toluene	1	ug/L	0.18	ug/L	20010215	C	Y	20	ug/L	74	119	20	C	Y	20	ug/L	70	119	20
2515	1,2,4-Trichlorobenzene	1	ug/L	0.17	ug/L	20010215														
2518	1,1,1-Trichloroethane	1	ug/L	0.15	ug/L	20010221		Y	20	ug/L	78	140	30		Y	20	ug/L	71	162	30
2522	1,1,2-Trichloroethane	1	ug/L	0.3	ug/L	20010215		Y	20	ug/L	83	122	30		Y	20	ug/L	86	129	30
2525	Trichloroethene	1	ug/L	0.14	ug/L	20010215	C	Y	20	ug/L	75	122	20	C	Y	20	ug/L	62	130	20
1428	Trichlorofluoromethane	1	ug/L	0.28	ug/L	20010215														
2566	1,1,2-Trichloro-1,2,2-trifluoroethane	1	ug/L	0.32	ug/L	20010215														
2613	Vinyl chloride	1	ug/L	0.15	ug/L	20010215		Y	20	ug/L	61	120	30		Y	20	ug/L	88	126	30
2627	Xylenes (total)	1	ug/L	0.33	ug/L	20010221		Y	60	ug/L	87	116	30		Y	60	ug/L	89	121	30
337	4-Bromofluorobenzene						X	Y	10	ug/L	74	116	0	X	Y	20	ug/L	74	116	0
2735	1,2-Dichloroethane-d4						X	Y	10	ug/L	61	128	0	X	Y	10	ug/L	61	128	0
2740	Toluene-d8						X	Y	10	ug/L	76	110	0	X	Y	10	ug/L	76	110	0
2863	Dibromofluoromethane						X	Y	10	ug/L	73	122	0	X	Y	10	ug/L	73	122	0

STL Reference Data Summary

Structured Analysis Code: I-51-QL-01-01

Target Analyte List: NC: OLM03.1/4.2 Semivolatile List

Matrix: WATER
Extraction: LIQ/LIQ, CONT (Acids,Phenols) - Acid
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 4122			Detection Limits				Check List 1114							Spike List 1115						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
1	Acenaphthene	10	ug/L	0.8	ug/L	20010424	C	Y	50	ug/L	39	118	35	C	Y	50	ug/L	26	118	35
5	Acenaphthylene	10	ug/L	0.71	ug/L	20010424		Y	50	ug/L	51	110	30		Y	50	ug/L	49	111	51
24	Acetophenone	10	ug/L	2.6	ug/L	20010329														
122	Anthracene	10	ug/L	0.68	ug/L	20010424		Y	50	ug/L	54	116	33		Y	50	ug/L	49	118	51
158	Atrazine	10	ug/L	2.5	ug/L	20010329														
3398	Benzaldehyde	10	ug/L	2.8	ug/L	20010329														
202	Benzo(a)anthracene	10	ug/L	0.7	ug/L	20010424		Y	50	ug/L	49	112	34		Y	50	ug/L	48	115	51
205	Benzo(b)fluoranthene	10	ug/L	0.94	ug/L	20010424		Y	50	ug/L	51	115	38		Y	50	ug/L	44	123	54
208	Benzo(k)fluoranthene	10	ug/L	0.84	ug/L	20010424		Y	50	ug/L	52	116	34		Y	50	ug/L	46	123	53
210	Benzo(ghi)perylene	10	ug/L	0.85	ug/L	20010424		Y	50	ug/L	47	118	35		Y	50	ug/L	44	122	55
211	Benzo(a)pyrene	10	ug/L	0.73	ug/L	20010424		Y	50	ug/L	50	116	34		Y	50	ug/L	44	122	51
3474	1,1'-Biphenyl	10	ug/L	2.6	ug/L	20010329														
289	bis(2-Chloroethoxy)methane	10	ug/L	0.87	ug/L	20010424		Y	50	ug/L	54	112	32		Y	50	ug/L	57	114	49
293	bis(2-Chloroethyl) ether	10	ug/L	0.97	ug/L	20010424		Y	50	ug/L	59	114	30		Y	50	ug/L	57	120	51
302	bis(2-Ethylhexyl) phthalate	10	ug/L	2.7	ug/L	20010222		Y	50	ug/L	49	124	39		Y	50	ug/L	43	128	84
348	4-Bromophenyl phenyl ether	10	ug/L	0.88	ug/L	20010424		Y	50	ug/L	56	113	33		Y	50	ug/L	51	119	51
403	Butyl benzyl phthalate	10	ug/L	0.88	ug/L	20010424		Y	50	ug/L	46	121	40		Y	50	ug/L	41	127	84
5101	Caprolactam	10	ug/L	2.5	ug/L	20010329														
2751	Carbazole	10	ug/L	0.82	ug/L	20010424		Y	50	ug/L	50	118	34		Y	50	ug/L	48	119	53
518	4-Chloroaniline	10	ug/L	0.61	ug/L	20010424		Y	50	ug/L	21	110	41		Y	50	ug/L	10	110	99
578	4-Chloro-3-methylphenol	10	ug/L	0.99	ug/L	20010205	C	Y	50	ug/L	29	124	55	C	Y	50	ug/L	21	124	55
589	2-Chloronaphthalene	10	ug/L	0.85	ug/L	20010424		Y	50	ug/L	50	116	30		Y	50	ug/L	51	119	51
600	2-Chlorophenol	10	ug/L	1	ug/L	20010220	C	Y	50	ug/L	19	124	43	C	Y	50	ug/L	19	124	43
602	4-Chlorophenyl phenyl ether	10	ug/L	0.79	ug/L	20010424		Y	50	ug/L	56	112	31		Y	50	ug/L	51	118	51
633	Chrysene	10	ug/L	0.75	ug/L	20010424		Y	50	ug/L	49	118	34		Y	50	ug/L	49	118	52
860	Dibenz(a,h)anthracene	10	ug/L	0.79	ug/L	20010205		Y	50	ug/L	48	124	36		Y	50	ug/L	45	127	57
863	Dibenzofuran	10	ug/L	1.1	ug/L	20010215		Y	50	ug/L	53	112	30		Y	50	ug/L	51	117	51
891	Di-n-butyl phthalate	10	ug/L	0.67	ug/L	20010424		Y	50	ug/L	50	116	36		Y	50	ug/L	41	121	53
918	3,3'-Dichlorobenzidine	50	ug/L	0.93	ug/L	20010205		Y	50	ug/L	10	110	75		Y	50	ug/L	10	110	99
971	2,4-Dichlorophenol	10	ug/L	0.85	ug/L	20010424		Y	50	ug/L	51	118	43		Y	50	ug/L	52	121	88
1082	Diethyl phthalate	10	ug/L	0.65	ug/L	20010424		Y	50	ug/L	14	135	47		Y	50	ug/L	10	148	81
1145	2,4-Dimethylphenol	10	ug/L	0.85	ug/L	20010220		Y	50	ug/L	10	123	44		Y	50	ug/L	10	125	62
1149	Dimethyl phthalate	10	ug/L	0.76	ug/L	20010424		Y	50	ug/L	10	150	51		Y	50	ug/L	10	159	99
1167	4,6-Dinitro-2-methylphenol	50	ug/L	1.3	ug/L	20010329		Y	50	ug/L	22	143	79		Y	50	ug/L	20	158	99
1187	2,4-Dinitrophenol	50	ug/L	3	ug/L	20010215		Y	50	ug/L	10	146	99		Y	50	ug/L	10	168	99
1191	2,4-Dinitrotoluene	10	ug/L	0.79	ug/L	20010424	C	Y	50	ug/L	47	131	32	C	Y	50	ug/L	31	131	32

Structured Analysis Code: I-51-QL-01-01

Target Analyte List: NC: OLM03.1/4.2 Semivolatile List

Matrix: WATER
Extraction: LIQ/LIQ, CONT (Acids,Phenols) - Acid
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 4122			Detection Limits				Check List 1114							Spike List 1115						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
1193	2,6-Dinitrotoluene	10	ug/L	0.57	ug/L	20010424		Y	50	ug/L	59	125	32		Y	50	ug/L	58	127	82
1162	Di-n-octyl phthalate	10	ug/L	1.1	ug/L	20010424		Y	50	ug/L	49	130	42		Y	50	ug/L	39	144	89
1414	Fluoranthene	10	ug/L	0.81	ug/L	20010424		Y	50	ug/L	54	118	33		Y	50	ug/L	49	122	53
1417	Fluorene	10	ug/L	1	ug/L	20010215		Y	50	ug/L	53	114	31		Y	50	ug/L	51	119	51
1482	Hexachlorobenzene	10	ug/L	0.74	ug/L	20010205		Y	50	ug/L	54	114	34		Y	50	ug/L	48	123	51
1489	Hexachlorobutadiene	10	ug/L	0.73	ug/L	20010220		Y	50	ug/L	36	113	47		Y	50	ug/L	35	118	56
1492	Hexachlorocyclopentadiene	50	ug/L	1.1	ug/L	20010327		Y	50	ug/L	10	114	51		Y	50	ug/L	10	113	97
1497	Hexachloroethane	10	ug/L	0.74	ug/L	20010424		Y	50	ug/L	29	115	49		Y	50	ug/L	31	118	92
1535	Indeno(1,2,3-cd)pyrene	10	ug/L	0.63	ug/L	20010219		Y	50	ug/L	45	120	43		Y	50	ug/L	39	126	59
1566	Isophorone	10	ug/L	0.63	ug/L	20010205		Y	50	ug/L	54	111	33		Y	50	ug/L	56	112	50
1829	2-Methylnaphthalene	10	ug/L	0.64	ug/L	20010424		Y	50	ug/L	52	110	32		Y	50	ug/L	45	119	51
1851	2-Methylphenol	10	ug/L	1.1	ug/L	20010219		Y	50	ug/L	50	110	34		Y	50	ug/L	42	113	73
1857	4-Methylphenol	10	ug/L	1.3	ug/L	20010219		Y	50	ug/L	49	110	35		Y	50	ug/L	29	122	55
1932	Naphthalene	10	ug/L	0.75	ug/L	20010424		Y	50	ug/L	49	112	35		Y	50	ug/L	50	176	52
1960	2-Nitroaniline	50	ug/L	0.7	ug/L	20010205		Y	50	ug/L	53	122	34		Y	50	ug/L	48	125	83
1964	3-Nitroaniline	50	ug/L	0.71	ug/L	20010205		Y	50	ug/L	10	140	39		Y	50	ug/L	10	140	99
1968	4-Nitroaniline	50	ug/L	1.3	ug/L	20010220		Y	50	ug/L	32	116	49		Y	50	ug/L	20	122	99
1972	Nitrobenzene	10	ug/L	0.81	ug/L	20010424		Y	50	ug/L	56	119	32		Y	50	ug/L	56	125	81
1998	2-Nitrophenol	10	ug/L	2.1	ug/L	20010219		Y	50	ug/L	46	129	49		Y	50	ug/L	51	131	77
2001	4-Nitrophenol	50	ug/L	1.9	ug/L	20010329	C	Y	50	ug/L	19	144	34	C	Y	50	ug/L	10	145	34
2028	N-Nitrosodiphenylamine	10	ug/L	1.4	ug/L	20010215		Y	50	ug/L	49	117	36		Y	50	ug/L	49	117	51
2024	N-Nitrosodi-n-propylamine	10	ug/L	0.67	ug/L	20010424	C	Y	50	ug/L	30	115	36	C	Y	50	ug/L	18	115	36
3597	2,2'-oxybis(1-Chloropropane)	10	ug/L	0.78	ug/L	20010424														
2118	Pentachlorophenol	10	ug/L	2.4	ug/L	20010329	C	Y	50	ug/L	10	140	56	C	Y	50	ug/L	10	140	56
2154	Phenanthrene	10	ug/L	0.82	ug/L	20010424		Y	50	ug/L	53	112	32		Y	50	ug/L	52	117	51
2155	Phenol	10	ug/L	2.6	ug/L	20010219	C	Y	50	ug/L	10	131	43	C	Y	50	ug/L	10	131	43
2252	Pyrene	10	ug/L	1.1	ug/L	20010424	C	Y	50	ug/L	46	130	31	C	Y	50	ug/L	27	138	31
2555	2,4,5-Trichlorophenol	10	ug/L	1.2	ug/L	20010219		Y	50	ug/L	46	122	48		Y	50	ug/L	45	125	74
2559	2,4,6-Trichlorophenol	10	ug/L	1.7	ug/L	20010219		Y	50	ug/L	46	118	48		Y	50	ug/L	46	122	98
1425	2-Fluorobiphenyl						X	Y	50	ug/L	30	110	0	X	Y	50	ug/L	30	110	0
1426	2-Fluorophenol						X	Y	75	ug/L	13	110	0	X	Y	75	ug/L	13	110	0
2512	2,4,6-Tribromophenol						X	Y	75	ug/L	21	122	0	X	Y	75	ug/L	21	122	0
2736	Nitrobenzene-d5						X	Y	50	ug/L	32	112	0	X	Y	50	ug/L	32	112	0
2737	Phenol-d5						X	Y	75	ug/L	10	113	0	X	Y	75	ug/L	10	113	0
2738	Terphenyl-d14						X	Y	50	ug/L	10	144	0	X	Y	50	ug/L	10	144	0

STL Reference Data Summary

Structured Analysis Code: I-61-QH-72-01

Target Analyte List: Q: Aroclors Only Standard List

Matrix: WATER
Extraction: LIQ/LIQ, CONT w/ACID STRIP (PCB) - Nominal
Method: PCBs (8082)
QC Program: MICHIGAN
Location: STL North Canton

Target List 2207		Detection Limits				Check List 928							Spike List 929						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2082	Aroclor 1016	0.2	ug/L	0.054	ug/L	20000229	C	Y	10	ug/L	61	118	20	C	Y	10	ug/L	56	119	20
2085	Aroclor 1221	0.2	ug/L	0.11	ug/L	20000229														
2088	Aroclor 1232	0.4	ug/L	0.056	ug/L	20000229														
2091	Aroclor 1242	0.2	ug/L	0.075	ug/L	20000229														
2094	Aroclor 1248	0.2	ug/L	0.061	ug/L	20000229														
2097	Aroclor 1254	0.2	ug/L	0.082	ug/L	20000229														
2100	Aroclor 1260	0.2	ug/L	0.044	ug/L	20000229	C	Y	10	ug/L	61	124	27	C	Y	10	ug/L	31	138	27
2732	Decachlorobiphenyl						X	Y	0.2	ug/L	24	128	0	X	Y	0.2	ug/L	24	128	0
2739	Tetrachloro-m-xylene						X	Y	0.2	ug/L	45	120	0	X	Y	0.2	ug/L	45	120	0

STL Reference Data Summary

Structured Analysis Code: I-04-QO-01-01

Target Analyte List: Q: CLP ICP Metals TAL Standard List

Matrix: WATER
Extraction: METALS, TOTAL RECOVERABLE
Method: Inductively Coupled Plasma (6010B)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 2600		Detection Limits				Check List 906							Spike List 907						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
88	Aluminum	0.2	mg/L	0.028	mg/L	20000224		Y	2000	ug/L	80	120	20		Y	2000	ug/L	75	125	20
128	Antimony	0.06	mg/L	0.0022	mg/L	20000412		Y	500	ug/L	80	120	20		Y	500	ug/L	75	125	20
194	Barium	0.20	mg/L	0.003	mg/L	20000412		Y	2000	ug/L	80	120	20		Y	2000	ug/L	75	125	20
222	Beryllium	0.005	mg/L	0.00054	mg/L	20000224		Y	50	ug/L	80	120	20		Y	50	ug/L	75	125	20
411	Cadmium	0.005	mg/L	0.00028	mg/L	20000412		Y	50	ug/L	80	120	20		Y	50	ug/L	75	125	20
413	Calcium	5.0	mg/L	0.250	mg/L	20000224		Y	50000	ug/L	80	120	20		Y	50000	ug/L	75	125	20
2952	Chromium	0.01	mg/L	0.0014	mg/L	20000412		Y	200	ug/L	80	120	20		Y	200	ug/L	75	125	20
637	Cobalt	0.05	mg/L	0.0013	mg/L	20000224		Y	500	ug/L	80	120	20		Y	500	ug/L	75	125	20
643	Copper	0.025	mg/L	0.0042	mg/L	20000224		Y	250	ug/L	80	120	20		Y	250	ug/L	75	125	20
1539	Iron	0.1	mg/L	0.088	mg/L	20000412		Y	1000	ug/L	77	127	20		Y	1000	ug/L	75	125	20
1618	Magnesium	5.0	mg/L	0.030	mg/L	20000224		Y	50000	ug/L	80	120	20		Y	50000	ug/L	75	125	20
1659	Manganese	0.015	mg/L	0.0009	mg/L	20000412		Y	500	ug/L	80	120	20		Y	500	ug/L	75	125	20
1956	Nickel	0.04	mg/L	0.0022	mg/L	20000412		Y	500	ug/L	80	120	20		Y	500	ug/L	75	125	20
2214	Potassium	5	mg/L	0.041	mg/L	20000224		Y	50000	ug/L	80	120	20		Y	50000	ug/L	75	125	20
2285	Silver	0.01	mg/L	0.0015	mg/L	20000224		Y	50	ug/L	80	120	20		Y	50	ug/L	75	125	20
2315	Sodium	5	mg/L	0.630	mg/L	20000224		Y	50000	ug/L	80	120	20		Y	50000	ug/L	75	125	20
2607	Vanadium	0.05	mg/L	0.00082	mg/L	20000224		Y	500	ug/L	80	120	20		Y	500	ug/L	75	125	20
2649	Zinc	0.02	mg/L	0.012	mg/L	20000224		Y	500	ug/L	80	120	20		Y	500	ug/L	75	125	20

STL Reference Data Summary

Structured Analysis Code: I-04-QM-01-01

Target Analyte List: Q: CLP Trace ICP Metals TAL Standard List

Matrix: WATER
Extraction: METALS, TOTAL RECOVERABLE
Method: Inductively Coupled Plasma (6010B Trace)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 2610		Detection Limits					Check List 906							Spike List 907						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
140	Arsenic	0.01	mg/L	0.0039	mg/L	20010218		Y	2000	ug/L	80	120	20		Y	2000	ug/L	75	125	20
1605	Lead	0.003	mg/L	0.0025	mg/L	20000412		Y	500	ug/L	80	120	20		Y	500	ug/L	75	125	20
2281	Selenium	0.005	mg/L	0.0045	mg/L	20000224		Y	2000	ug/L	80	120	20		Y	2000	ug/L	75	125	20
2477	Thallium	0.010	mg/L	0.005	mg/L	20000224		Y	2000	ug/L	80	120	20		Y	2000	ug/L	75	125	20

STL Reference Data Summary

Structured Analysis Code: I-19-O8-01-01
Target Analyte List: All Analytes

Matrix: WATER
Extraction: METALS, TOTAL (Method exclusive) - Waters
Method: Mercury (7470A, Cold Vapor) - Liquid
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits					Check List 1021							Spike List 1026						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
1701	Mercury	0.0002	mg/L	0.000069	mg/L	20001031		Y	5	ug/L	70	118	20		Y	1	ug/L	53	135	20

STL Reference Data Summary

Structured Analysis Code: I-06-QP-01-01
Target Analyte List: All Analytes

Matrix: WATER
Extraction: DISTILLATION, MICRO/MIDI - Acid
Method: Cyanide, Total (9012A, Automated)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits					Check List 1180							Spike List 1181						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
667	Total Cyanide	0.01	mg/L	0.0017	mg/L	20001206		Y			65	114	20		Y	0.04	mg/L	40	130	99

STL Reference Data Summary

Structured Analysis Code: I-06-N4-01-01
Target Analyte List: All Analytes

Matrix: WATER
Extraction: DISTILLATION, MICRO/MIDI - Acid
Method: Cyanide, Amenable (9012)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List			Detection Limits				Check List 1180							Spike List 1181						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
664	Cyanide Amenable to Chlorination	0.01	mg/L	0.01	mg/L	20010927		Y			65	114	20		Y	0.04	mg/L	40	130	99

STL Reference Data Summary

Structured Analysis Code: I-88-CX-01-01
Target Analyte List: All Analytes

Matrix: WATER
Extraction: NO SAMPLE PREPARATION PERFORMED / DIRECT INJE
Method: Chloride (300.0, Ion Chromatography)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits					Check List 1045							Spike List 1046						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
512	Chloride	1	mg/L	0.17	mg/L	20001201	C	Y			90	110	20	C	Y	50	mg/L	56	137	20

STL Reference Data Summary

Structured Analysis Code: A-I5-QK-01-01

Target Analyte List: NC: OLM03.1/4.2 Volatile List

Matrix: SOLID
Extraction: Indiana Modified Method 5035 (IN 5035-M)
Method: Volatile Organics, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: STL North Canton

	Target List 4121		Detection Limits				Check List 924							Spike List 925						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
11	Acetone	20	ug/kg	2.1	ug/kg	20010222		Y	20	ug/kg	58	130	30		Y	50	ug/kg	10	200	66
196	Benzene	5	ug/kg	0.65	ug/kg	20010122	C	Y	20	ug/kg	75	129	20	C	Y	50	ug/kg	55	138	20
323	Bromodichloromethane	5	ug/kg	1.2	ug/kg	20010308		Y	20	ug/kg	72	125	30		Y	50	ug/kg	47	131	51
340	Bromoform	5	ug/kg	1.6	ug/kg	20010308		Y	20	ug/kg	43	149	30		Y	50	ug/kg	26	141	64
343	Bromomethane	5	ug/kg	1.8	ug/kg	20010308		Y	20	ug/kg	24	152	30		Y	50	ug/kg	15	152	72
372	2-Butanone	20	ug/kg	2.7	ug/kg	20010308		Y	20	ug/kg	27	200	46		Y	50	ug/kg	21	195	60
459	Carbon disulfide	5	ug/kg	0.78	ug/kg	20010222		Y	20	ug/kg	50	137	30		Y	50	ug/kg	27	149	73
463	Carbon tetrachloride	5	ug/kg	1	ug/kg	20010125		Y	20	ug/kg	57	137	30		Y	50	ug/kg	32	143	68
521	Chlorobenzene	5	ug/kg	0.52	ug/kg	20010122	C	Y	20	ug/kg	75	127	22	C	Y	50	ug/kg	49	139	22
535	Dibromochloromethane	5	ug/kg	1.1	ug/kg	20010308		Y	20	ug/kg	49	135	30		Y	50	ug/kg	44	135	61
550	Chloroethane	5	ug/kg	2.2	ug/kg	20010308		Y	20	ug/kg	31	144	30		Y	50	ug/kg	32	140	66
569	Chloroform	5	ug/kg	0.82	ug/kg	20010122		Y	40	ug/kg	73	115	30		Y	50	ug/kg	59	128	46
574	Chloromethane	5	ug/kg	1.3	ug/kg	20010122		Y	20	ug/kg	15	136	30		Y	50	ug/kg	28	130	81
669	Cyclohexane	10	ug/kg	1	ug/kg	20010125														
539	1,2-Dibromo-3-chloropropane	10	ug/kg	1.6	ug/kg	20010222														
870	1,2-Dibromoethane	5	ug/kg	1.2	ug/kg	20010308														
904	1,2-Dichlorobenzene	5	ug/kg	0.71	ug/kg	20010122														
907	1,3-Dichlorobenzene	5	ug/kg	0.8	ug/kg	20010122														
910	1,4-Dichlorobenzene	5	ug/kg	0.67	ug/kg	20010122														
924	Dichlorodifluoromethane	5	ug/kg	1.1	ug/kg	20010222														
933	1,1-Dichloroethane	5	ug/kg	0.58	ug/kg	20010122		Y	20	ug/kg	77	119	30		Y	50	ug/kg	56	130	54
936	1,2-Dichloroethane	5	ug/kg	0.73	ug/kg	20010122		Y	20	ug/kg	78	121	30		Y	50	ug/kg	56	126	38
948	cis-1,2-Dichloroethene	2.5	ug/kg	0.84	ug/kg	20010122		Y	20	ug/kg	77	114	30		Y	50	ug/kg	48	127	52
950	trans-1,2-Dichloroethene	2.5	ug/kg	0.73	ug/kg	20010122		Y	20	ug/kg	68	117	30		Y	50	ug/kg	47	127	58
943	1,1-Dichloroethene	5	ug/kg	1.2	ug/kg	20010308	C	Y	20	ug/kg	55	142	27	C	Y	50	ug/kg	43	147	27
986	1,2-Dichloropropane	5	ug/kg	0.75	ug/kg	20010122		Y	20	ug/kg	78	116	30		Y	50	ug/kg	54	125	43
998	cis-1,3-Dichloropropene	5	ug/kg	0.64	ug/kg	20010122		Y	20	ug/kg	71	125	30		Y	50	ug/kg	30	138	49
1000	trans-1,3-Dichloropropene	5	ug/kg	0.86	ug/kg	20010125		Y	20	ug/kg	67	125	30		Y	50	ug/kg	34	134	57
1332	Ethylbenzene	5	ug/kg	0.59	ug/kg	20010122		Y	20	ug/kg	79	114	30		Y	50	ug/kg	36	133	72
1515	2-Hexanone	20	ug/kg	1.7	ug/kg	20010308		Y	20	ug/kg	29	200	41		Y	50	ug/kg	20	190	70
1578	Isopropylbenzene	5	ug/kg	0.7	ug/kg	20010122														
1774	Methyl acetate	10	ug/kg	1.1	ug/kg	20010122														
1799	Methylcyclohexane	10	ug/kg	0.59	ug/kg	20010122														
1811	Methylene chloride	5	ug/kg	1.3	ug/kg	20010308		Y	20	ug/kg	58	130	30		Y	50	ug/kg	45	129	49
1845	4-Methyl-2-pentanone	20	ug/kg	1.9	ug/kg	20010308		Y	20	ug/kg	68	142	60		Y	50	ug/kg	42	143	60
2772	Methyl tert-butyl ether	20	ug/kg	0.84	ug/kg	20010122														

Structured Analysis Code: A-I5-QK-01-01

Target Analyte List: NC: OLM03.1/4.2 Volatile List

Matrix: SOLID
Extraction: Indiana Modified Method 5035 (IN 5035-M)
Method: Volatile Organics, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 4121			Detection Limits				Check List 924								Spike List 925						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2355	Styrene	5	ug/kg	0.5	ug/kg	20010122		Y	20	ug/kg	80	114	30		Y	50	ug/kg	23	136	65
2439	1,1,2,2-Tetrachloroethane	5	ug/kg	1.8	ug/kg	20010308		Y	20	ug/kg	70	133	30		Y	50	ug/kg	33	162	90
2445	Tetrachloroethene	5	ug/kg	1.2	ug/kg	20010308		Y	20	ug/kg	72	120	30		Y	50	ug/kg	31	137	81
2489	Toluene	5	ug/kg	0.53	ug/kg	20010122	C	Y	20	ug/kg	71	130	24	C	Y	50	ug/kg	46	147	24
2515	1,2,4-Trichlorobenzene	5	ug/kg	0.89	ug/kg	20010122														
2518	1,1,1-Trichloroethane	5	ug/kg	0.92	ug/kg	20010122		Y	20	ug/kg	67	123	30		Y	50	ug/kg	48	132	57
2522	1,1,2-Trichloroethane	5	ug/kg	1.2	ug/kg	20010308		Y	20	ug/kg	82	116	30		Y	50	ug/kg	58	128	52
2525	Trichloroethene	5	ug/kg	0.76	ug/kg	20010122	C	Y	20	ug/kg	70	131	23	C	Y	50	ug/kg	46	143	23
1428	Trichlorofluoromethane	5	ug/kg	1.1	ug/kg	20010122														
2566	1,1,2-Trichloro-1,2,2-trifluoroethane	5	ug/kg	0.93	ug/kg	20010125														
2613	Vinyl chloride	5	ug/kg	1.4	ug/kg	20010122		Y	20	ug/kg	24	152	30		Y	50	ug/kg	30	136	80
2627	Xylenes (total)	5	ug/kg	1.9	ug/kg	20010122		Y	60	ug/kg	80	114	30		Y	150	ug/kg	33	135	78
337	4-Bromofluorobenzene						X	Y	50	ug/kg	47	158	0	X	Y	50	ug/kg	47	158	0
2735	1,2-Dichloroethane-d4						X	Y	50	ug/kg	61	130	0	X	Y	50	ug/kg	61	130	0
2740	Toluene-d8						X	Y	50	ug/kg	60	143	0	X	Y	50	ug/kg	60	143	0
2863	Dibromofluoromethane						X	Y	50	ug/kg	59	138	0	X	Y	50	ug/kg	59	138	0

STL Reference Data Summary

Structured Analysis Code: A-13-QL-01-01

Target Analyte List: NC: OLM03.1/4.2 Semivolatile List

Matrix: SOLID
Extraction: SONICATION - Low Level
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 4122			Detection Limits				Check List 1114							Spike List 1115						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
1	Acenaphthene	330	ug/kg	51	ug/kg	20010227	C	Y	1700	ug/kg	44	108	44	C	Y	1700	ug/kg	13	133	44
5	Acenaphthylene	330	ug/kg	47	ug/kg	20010227		Y	1700	ug/kg	55	110	30		Y	1700	ug/kg	43	120	75
24	Acetophenone	330	ug/kg	50	ug/kg	20010219														
122	Anthracene	330	ug/kg	49	ug/kg	20010227		Y	1700	ug/kg	57	116	30		Y	1700	ug/kg	34	137	83
158	Atrazine	330	ug/kg	150	ug/kg	20010329														
3398	Benzaldehyde	330	ug/kg	170	ug/kg	20010329														
202	Benzo(a)anthracene	330	ug/kg	55	ug/kg	20010220		Y	1700	ug/kg	56	110	30		Y	1700	ug/kg	30	136	63
205	Benzo(b)fluoranthene	330	ug/kg	58	ug/kg	20010220		Y	1700	ug/kg	54	116	30		Y	1700	ug/kg	31	139	67
208	Benzo(k)fluoranthene	330	ug/kg	53	ug/kg	20010220		Y	1700	ug/kg	54	110	36		Y	1700	ug/kg	30	133	84
210	Benzo(ghi)perylene	330	ug/kg	55	ug/kg	20010220		Y	1700	ug/kg	48	124	31		Y	1700	ug/kg	23	142	89
211	Benzo(a)pyrene	330	ug/kg	60	ug/kg	20010220		Y	1700	ug/kg	56	115	30		Y	1700	ug/kg	28	142	86
3474	1,1'-Biphenyl	330	ug/kg	140	ug/kg	20010329														
289	bis(2-Chloroethoxy)methane	330	ug/kg	47	ug/kg	20010227		Y	1700	ug/kg	59	110	30		Y	1700	ug/kg	46	116	97
293	bis(2-Chloroethyl) ether	330	ug/kg	51	ug/kg	20010227		Y	1700	ug/kg	62	111	30		Y	1700	ug/kg	49	123	93
302	bis(2-Ethylhexyl) phthalate	330	ug/kg	54	ug/kg	20010220		Y	1700	ug/kg	53	115	30		Y	1700	ug/kg	36	131	54
348	4-Bromophenyl phenyl ether	330	ug/kg	61	ug/kg	20010131		Y	1700	ug/kg	58	110	30		Y	1700	ug/kg	47	121	95
403	Butyl benzyl phthalate	330	ug/kg	51	ug/kg	20010220		Y	1700	ug/kg	54	113	30		Y	1700	ug/kg	34	136	94
5101	Caprolactam	330	ug/kg	180	ug/kg	20010329														
2751	Carbazole	330	ug/kg	52	ug/kg	20010227		Y	1700	ug/kg	56	117	31		Y	1700	ug/kg	40	130	85
518	4-Chloroaniline	330	ug/kg	51	ug/kg	20010220		Y	1700	ug/kg	28	110	33		Y	1700	ug/kg	10	110	99
578	4-Chloro-3-methylphenol	330	ug/kg	51	ug/kg	20010131	C	Y	1700	ug/kg	43	110	55	C	Y	1700	ug/kg	17	128	55
589	2-Chloronaphthalene	330	ug/kg	48	ug/kg	20010227		Y	1700	ug/kg	52	116	30		Y	1700	ug/kg	43	125	75
600	2-Chlorophenol	330	ug/kg	52	ug/kg	20010227	C	Y	1700	ug/kg	43	110	54	C	Y	1700	ug/kg	17	116	54
602	4-Chlorophenyl phenyl ether	330	ug/kg	52	ug/kg	20010227		Y	1700	ug/kg	59	110	30		Y	1700	ug/kg	47	119	74
633	Chrysene	330	ug/kg	46	ug/kg	20010424		Y	1700	ug/kg	56	110	30		Y	1700	ug/kg	28	139	63
860	Dibenz(a,h)anthracene	330	ug/kg	51	ug/kg	20010220		Y	1700	ug/kg	50	129	33		Y	1700	ug/kg	31	142	99
863	Dibenzofuran	330	ug/kg	50	ug/kg	20010227		Y	1700	ug/kg	57	110	30		Y	1700	ug/kg	35	132	58
891	Di-n-butyl phthalate	330	ug/kg	150	ug/kg	20010329		Y	1700	ug/kg	58	114	30		Y	1700	ug/kg	44	128	74
918	3,3'-Dichlorobenzidine	1600	ug/kg	39	ug/kg	20010220		Y	1700	ug/kg	22	110	33		Y	1700	ug/kg	10	110	99
971	2,4-Dichlorophenol	330	ug/kg	50	ug/kg	20010220		Y	1700	ug/kg	60	111	30		Y	1700	ug/kg	45	122	85
1082	Diethyl phthalate	330	ug/kg	150	ug/kg	20010329		Y	1700	ug/kg	57	112	30		Y	1700	ug/kg	45	122	76
1145	2,4-Dimethylphenol	330	ug/kg	80	ug/kg	20010430		Y	1700	ug/kg	41	110	35		Y	1700	ug/kg	31	119	99
1149	Dimethyl phthalate	330	ug/kg	160	ug/kg	20010329		Y	1700	ug/kg	60	111	30		Y	1700	ug/kg	48	124	75
1167	4,6-Dinitro-2-methylphenol	1600	ug/kg	160	ug/kg	20010329		Y	1700	ug/kg	37	126	37		Y	1700	ug/kg	10	142	99
1187	2,4-Dinitrophenol	1600	ug/kg	110	ug/kg	20010329		Y	1700	ug/kg	15	131	53		Y	1700	ug/kg	10	149	99
1191	2,4-Dinitrotoluene	330	ug/kg	46	ug/kg	20010227	C	Y	1700	ug/kg	48	111	45	C	Y	1700	ug/kg	10	171	45

Structured Analysis Code: A-13-QL-01-01

Target Analyte List: NC: OLM03.1/4.2 Semivolatile List

Matrix: SOLID
Extraction: SONICATION - Low Level
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

	Target List 4122	Detection Limits					Check List 1114							Spike List 1115						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
1193	2,6-Dinitrotoluene	330	ug/kg	48	ug/kg	20010131		Y	1700	ug/kg	61	121	30		Y	1700	ug/kg	45	124	99
1162	Di-n-octyl phthalate	330	ug/kg	54	ug/kg	20010220		Y	1700	ug/kg	47	129	33		Y	1700	ug/kg	34	143	99
1414	Fluoranthene	330	ug/kg	56	ug/kg	20010227		Y	1700	ug/kg	57	118	30		Y	1700	ug/kg	25	150	92
1417	Fluorene	330	ug/kg	48	ug/kg	20010227		Y	1700	ug/kg	57	110	30		Y	1700	ug/kg	35	133	80
1482	Hexachlorobenzene	330	ug/kg	56	ug/kg	20010131		Y	1700	ug/kg	57	111	30		Y	1700	ug/kg	45	123	74
1489	Hexachlorobutadiene	330	ug/kg	45	ug/kg	20010227		Y	1700	ug/kg	56	113	30		Y	1700	ug/kg	43	126	86
1492	Hexachlorocyclopentadiene	1600	ug/kg	60	ug/kg	20010220		Y	1700	ug/kg	10	113	57		Y	1700	ug/kg	10	110	99
1497	Hexachloroethane	330	ug/kg	48	ug/kg	20010227		Y	1700	ug/kg	61	110	30		Y	1700	ug/kg	36	134	97
1535	Indeno(1,2,3-cd)pyrene	330	ug/kg	70	ug/kg	20010220		Y	1700	ug/kg	44	128	54		Y	1700	ug/kg	20	147	73
1566	Isophorone	330	ug/kg	44	ug/kg	20010227		Y	1700	ug/kg	58	110	30		Y	1700	ug/kg	45	116	95
1829	2-Methylnaphthalene	330	ug/kg	48	ug/kg	20010227		Y	1700	ug/kg	58	110	30		Y	1700	ug/kg	30	137	79
1851	2-Methylphenol	330	ug/kg	46	ug/kg	20010220		Y	1700	ug/kg	51	110	30		Y	1700	ug/kg	41	116	99
1857	4-Methylphenol	330	ug/kg	47	ug/kg	20010227		Y	1700	ug/kg	56	110	30		Y	1700	ug/kg	36	123	86
1932	Naphthalene	330	ug/kg	49	ug/kg	20010227		Y	1700	ug/kg	59	110	30		Y	1700	ug/kg	31	138	65
1960	2-Nitroaniline	1600	ug/kg	46	ug/kg	20010227		Y	1700	ug/kg	58	113	30		Y	1700	ug/kg	42	126	99
1964	3-Nitroaniline	1600	ug/kg	140	ug/kg	20010329		Y	1700	ug/kg	42	110	37		Y	1700	ug/kg	17	113	99
1968	4-Nitroaniline	1600	ug/kg	68	ug/kg	20010424		Y	1700	ug/kg	42	116	40		Y	1700	ug/kg	14	125	99
1972	Nitrobenzene	330	ug/kg	46	ug/kg	20010227		Y	1700	ug/kg	62	110	30		Y	1700	ug/kg	49	120	99
1998	2-Nitrophenol	330	ug/kg	46	ug/kg	20010131		Y	1700	ug/kg	55	118	30		Y	1700	ug/kg	44	125	99
2001	4-Nitrophenol	1600	ug/kg	190	ug/kg	20010329	C	Y	1700	ug/kg	22	128	64	C	Y	1700	ug/kg	10	148	64
2028	N-Nitrosodiphenylamine	330	ug/kg	49	ug/kg	20010227		Y	1700	ug/kg	58	113	30		Y	1700	ug/kg	37	135	94
2024	N-Nitrosodi-n-propylamine	330	ug/kg	44	ug/kg	20010220	C	Y	1700	ug/kg	38	110	50	C	Y	1700	ug/kg	12	128	50
3597	2,2'-oxybis(1-Chloropropane)	330	ug/kg	52	ug/kg	20010227														
2118	Pentachlorophenol	330	ug/kg	120	ug/kg	20010329	C	Y	1700	ug/kg	10	123	87	C	Y	1700	ug/kg	10	144	87
2154	Phenanthrene	330	ug/kg	52	ug/kg	20010227		Y	1700	ug/kg	56	110	30		Y	1700	ug/kg	24	145	81
2155	Phenol	330	ug/kg	49	ug/kg	20010227	C	Y	1700	ug/kg	35	110	50	C	Y	1700	ug/kg	10	148	50
2252	Pyrene	330	ug/kg	51	ug/kg	20010220	C	Y	1700	ug/kg	42	122	66	C	Y	1700	ug/kg	10	218	66
2555	2,4,5-Trichlorophenol	330	ug/kg	55	ug/kg	20010227		Y	1700	ug/kg	52	115	37		Y	1700	ug/kg	39	123	99
2559	2,4,6-Trichlorophenol	330	ug/kg	63	ug/kg	20010220		Y	1700	ug/kg	51	112	30		Y	1700	ug/kg	43	123	99
1425	2-Fluorobiphenyl						X	Y	1700	ug/kg	43	110	0	X	Y	1700	ug/kg	43	110	0
1426	2-Fluorophenol						X	Y	2500	ug/kg	11	116	0	X	Y	2500	ug/kg	11	116	0
2512	2,4,6-Tribromophenol						X	Y	2500	ug/kg	35	116	0	X	Y	2500	ug/kg	35	116	0
2736	Nitrobenzene-d5						X	Y	1700	ug/kg	42	110	0	X	Y	1700	ug/kg	42	110	0
2737	Phenol-d5						X	Y	2500	ug/kg	25	115	0	X	Y	2500	ug/kg	25	115	0
2738	Terphenyl-d14						X	Y	1700	ug/kg	37	137	0	X	Y	1700	ug/kg	37	137	0

STL Reference Data Summary

Structured Analysis Code: A-71-QH-01-01

Target Analyte List: Q: Aroclors Only Standard List

Matrix: SOLID
Extraction: SONICATION w/ACID STRIP (PCB)
Method: PCBs (8082)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 2207		Detection Limits				Check List 928							Spike List 929						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2082	Aroclor 1016	33	ug/kg	5.3	ug/kg	20000222	C	Y	333	ug/kg	49	122	39	C	Y	333	ug/kg	26	144	39
2085	Aroclor 1221	33	ug/kg	19	ug/kg	20000222														
2088	Aroclor 1232	33	ug/kg	11	ug/kg	20000222														
2091	Aroclor 1242	33	ug/kg	18	ug/kg	20000222														
2094	Aroclor 1248	33	ug/kg	4.6	ug/kg	20000222														
2097	Aroclor 1254	33	ug/kg	20	ug/kg	20000222														
2100	Aroclor 1260	33	ug/kg	7.4	ug/kg	20000222	C	Y	333	ug/kg	51	127	33	C	Y	333	ug/kg	37	138	33
2732	Decachlorobiphenyl						X	Y	6.67	ug/kg	23	141	0	X	Y	6.67	ug/kg	23	141	0
2739	Tetrachloro-m-xylene						X	Y	6.67	ug/kg	31	127	0	X	Y	6.67	ug/kg	31	127	0

STL Reference Data Summary

Structured Analysis Code: A-46-QO-01-01

Target Analyte List: Q: CLP ICP Metals TAL Standard List

Matrix: SOLID
Extraction: METALS, TOTAL - Soils
Method: Inductively Coupled Plasma (6010B)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 2600			Detection Limits				Check List 906							Spike List 907						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
88	Aluminum	20	mg/kg	1.1	mg/kg	20000224		Y	200	mg/kg	80	120	20		Y	200	mg/kg	75	125	20
128	Antimony	6	mg/kg	0.49	mg/kg	20000224		Y	50	mg/kg	80	120	20		Y	50	mg/kg	75	125	20
194	Barium	20	mg/kg	0.13	mg/kg	20000224		Y	200	mg/kg	80	120	20		Y	200	mg/kg	75	125	20
222	Beryllium	0.5	mg/kg	0.046	mg/kg	20000224		Y	5	mg/kg	80	120	20		Y	5	mg/kg	75	125	20
411	Cadmium	0.5	mg/kg	0.043	mg/kg	20000224		Y	5	mg/kg	80	120	20		Y	5	mg/kg	75	125	20
413	Calcium	500	mg/kg	37	mg/kg	20000224		Y	5000	mg/kg	80	120	20		Y	5000	mg/kg	75	125	20
2952	Chromium	1	mg/kg	0.38	mg/kg	20000412		Y	20	mg/kg	80	120	20		Y	20	mg/kg	75	125	20
637	Cobalt	5	mg/kg	0.15	mg/kg	20000224		Y	50	mg/kg	80	120	20		Y	50	mg/kg	75	125	20
643	Copper	2.5	mg/kg	0.27	mg/kg	20000412		Y	25	mg/kg	80	120	20		Y	25	mg/kg	75	125	20
1539	Iron	10	mg/kg	6.6	mg/kg	20000412		Y	100	mg/kg	73	137	20		Y	100	mg/kg	75	125	20
1618	Magnesium	500	mg/kg	12	mg/kg	20000224		Y	5000	mg/kg	80	120	20		Y	5000	mg/kg	75	125	20
1659	Manganese	1.5	mg/kg	0.15	mg/kg	20000224		Y	50	mg/kg	80	120	20		Y	50	mg/kg	75	125	20
1956	Nickel	4	mg/kg	0.27	mg/kg	20000224		Y	50	mg/kg	80	120	20		Y	50	mg/kg	75	125	20
2214	Potassium	500	mg/kg	5.1	mg/kg	20000224		Y	5000	mg/kg	80	120	20		Y	5000	mg/kg	75	125	20
2285	Silver	1	mg/kg	0.15	mg/kg	20000224		Y	5	mg/kg	80	120	20		Y	5	mg/kg	75	125	20
2315	Sodium	500	mg/kg	50	mg/kg	20000224		Y	5000	mg/kg	80	120	20		Y	5000	mg/kg	75	125	20
2607	Vanadium	5	mg/kg	0.13	mg/kg	20000224		Y	50	mg/kg	80	120	20		Y	50	mg/kg	75	125	20
2649	Zinc	2	mg/kg	1.2	mg/kg	20000412		Y	50	mg/kg	80	120	20		Y	50	mg/kg	75	125	20

STL Reference Data Summary

Structured Analysis Code: A-46-QM-01-01

Target Analyte List: Q: CLP Trace ICP Metals TAL Standard List

Matrix: SOLID
Extraction: METALS, TOTAL - Soils
Method: Inductively Coupled Plasma (6010B Trace)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 2610		Detection Limits				Check List 906							Spike List 907						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
140	Arsenic	1.0	mg/kg	0.3	mg/kg	20000224		Y	200	mg/kg	80	120	20		Y	200	mg/kg	75	125	20
1605	Lead	0.3	mg/kg	0.24	mg/kg	20000224		Y	50	mg/kg	80	120	20		Y	50	mg/kg	75	125	20
2281	Selenium	0.5	mg/kg	0.31	mg/kg	20000224		Y	200	mg/kg	80	120	20		Y	200	mg/kg	75	125	20
2477	Thallium	1.0	mg/kg	0.5	mg/kg	20000224		Y	200	mg/kg	80	120	20		Y	200	mg/kg	75	125	20

STL Reference Data Summary

Structured Analysis Code: A-70-O9-01-01
Target Analyte List: All Analytes

Matrix: SOLID
Extraction: METALS, TOTAL (Method Exclusive) - Solids
Method: Mercury (7471A, Cold Vapor) - Solids
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits					Check List 1021							Spike List 1026						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
1701	Mercury	0.1	mg/kg	0.0084	mg/kg	20000613		Y	.833333	mg/kg	52	127	20		Y	0.1666	mg/kg	10	209	20

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STL Reference Data Summary

Structured Analysis Code: A-06-QP-01-01
Target Analyte List: All Analytes

Matrix: SOLID
Extraction: DISTILLATION, MICRO/MIDI - Acid
Method: Cyanide, Total (9012A, Automated)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List			Detection Limits				Check List 1180							Spike List 1181						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
667	Total Cyanide	0.5	mg/kg	0.17	mg/kg	20000727		Y			65	124	20		Y	2.0	mg/kg	23	155	94

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STL Reference Data Summary

Structured Analysis Code: A-06-N4-01-01

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: DISTILLATION, MICRO/MIDI - Acid
Method: Cyanide, Amenable (9012)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List			Detection Limits				Check List 1180							Spike List 1181						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3623	Amenable Cyanide	0.5	mg/kg	0.5	mg/kg	20010927		Y			65	124	20		Y	2.0	mg/kg	23	155	94
664	Cyanide Amenable to Chlorination	0.5	mg/kg	0.5	mg/kg	20010927		Y			65	124	20		Y	2.0	mg/kg	23	155	94

STL Reference Data Summary

Structured Analysis Code: A-82-VR-01-01
Target Analyte List: All Analytes

Matrix: SOLID
Extraction: LEACHATE, DI (Routine)
Method: Carbon, Total Organic "TOC" (WALKLEY) - Solids
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List			Detection Limits				Check List 1215							Spike List 1215						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
470	Total Organic Carbon	100	mg/kg	100	mg/kg	20010924	C	Y			51	128	20	C	Y			51	128	20

STL Reference Data Summary

Structured Analysis Code: D-1U-QH-01-01

Target Analyte List: Q: Aroclors Only Standard List

Matrix: WIPE
Extraction: EXTRACTION, SOLID/SOLVENT (Auto shaker) w/ACID STR
Method: PCBs (8082)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 2207		Detection Limits				Check List 928							Spike List 929						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2082	Aroclor 1016	4	ug	4	ug	20001222	C	Y	10	mg/kg	90	138	30	C	Y			26	144	39
2085	Aroclor 1221	4	ug	4	ug	20001222														
2088	Aroclor 1232	4	ug	4	ug	20001222														
2091	Aroclor 1242	4	ug	4	ug	20001222														
2094	Aroclor 1248	4	ug	4	ug	20001222														
2097	Aroclor 1254	4	ug	4	ug	20001222														
2100	Aroclor 1260	4	ug	4	ug	20001222														
2732	Decachlorobiphenyl						C	Y	10	mg/kg	88	125	30	C	Y			37	138	33
2739	Tetrachloro-m-xylene						X	Y	0.2	mg/kg	64	181	0	X	Y	0.2	mg/kg	64	181	0
							X	Y	0.2	mg/kg	66	135	0	X	Y	0.2	mg/kg	66	135	0

ATTACHMENT 4

FIELD AUDIT CHECKLIST

FIELD CHECKLIST

Briefing with Project Manager

SIGNATURE OF AUDITOR _____ DATE OF AUDIT _____

PROJECT MANAGER _____ PROJECT NO. _____

PROJECT LOCATION _____

TYPE OF INVESTIGATION _____
(authority, agency)

Yes ___ No ___ N/A ___ 1. Was a project plan prepared? If yes, what items are addressed in the Plan?

_____

_____

_____

Yes ___ No ___ N/A ___ 2. Were additional instructions given to project participants (i.e., changes in project plan)? If yes, describe these changes.

_____

_____

_____

Yes ___ No ___ N/A ___ 3. Is there a written list of sampling locations and descriptions? If yes, describe where documents are.

_____

_____

_____

Yes ___ No ___ N/A ___ 4. Is there a map of sampling location? If yes, where is the map?

_____

_____

_____

FIELD CHECKLIST

Field Observations

Yes ___ No ___ N/A ___ 1. Are the number, frequency and types of field measurements and observations taken as specified in the project plan or as directed by the project manager? If yes, where are they recorded?

Yes ___ No ___ N/A ___ 2. Are samples collected in the types of containers specified for each type of analysis? If no, what kind of sample containers are used?

Yes ___ No ___ N/A ___ 3. Are samples preserved as required? If no or N/A, explain.

Yes ___ No ___ N/A ___ 4. Are the number, frequency and types of samples collected as specified in the project plan or as directed by the project manager? If no, explain why not.

Yes ___ No ___ N/A ___ 5. Are samples packed for preservation when required (i.e., packed in ice, etc.)? If no or N/A, explain why.

Yes ___ No ___ N/A ___ 6. Is sample custody maintained? How?

FIELD CHECKLIST

Document Control

Yes ___ No ___ N/A ___ 1. Are samples identified with sample tags? If no, how are samples identified?

Yes ___ No ___ N/A ___ 2. Are sample tags completed (e.g., station no., location, date, time, analyses, signatures on samples, type, preservatives, etc.)? If yes, describe types of information recorded.

Yes ___ No ___ N/A ___ 3. Are samples collected listed on a chain-of-custody record? If yes, describe the type of chain-of-custody record used and what information is recorded.

Yes ___ No ___ N/A ___ 4. If used, are the sample tag numbers recorded on the chain-of-custody documents?

Yes ___ No ___ N/A ___ 5. Does information on sample tags and chain-of-custody records match?

Yes ___ No ___ N/A ___ 6. Does the chain-of-custody record indicate the method of sample shipment?

Yes ___ No ___ N/A ___ 7. Is the chain-of-custody record included with the samples in the shipping container?

Yes ___ No ___ N/A ___ 8. If used, do the sample traffic reports agree with the sample tags?

Yes ___ No ___ N/A ___ 9. If used, are blank samples identified?

Yes ___ No ___ N/A ___ 10. If collected, are duplicate samples identified on sample tags and chain-of-custody records?

Yes ___ No ___ N/A ___ 11. If used, are spiked samples identified?

Yes ___ No ___ N/A ___ 12. Are logbooks signed by the individual who checked out the logbook from the project coordinator?

Yes ___ No ___ N/A ___ 13. Are logbooks dated upon receipt from the project coordinator?

Yes ___ No ___ N/A ___ 14. Are logbooks project-specific (by logbook or by page)?

Yes ___ No ___ N/A ___ 15. Are logbook entries dated and identified by author?

Yes ___ No ___ N/A ___ 16. Are photographs documented in logbooks (e.g., time, date, description of subject, photographer, etc.)?

Yes ___ No ___ N/A ___ 17. If film from a self-developing camera is used, are photos matched with logbook documentation?

Yes ___ No ___ N/A ___ 18. Are sample tag numbers recorded? If yes, describe where they are recorded.

Yes ___ No ___ N/A ___ 19. Are calibration of pH meters, conductivity meters, etc., documented? If yes, describe where this is documented.

Yes ___ No ___ N/A ___ 20. Are amendments to the project plan documented? If yes, describe where the amendments are documented.

FIELD CHECKLIST

Debriefing with Project Manager

Yes ___ No ___ N/A ___ 1. Was a debriefing held with project manager and/or other participants?

Yes ___ No ___ N/A ___ 2. Were any recommendations made to the project participants during the debriefing? If yes, list recommendations.

APPENDIX C

QUALITY ASSURANCE PROJECT PLAN
RCRA FACILITY INVESTIGATION WORK PLAN

GM POWERTRAIN - BEDFORD PLANT
105 GM DRIVE
BEDFORD, INDIANA

EPA ID# IND 006036099

Prepared for:
GENERAL MOTORS CORPORATION

Prepared by:
Conestoga-Rovers & Associates

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web: http:\\www.CRAworld.com

NOVEMBER 5, 2001
REF. NO. 13968 (9)

QUALITY ASSURANCE PROJECT PLAN (QAPP)

FOR THE RCRA FACILITY INVESTIGATION WORK PLAN
GENERAL MOTORS POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA
U.S. EPA ID NUMBER – IND 006036099
REVISION NUMBER 0
NOVEMBER 5, 2001

Prepared By: Conestoga-Rovers & Associates (CRA)

Prepared For: General Motors Corporation (GM)

Approved By: _____ Date: _____
Cheryl Hiatt
GM Project Manager

Approved By: _____ Date: _____
Bill Steinmann
CRA Project Manager

Approved By: _____ Date: _____
Paul Wiseman
CRA QA Officer

Approved By: _____ Date: _____
Jeff Nichols
CRA Field QA Officer

Approved By: _____ Date: _____
Amy McCormick
STL Project Manager

Approved By: _____ Date: _____
Opal Davis-Johnson
STL QA Officer

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1.0 PROJECT DESCRIPTION

The following outlines the scope of the Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) to be performed in accordance with the Voluntary Corrective Action Agreement executed between the United States Environmental Protection Agency (U.S. EPA) and General Motors Corporation (GM) on March, 2001 for the GM Powertrain – Bedford Plant (Facility) in Bedford, Indiana, U.S. EPA Facility Identification Number IND 006036099. This Quality Assurance Project Plan (QAPP) presents the organization, objectives, planned activities, and specific quality assurance / quality control (QA/QC) procedures associated with the RFI for the Facility. Protocols for sample collection, sample handling and storage, chain-of-custody procedures, and laboratory and field analyses are described or specifically referenced to related investigation documents.

This QAPP addresses the QA/QC elements in the U.S. EPA Region 5 QAPP policy presented in the "U. S. EPA RCRA QAPP Instructions" dated April 1998 and other relevant guidance documents, including "The Use of Field Methods to Support RFI Streamlining", U.S. EPA Region 5 Memorandum, June 20, 1999. The QA/QC procedures described in this QAPP are consistent with U.S. EPA guidance in general.

1.1. INTRODUCTION

This QAPP has been prepared for GM by Conestoga-Rovers & Associates (CRA). The QAPP is a component of the RFI Work Plan, that also includes a Field Sampling Plan (FSP), Project Management Plan (PMP), Data Management Plan (DMP), Health and Safety Plan (HASP), and Community Relations Plan (CRP).

1.1.1 PROJECT OBJECTIVES AND DECISION STATEMENT

The primary objectives for data collection activities include:

i) Define the nature and extent of releases of hazardous wastes and/or hazardous constituents in environmental media (soil, sediment, groundwater, and/or surface water) at the Facility;

ii) Collect sufficient data and information to evaluate the risk to human health and the environment, if any, associated with any releases of hazardous waste and/or hazardous constituents; and

iii) Collect sufficient data for all contaminated media to support the Environmental Indicators Determination (i.e., actual or potential receptors and migration pathways).

The Decision Statement for the investigation is as follows:

i) Determine whether interim remedial actions are necessary to control current unacceptable risks, if any, to human health or the environment; and

ii) Provide data for the development of a study of appropriate corrective measures (if warranted) to control current and future unacceptable risks to human health and the environment.

Associated specific objectives for field and laboratory data collection are discussed in Section 1.4 of this plan.

1.1.2 PROJECT STATUS/PHASE

The project status and investigation approach are presented in the RFI Work Plan. Section 2.0 summarizes the Facility background. Section 5.0 describes the Areas of Interest (AOI) to be investigated and gives a brief description of each area and the proposed investigation for each AOI at the Facility. Section 6.0 describes the data evaluation procedures for this project will be conducted.

1.1.3 QAPP PREPARATION GUIDELINES

As previously noted, this QAPP has been prepared to address the QA/QC elements in the "U.S. EPA Region 5 QAPP Instructions" dated April 1998.

1.2 FACILITY DESCRIPTION

The general facility description is provided in Section 2, 3, and 4 of the RFI Work Plan and is incorporated here by reference.

Location	Work Plan Section 2.1
Facility Dimensions/Borders	Work Plan Section 2.1 & 3.1
Topography	Work Plan Section 4.1
Local Geology and Hydrology	Work Plan Section 4.2 & 4.3
Surrounding Land Use	Work Plan Section 3.1

1.3 FACILITY HISTORY

The Facility history is provided in Section 2.0 of the RFI Work Plan and incorporated here by reference.

1.4 PROJECT OBJECTIVES AND INTENDED DATA USE

The objectives and intended data uses include:

1.4.1 TARGET PARAMETER LIST

The investigative program includes the sampling and analysis of environmental media for the presence of organic and inorganic constituents based on current and historical operations at the Facility. The field and laboratory parameters are summarized below and presented in Table 1.1 with sample matrices, and the quantity of investigative and quality control samples.

1.4.1.1 FIELD PARAMETERS

Concurrent with sample collection, several field parameters will be determined by the field sampling personnel. Soils and solid matrices field parameters will include visual

observations, odor identification, and organics screening using handheld monitoring equipment. For aqueous samples (groundwater and surface water samples), at a minimum, the following parameters will be determined with field testing equipment: pH, specific conductivity, dissolved oxygen, turbidity, and temperature. The low-flow purging techniques utilized for groundwater sampling will include the field measurement of oxidation/reduction potential (ORP) as an additional field parameter.

1.4.1.2 LABORATORY PARAMETERS

The laboratory parameters include target compound list (TCL) volatile organic compounds (VOC), TCL semi volatile organic compounds (SVOC), TCL polychlorinated biphenyls (PCB), and target analyte list (TAL) inorganics including cyanide and excluding earth metals calcium, magnesium, potassium, and sodium. These four metals are not included due to low risk to human health and environment. In addition, chloride and total organic carbon (TOC) will be determined on select samples. Table 1.2 presents the estimated quantitation limits (EQL) for each compound or analyte. Method detection limit (MDL) studies have been performed by Severn Trent Laboratories (STL) in accordance with their SOPs developed to address the procedure established in the Federal Register, Volume 49, number 209, October 26, 1984 pp. 198-199, the results of these MDL studies are presented in Attachment 3.

1.5 SAMPLING LOCATIONS

The RFI Work Plan provides a summary and rationale for the location of soil, concrete, sediment, surface water, and groundwater sampling within each AOI at the Facility. It is possible, however, that depending on the nature of encountered field conditions, sampling locations may change. The person responsible for making such decisions will be the Field Quality Assurance (QA) Officer whose responsibilities are described in Section 2.0 of this QAPP. Any change of the sampling strategy will only be implemented after approval from the GM Project Manager.

1.6 PROJECT SCHEDULE

The schedule of projected milestones is presented in Section 8.0 of the RFI Work Plan.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The General Motors Project Manager will have the primary responsibility for the implementation of the RFI Work Plan. STL in North Canton, Ohio will perform the analyses of environmental samples collected at the Facility. This section defines the roles and responsibilities of the individuals who will perform the RFI activities.

Figure A.4.1 of the Project Management Plan provides the organization chart for the project team.

2.1 MANAGEMENT RESPONSIBILITIES

A description of the project organization and responsibilities of key personnel is presented in detail in the Project Management Plan. Management responsibilities are as follows:

Peter Ramanauskas– U.S. EPA Project Coordinator

The U.S. EPA Project Coordinator has responsibility for oversight of all phases of the Investigation.

Cheryl Hiatt – GM Project Manager

The GM Project Manager is responsible for implementing the project, and has the authority to commit the resources necessary to meet project objectives and requirements. The GM Project Manager's primary function is to ensure that technical, financial, and scheduling objectives are achieved successfully. The GM Project Manager will provide the major point of contact and control matters concerning the project and represent the project team at regulatory agency meetings and public hearings. The Project Manager will define project objectives and develop a detailed project schedule. The GM Project Manager will establish project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task. The GM Project Manager also provides approval of the QAPP.

Glenn Turchan – CRA Project Director

The Project Director will provide final review of significant work products and may participate in technical meetings with the U.S. EPA. The Project Director will ensure that overall technical quality is maintained. He will be actively involved in the direction of the project. The Project Director has overall responsibility for ensuring that the project meets RFI objectives. The Project Director will:

i) Acquire and apply technical and other resources as needed to ensure performance within budget and schedule restraints;
ii) Review work performed on each task to ensure quality, responsiveness, and timeliness;
iii) Be responsible for the preparation and quality of interim and final reports; and
iv) Communicate with GM about the progress of the project.

Jim McGuigan – CRA Senior Project Engineer/Advisor

The Senior Project Engineer/Advisor will assist the Project Director and the Project Manager in day-to-day project management. The Senior Project Engineer/Advisor is responsible for coordinating all field activities and the procurement of project subcontractors. Additional responsibilities include:

i) Assisting in monitoring the RFI progress and quality;
ii) Preparing and reviewing RFI reports;
iii) Providing technical support of project activities; and

Bill Steinmann – CRA Project Manager

The Project Manager is responsible for managing the implementation of the RFI Work Plan and coordinating the collection of data pertaining to the Investigation. The Project Manager is responsible for technical quality control and project oversight. The Project Manager also provides approval of the QAPP.

2.2 QUALITY ASSURANCE RESPONSIBILITIES

The Quality Assurance team will consist of a Quality Assurance Officer and the Project Manager. Quality Assurance responsibilities are described as follows:

Paul Wiseman – CRA Quality Assurance (QA) Officer

The QA Officer reports directly to the CRA Project Manager and will be responsible for ensuring that all QA/QC procedures are being followed. The QAO will be responsible for overseeing the review of all field and laboratory data. Additional responsibilities include:

i) Assuring the application and effectiveness of the QAPP by the analytical laboratory and the project staff;
ii) Conducting internal QA/QC of the investigation activities;
iii) Providing input to the Project Director, Project Manager and the Project Engineer as to corrective actions required resulting from the above-mentioned evaluations;
iv) Preparation and review of data validation and audit reports; and
v) Approval of the QAPP.

The QA Officer will be assisted by the data validation staff in the evaluation and validation of field and laboratory generated data. The QA Officer will monitor the performance of the laboratory to ensure that the Data Quality Objectives for the project are met.

U.S. EPA Quality Assurance Officer

The U.S. EPA Quality Assurance Officer may review this QAPP. Additional U.S. EPA Region 5 responsibilities for the project may include:

i) Conducting external performance and system audits of the RFI laboratories; and
ii) Reviewing and evaluating field and analytical laboratory procedures.

2.3 LABORATORY RESPONSIBILITIES

STL will provide laboratory services in support of the RFI Work Plan.

Amy McCormick - STL Project Manager

The Laboratory Project Manager will report directly to the CRA QA Officer and will be responsible for ensuring all resources of the laboratory are available on an as-required basis. The Laboratory Project Manager will also be responsible for the approval of the final analytical reports and approval of the laboratory's ability to adhere to the QAPP.

Chris Oprandi - STL Operations Manager

The Laboratory Operations Manager will report to the Laboratory Project Manager and will be responsible for coordinating laboratory analysis, supervising in-house chain-of-custody reports, scheduling sample analyses, overseeing data review, and overseeing preparation of analytical reports

Opal Davis-Johnson -STL QA Officer

The Laboratory QA Officer will have sole responsibility for review and validation of the analytical laboratory data generated as part of the RFI. The Laboratory QA Officer will sign all final laboratory data reports provided from the analysis of the project samples and will provide Case Narrative descriptions of any data quality issues encountered during the analyses conducted by the laboratory. The QA Officer will also define appropriate QA procedures, overview QA/QC documentation, and approve this QAPP.

Lois Ezzo - STL Sample Custodian

The Laboratory Sample Custodian will report to the Laboratory Operations Manager and will be responsible for the following:

i) Receiving and inspecting the incoming sample containers;
ii) Recording the condition of the incoming sample containers;
iii) Signing appropriate documents;
iv) Verifying chain-of-custody and its correctness;
v) Notifying the Project Manager and Operations Manager of sample receipt and inspection;
vi) Assigning a unique identification number and customer number, and entering each into the sample receiving log;

vii) Initiating transfer of samples to lab sections; and
viii) Controlling and monitoring access/storage of samples and extracts.

Laboratory Technical Personnel

The laboratory technical staff will have the primary responsibility in the performance of sample analysis and the execution of the QA procedures developed to determine the data quality. These activities will include the proper preparation and analysis of the project samples in accordance with the contract laboratory's Quality Assurance Manual and associated Standard Operating Procedures.

Data Validation Staff

The data validation staff will be independent of the laboratory and familiar with the analytical procedures performed. The validation will include a review of each validation criterion as prescribed by the guidelines presented in Section 12.2 of this document and be presented in a formal written report for submittal to CRA Senior Project Engineer/Advisor.

2.4 FIELD RESPONSIBILITIES

Jeffrey Nichols – Field QA Officer

The Field QA Officer is responsible for the overall operation of the field team and reports directly to the CRA Senior Project Engineer/Advisor and CRA Project Manager. The Field QA Officer works with the project Health & Safety Officer to conduct operations in compliance with the project Health & Safety Plan. The Field QA Officer will facilitate communication and coordinate efforts between the CRA Project Manager and the field team members. Other responsibilities include:

i) Developing and implementing field-related work plans, ensuring schedule compliance, and adhering to management-developed project requirements;
ii) Coordinating and managing field staff, including sampling and drilling;
iii) Performing field system audits;
iv) Overseeing quality control for technical data provided by the field staff;
v) Preparing, and approving of text and graphics required for field team efforts;

vi) Coordinating and overseeing technical efforts of subcontractors assisting the field team;

vii) Identifying problems in the field, resolving difficulties in consultation with the CRA Senior Project Engineer/Advisor, CRA Project QAO, and CRA Project Manager, implementing and documenting corrective action procedures;

viii) Approving the QAPP; and

ix) Participating in preparation of the final reports.

Field Team Personnel

Field Team Personnel involved in the RFI will be responsible for:

i) Performance of field activities as detailed in the RFI Work Plan and in compliance with the DQO's outlined in this QAPP; and

ii) Immediately reporting any accidents and/or unsafe conditions to the Site Health & Safety Officer and taking all reasonable precautions to prevent injury.

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The RFI Work Plan and associated QAPP are designed to produce data of the quality necessary to achieve the project objectives and meet or exceed the minimum standard requirements for field and analytical methods. The overall QA objective for measurement data is to develop and implement procedures for field sampling, chain-of-custody, lab analyses, and reporting that will provide results which are adequate for supporting the RFI objectives and legally defensible in a court of law. The QAPP program will include:

i) A mechanism for ongoing control of measurements and evaluation of data quality; and

ii) A measure of data quality in terms of precision, accuracy, representativeness, completeness, and comparability.

The following section is a general discussion of the criteria used to measure the field and laboratory analytical data quality. The attached laboratory reference data and laboratory standard operating procedures (SOPs) provide the performance criteria for laboratory sample analytical data.

3.1 PRECISION

3.1.1 DEFINITION

Precision is defined as a quantitative measure of the degree to which two or more measurements are in agreement. Precision will be stated in terms of relative percent difference (RPD). The overall precision of measurement data is a mixture of sampling and analytical factors. Precision will be determined by collecting and analyzing field duplicate samples and by creating and analyzing laboratory duplicates from one or more of the field samples. The analytical results from the field duplicate samples will provide data on sampling precision. The results from the laboratory created duplicate samples will provide data on analytical precision.

3.1.2 FIELD PRECISION SAMPLE OBJECTIVES

Field precision will be assessed through collection and measurement of field duplicates at a rate of 1 duplicate per 10 investigative samples.

3.1.3 LABORATORY PRECISION SAMPLE OBJECTIVES

Laboratory duplicate analyses will be performed through the use of matrix spike/matrix spike duplicates (MS/MSD) analyses. The analytical data will be presented in summary table format. The precision criteria for laboratory MS/MSD analyses are provided in the laboratory reference data provided in Attachment 3.

3.2 ACCURACY

3.2.1 DEFINITION

Accuracy relates to the bias in a measurement system. Bias is the difference between the observed and the "true" value. Sources of error are the sampling process, field contamination, preservation techniques, sample handling, sample matrix, sample preparation and analytical procedure limitations.

3.2.2 FIELD ACCURACY OBJECTIVES

Sampling bias will be assessed by evaluating the results of field and trip blanks. Field blank samples as equipment rinsate blanks and trip blanks will be collected as appropriate for each sampling effort.

Equipment rinsate blanks will be collected by passing laboratory purified water over and/or through the respective field equipment utilized during each sampling effort if appropriate. One equipment rinsate blank will be collected every 20 investigative samples. However, at a minimum, one equipment rinsate blank will be collected per day for each type of non-dedicated field equipment used for the sampling effort. Equipment rinsate blanks will be analyzed for each target parameter for the respective sampling effort for which

environmental media have been collected. (Note: If dedicated or disposable sampling equipment is used, equipment rinsate blank samples may not be collected as part of that field effort.)

Trip blank samples will be prepared by the laboratory and provided with each cooler that includes aqueous volatile organic compound (VOC) analysis containers. Trip blank samples will be analyzed for each aqueous VOC for which environmental media have been collected for analysis.

3.2.3 LABORATORY ACCURACY SAMPLE OBJECTIVES

Analytical bias will be assessed through the use of known laboratory control samples and site-specific matrix spike sample analyses. Laboratory control samples (LCS) and MS/MSD sample analysis will be performed as prescribed by the analytical method SOPs. LCS analyses will be performed with each analytical batch of project samples to determine the accuracy of the analytical system.

One (1) set of MS/MSD analyses will be performed with each batch of twenty (20) project samples to assess the accuracy of identification and quantification of analytes within the site-specific sample matrices. Additional sample volume will be collected at sample locations selected for MS/MSD analyses so that MDLs and EQLs can be met.

The results of the LCS and MS/MSD analyses will be presented in a summary table reporting format and evaluated against the acceptance criteria presented in the laboratory reference data provided in Attachment 3.

The accuracy of organic parameter analyses is also monitored through the analysis of system monitoring or surrogate compounds. Surrogate compounds are added to each sample, standard, blank, and QC samples prior to the sample preparation and analysis. Surrogate compound percent recoveries provide information on the effect of the sample matrix on the accuracy of the analyses and are evaluated against the acceptance criteria presented in the attached laboratory reference data and/or associated analytical method SOPs.

3.3 REPRESENTATIVENESS

3.3.1 DEFINITION

Representativeness expresses the degree to which sample data accurately and precisely represents a characteristic of a population, a parameter variation at a sampling point, or an environmental condition. Representativeness is a qualitative parameter that is dependent upon the design of the sampling program. The representativeness criterion is satisfied by proper selection of sampling locations and quantity of samples collected.

3.3.2 MEASURES TO ENSURE REPRESENTATIVENESS OF FIELD DATA

Representativeness will be addressed by describing sampling techniques and the rationale used to select sampling locations. Sampling locations may be biased (based on existing data, instrument surveys, observations, etc.) or unbiased (completely random or stratified-random approaches).

For this project, sampling will be biased; that is, sampling associated with the soil, concrete, sediment, surface water, and groundwater will be based on the observed presence/absence of site specific contaminants, and/or site knowledge. Specific sampling technique descriptions, which allow consistency, repetitiveness and thus representativeness, are provided as SOPs in the Field Sampling Plan (FSP).

3.3.3 MEASURES TO ENSURE REPRESENTATIVENESS OF LABORATORY DATA

Representativeness in the laboratory is ensured by using proper analytical procedures, and analyzing field duplicate samples. By definition, field duplicate samples are collected to be representative of a given point in space and time. Thus, sample duplicates provide both precision and representativeness information.

3.4 COMPLETENESS

3.4.1 DEFINITION

Completeness is a measure of the amount of valid (usable) data obtained from a measuring system compared to the amount that was expected to be obtained under normal conditions. The completeness goal for all data uses is that a sufficient amount of valid data be generated so that determinations can be made related to the intended data use with a high degree of confidence.

3.4.2 FIELD COMPLETENESS OBJECTIVES

Completeness is a measure of the amount of valid measurements obtained from all measurements taken in this project. Field completeness objective for this project will be 90 percent.

3.4.3 LABORATORY COMPLETENESS OBJECTIVES

Laboratory data completeness is a measure of the amount of valid data obtained from all laboratory measurements. The evaluation of the data completeness will be performed at the conclusion of each sampling and analysis effort. Corrective actions such as revised sample handling procedures will be implemented if problems are noted.

The completeness of the data generated will be determined by comparing the amount of valid data, based on independent validation, with the total data set. The completeness objective will be 90 percent.

3.5 COMPARABILITY

3.5.1 DEFINITION

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another.

3.5.2 MEASURES TO ENSURE COMPARABILITY OF FIELD DATA

Sample data should be comparable with other measurement data for similar samples and sample conditions. This goal is achieved through using standard operating procedures to collect, preserve, store, and analyze representative samples and the reporting of analytical results. The field SOPs for the various activities to be conducted during this investigation provide guidelines to generate reproducible results.

3.5.3 MEASURES TO ENSURE COMPARABILITY OF LABORATORY DATA

Comparability of laboratory data will also be measured with the results from the analysis of Standard Reference Materials (SRM) obtained from either EPA Cooperative Research and Development Agreement (CRADA) suppliers or the National Institute of Standards and Technology (NIST) for instrument initial and continuing calibration verification. The reported analytical data will be presented in standard units of mass of contaminant within a known volume of environmental media.

i) Solid Matrices - micrograms (μg) contaminant per kilogram (kg) for organic analyses, and milligrams (mg) per kilogram (kg) for inorganic analyses of media (Dry Weight);
ii) Aqueous Matrices - micrograms (μg) contaminant per liter (L) of media for organic analyses, and milligrams (mg) per liter (L) for inorganic analyses; and
iii) Non-Aqueous Phase Liquids - (NAPL) – milligrams (mg) contaminant per kilogram (kg) of media.

Additional guidance on analytical data reporting is contained in the laboratory SOPs.

3.6 DECISION RULES

3.6.1 DEFINITION

The decision rule is a statement that prescribes a course of action or non-action to be taken, based on assumptions to test its logical and empirical consequences.

3.6.2 DECISION RULE OBJECTIVE

The rationale for sample locations, sample number, and analytical parameters is provided in the main text of the RFI Work Plan. The decision rule for the sampling and analysis data collected is provided in the RFI Work Plan and supplemental as follows:

1. Define statistical parameters characterizing the population (e.g., mean, maximum, and percentile) and incorporate the scale of decision making (e.g. AOI size). Samples will be collected discreetly and biased towards locations with the highest likelihood to encounter maximum contaminant levels to provide an indication of impacted zones.

2. Identify action levels (e.g., Soil Screening Levels; Risk-based Screening Levels; Preliminary Remediation Goals (PRGs)); or a reference-based standard based on a Site Conceptual Model that will be developed as part of the RFI process.

3. The maximum contaminant concentration or the 95th percent Upper Confidence Limit of the valid data set for each parameter detected within an AOI will be used to conduct a preliminary baseline human health and ecological risk assessment for the determination of appropriate corrective actions.

3.7 LEVEL OF QUALITY CONTROL EFFORT

Equipment rinsate blank, trip, and method blanks samples, field duplicate samples, laboratory control and matrix spike samples will be prepared and analyzed to determine the data quality provided by the sampling and analysis activities conducted during the execution of the RFI Work Plan.

Equipment rinsate blanks will be prepared by field personnel and submitted for analysis of target parameters. Equipment rinsate blanks will provide the means to assess the quality of data resulting from the field program. Equipment rinsate blank samples will be analyzed to check for contamination of equipment introduced during sampling at the Facility. One equipment rinsate blank sample will be collected for every 20 investigative samples. However, a minimum of one will be collected each day for each type of equipment where non-dedicated or disposal equipment is utilized.

Trip blanks are used to assess the potential for contamination during sample storage and shipment. The trip blank consists of ASTM Type II water that has been purged with an inert gas by the laboratory and provided with the sample containers to be used when sampling for VOC. Trip blanks will be preserved and handled in the same manner as the investigation samples. One trip blank will be included along with each shipment cooler containing project samples to be analyzed for VOC.

Method blank samples will be prepared by the laboratory and analyzed concurrently with all project samples to assess potential contamination introduced during the analytical process.

Field duplicate samples are analyzed to check for sampling and analytical reproducibility. The general level of the QC effort will be one field duplicate will be collected for at least one in every 10 or fewer investigative samples.

Matrix spikes will provide information to assess the precision and accuracy of the analysis of the target parameters within the environmental media collected at the Facility. Matrix spikes will be performed in duplicate. One MS/MSD will be collected for every 20 or fewer investigative samples per sample matrix (i.e. soil, concrete, sediment, and groundwater). Soil MS/MSD samples require a triple sample volume for VOC only. Aqueous MS/MSD samples require triple the normal sample volume for VOC analysis and double the volume for the remaining parameters.

4.0 SAMPLING PROCEDURES

Samples of groundwater, surface water, soil, concrete, and sediment will be obtained during the RFI program. The sampling procedures for the RFI will be consistent for the objectives of the project. The FSP describes each of the sampling tasks and objectives.

Refer to the appropriate sections of the FSP for the field investigation standard operating procedures.

4.1 SAMPLE CONTAINERS

Sample containers for each sampling task will be provided by the project laboratory. The containers will be cleaned by the manufacturer to meet or exceed the analyte specifications established in the U.S. EPA, "Specifications and Guidance for Obtaining Contaminant-Free Sample Containers", April 1992, OSWER Directive #9240.0-0.5A. Certificates of analysis for each lot of sample containers used during the RFI will be maintained by the laboratory and will be available upon request.

The appropriate sample containers, preservation method, maximum holding times, and shipping information for each target parameter and sampling task are provided in Table 4.1.

4.2 SAMPLE LABELING

Each sample will be labeled with a unique sample number that will facilitate tracking and cross-referencing of sample information. Equipment rinsate blank and field duplicate samples also will be numbered with a unique sample number to prevent analytical bias of field QC samples.

Each sample will be labeled with a unique sample number that will facilitate tracking and cross-referencing of sample information. The sample numbering system to be used is described as follows:

An example of the sample numbering system follows:

Example: GW-MMDDYY-XX-001

GW	= GW-groundwater, SW-surface water, S–soil, SD-sediment, W-surface wipe
MMDDYY	= Date in month/day/year
XX	= Sampler's first and last initials
001	= Sequential number for event

Equipment rinsate blank and field duplicate samples also will be numbered with a unique sample number, consistent with the numbering system described above, to prevent laboratory bias of field QC samples.

Additionally, each sample container will have a sample tag affixed to its neck. The tag will provide information concerning the requested analysis and sample preservation in addition to the information provided on the sample label.

4.3 FIELD QC SAMPLE COLLECTION

4.3.1 EQUIPMENT RINSATE BLANK SAMPLE COLLECTION

Equipment rinsate blank samples will be collected when non-dedicated or disposable sampling equipment is used to collect samples. Equipment rinsate blanks consist of laboratory purified water that has been routed through decontaminated sampling equipment and collected into the appropriate containers. The containers will be filled in order of decreasing analyte volatility (i.e., VOC first, SVOC second, which are followed by the containers for the remaining analyses).

4.3.2 FIELD DUPLICATE SAMPLE COLLECTION

4.3.2.1 WATER SAMPLES

Equipment rinsate duplicate samples will be collected concurrently with the investigative sample alternating the filling of each sample container identified in Table 4.1 using the procedures identified in the FSP.

4.3.2.2 SOIL/SEDIMENT SAMPLES

Soil and sediment field duplicates will be collected as specified in the following:

1. The split-spoon sampler or trowel is retrieved from the sampling point.

2. Soil for VOC analysis is removed from the sampling device utilizing a specialized zero headspace sampling tool (EnCore Sampler™) as specified in the FSP. Sediment samples will typically require collection in a four-ounce jar due to moisture content.

3. Soil and sediment samples for non-VOC analysis is removed from the sampling device and placed in a stainless steel mixing bowl. The soil is thoroughly homogenized using stainless steel utensils and the sample containers are filled in order of decreasing analyte volatility as described in Section 4.3.1.

4.3.3 MS/MSD SAMPLE COLLECTION

MS/MSD sample collection for aqueous samples requires triple the sample volume for VOC analysis and double the volume for all remaining parameters. The sampling procedure specified in Section 4.3.2.1 is used to collect aqueous samples. Soil MS/MSD samples require triple volume for VOC analyses. No additional volume is required for remaining soil parameters. Section 4.3.2.2 describes the sampling procedures for soil sample collection.

5.0 CUSTODY PROCEDURES

Custody is one of several factors necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files. Final evidence files, including all originals of laboratory reports, are maintained under document control in a secure area.

Custody of a sample begins when it is collected by or transferred to an individual and ends when that individual relinquishes or disposes of the sample. A sample or evidence file is under your custody if:

1. the item is in actual possession of a person;

2. the item is in the view of the person after being in actual possession of the person;

3. the item was in actual possession but is stored to prevent tampering; or

4. the item is in a designated and identified secure area.

5.1 FIELD CUSTODY PROCEDURES

Field personnel will be required to keep written records of field activities on applicable preprinted field forms and in a bound field notebook. The logbooks provide the means of recording data collecting activities. These records will be written legibly in ink and will contain pertinent field data and observations. Entry errors or changes will be crossed out with a single line, dated and initialed by the person making the correction. Field forms and notebooks will be periodically reviewed by the Field Quality Assurance (QA) Officer. Each member of the field team will be assigned a logbook. Each logbook title page should include field team member's name, project name, project start date, project end date, and unique logbook number.

The beginning of each entry in the logbook will contain the following information:

i) Date;
ii) Start time;
iii) Weather;
iv) Names of field personnel (including subcontractors);
v) Level of personal protection used at the Facility; and
vi) Names of all visitors and the purpose of their visit,
vii) Relevant field forms documenting field collection data.

For each measurement and sample collected the following will be recorded:

i) Detailed description of sample point;
ii) Equipment used to collect sample or make measurement and the date equipment was last calibrated;
iii) Time sample was collected;
iv) Sample description;
v) Depth sample was collected;
vi) Volume and number of containers; and
vii) Sampler identification.

5.1.1 FIELD PROCEDURES

The data quality can be affected by sample collection activities. If the integrity of collected samples is questionable, the data, regardless of its analytical quality, will also be questionable. Field sampling SOPs will provide for the collection of representative samples.

The following procedure describes the process to maintain the integrity of the samples:

i) Upon collection samples are placed in the proper containers. In general, samples collected for organic analysis will be placed in pre-cleaned glass containers, and samples collected for inorganic analysis will be placed in pre-cleaned plastic (polyethylene) bottles.

ii) Samples will be assigned a unique sample number and will be affixed to a sample label. The information to be placed on the sample label will include the sample ID

number, the sample type, the sampler's name, date collected, preservation technique, and analytical parameter and method to be performed. Information on the labels will be completed with a ballpoint pen or indelible marker.

iii) Samples will be properly and appropriately preserved by field personnel in order to minimize loss of the constituent(s) of interest due to physical, chemical or biological mechanisms. Sample aliquots that require preservation (pH adjustment) will be checked with pH paper at the time of preservation. Confirmation of preservation will include capping of the preserved sample, inverting the sample three times, uncapping and confirming the sample pH. If required, additional preservative will be added and the procedure repeated until proper preservation is achieved. Samples for VOC analysis will be collected in pre-preserved containers and will not undergo this procedure.

iv) Appropriate volumes will be collected to ensure that the EQL can be successfully achieved and that the required QC sample analyses can be completed.

5.1.2 TRANSFER OF CUSTODY AND SHIPMENT PROCEDURES

i) A chain-of-custody (COC) record will be completed during sample collection and will accompany each shipment identifying the contents of the shipment. The COC record will accompany the samples to the laboratory. The field personnel collecting the samples will be responsible for the custody of the samples until the samples are relinquished to the laboratory. Sample transfer will require the individuals relinquishing and receiving the samples to sign, date and note the time of sample transfer on the COC record. As few people as possible should handle the samples.

ii) Samples will be shipped or delivered in a timely fashion to the laboratory so that holding-times and/or analysis times as prescribed by the methodology can be met.

iii) Samples will also be transported in containers (coolers) packaged with ice to maintain the temperature prescribed in Table 4.1. Samples will be packaged for shipment and shipped to the appropriate laboratory for analysis with a separate signed chain-of-custody record enclosed in each sample cooler. Water VOC samples will be placed in bubble wrap bags while soil VOC samples will be placed in sealed

sample pouches. The remaining samples in glass containers will be wrapped in bubble wrap and placed in the sample cooler. Samples in polyethylene containers will be placed upright directly in the sample cooler. All samples will be placed in an upright position and limited to one layer of samples per each cooler. Additional bubble wrap or packaging material will be added to fill the cooler. Shipping containers will be secured with strapping tape and custody tape for shipment to the laboratory.

iv) When samples are split with the U.S. EPA and Facility representatives, a separate chain-of-custody will be prepared for the samples and marked to indicate to whom the samples are being split. The person relinquishing the samples to the U.S. EPA or the Facility will require the representatives signature acknowledging sample receipt.

v) If samples are sent by a commercial carrier, a bill of lading will be used. A copy of the bill of lading will be retained as part of permanent documentation. Commercial carriers are not required to sign the custody record as long as the custody record is sealed inside the sample cooler and the custody tape remains intact.

vi) Samples will be picked up by a laboratory courier or transported overnight by a courier to the laboratory the same day they are collected or within twenty-four hours unless collected on a weekend or holiday. In these cases, the samples will be stored in a secure location until delivery to the lab. Additional ice will be added to the cooler as needed to maintain proper preservation temperatures.

5.2 LABORATORY CHAIN-OF-CUSTODY PROCEDURES

A full-time sample custodian will be assigned the responsibility of sample control. It will be the responsibility of the sample custodian to receive all incoming samples. Once received, the custodian will document that the custody tape on the coolers is unbroken, that each sample is received in good condition (i.e., unbroken, cooled, etc.), that the associated paperwork, such as chain-of-custody forms have been completed and will sign the chain-of-custody forms. In special cases, the custodian will document from appropriate sub-samples that chain-of-custody with proper preservation has been accomplished. The custodian will also document that sufficient sample volume has been received to complete the analytical

program. The sample custodian will then place the samples into secure, limited access storage (refrigerated storage, if required). The sample custodian will assign a unique number to each incoming sample for use in the laboratory. The unique number will then be entered into the sample-receiving log. The laboratory date of receipt will also be noted. Sample tags are checked against sample labels and the chain of custody records and are then maintained in the laboratory's project file.

Consistent with the analyses requested on the chain-of-custody form, analyses by the laboratory's analysts will begin in accordance with the appropriate methodologies. Samples will be removed from secure storage only after internal chain-of-custody sign-out procedures have been followed.

5.3 STORAGE OF SAMPLES

Sample bottles, when the available volume has been consumed by the analysis, will be returned to secure and limited access storage. Upon completion of all laboratory analyses for each sample submittal, empty sample bottles will be disposed of by the sample custodian. The length of time that samples are held will be at least thirty (30) days after reports have been submitted. Disposal of remaining samples will be completed in compliance with all Federal, State, and local requirements.

Laboratory custody procedures and document control for those samples analyzed by the project laboratory will be carried out using the laboratory's standard operating procedures.

5.4 FINAL PROJECT FILES CUSTODY PROCEDURES

The final project file will be the central repository for all documents with information relevant to sampling and analysis activities as described in this QAPP. The Senior Project Engineer/Advisor will be the custodian of the project file. The project files for the RFI, including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports, and data reviews will be maintained in a secured, limited access area and under custody of the Senior Project Engineer/Advisor, or his designee.

The final project file will include:

i) Project plans and drawings;
ii) Field data records;
iii) Sample identification documents and soil boring/monitoring well logs;
iv) All chain-of-custody documentation;
v) Correspondence;
vi) References, literature;
vii) Laboratory data deliverables;
viii) Data validation and assessment reports;
ix) Progress reports, QA reports; and
x) Final report.

The laboratory will be responsible for maintaining analytical logbooks, laboratory data and sample chain of custody documents. Raw laboratory data files and copies of hard copy reports will be inventoried and maintained by the laboratory for a period of six (6) years at which time the laboratory will contact CRA regarding the disposition of the project related files.

6.0 CALIBRATION PROCEDURES AND FREQUENCY

This section describes procedures for maintaining the accuracy for all the instruments and measurement equipment, which will be used for conducting field tests and laboratory analyses. These instruments and equipment will be calibrated prior to each use or according to a periodic schedule.

6.1 FIELD INSTRUMENT CALIBRATION PROCEDURES

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated as specified in the field SOPs.

The field instruments are likely to include dissolved oxygen meters, pH meters, turbidity meters, specific conductance meters and PIDs. Field instruments will be used for real-time sample measurement during monitoring well sampling and organics screening for both on-site screening of soil samples and for health and safety air monitoring, as described in the HASP. On-site air monitoring for health and safety purposes and the screening of soil samples may be accomplished using PIDs.

Field instruments will be calibrated prior to use and the calibration will be verified after a maximum of 10 measurements. Specific information concerning calibration frequency, acceptance criteria, and the conditions that require more frequent calibration is contained in the field SOPs in Attachment 1.

Satisfactory completion of the pre-operation inspection will be noted on the Field Sampling Record, along with the results of each field measurement.

6.2 LABORATORY INSTRUMENT CALIBRATION PROCEDURES

Calibration procedures for a specific laboratory instrument will consist of initial calibration, initial calibration verification and continuing calibration verification. The SOPs in Attachment 2 present the specific calibration procedures for each method of analysis. The SOP for each analysis performed in the laboratory describes the calibration procedures, their frequency, acceptance criteria and the conditions that will require calibration. In all

cases, the initial calibration will be verified using an independently prepared calibration verification solution.

The use of materials of known purity and quality will be utilized for the analysis of environmental samples. The laboratory will carefully monitor the use of all laboratory materials including solutions, standards and reagents through well-documented procedures.

All solid chemicals and acids/bases used by the laboratory will be reagent grade or better. All gases will be high purity or better. All Standard Reference Materials (SRMs) or Performance Evaluation (PE) Materials will be obtained from approved vendors of the National Institute of Standards and Technology (formerly National Bureau of Standards), the U.S. EPA Environmental Monitoring Support Laboratories (EMSL), or reliable Cooperative Research and Development Agreement (CRADA) certified commercial sources.

All materials including standards or standard solutions will be dated upon receipt, and will be identified by material name, lot number, purity or concentration, supplier, receipt/preparation date, recipient/preparer's name, expiration date and all other pertinent information.

7.0 ANALYTICAL PROCEDURES

Analytical procedures to be utilized by STL North Canton, 4101 Shuffel Drive NW, North Canton, Ohio 44720 for analysis of environmental samples will be based on referenced U.S. EPA analytical protocols and project specific SOPs provided as an Attachment 2 to this QAPP.

7.1 FIELD ANALYTICAL PROCEDURES

Field analytical procedures include the measurement of pH/temperature, dissolved oxygen, turbidity and specific conductivity during sampling of groundwater, and the qualitative measurement of VOC during the collection of soil samples at the Facility. Specific procedures for measurement of these parameters are presented in the SOPs provided in Attachment 1 of this QAPP.

7.2 LABORATORY ANALYTICAL PROCEDURES

Laboratory preparation and analytical SOPs and methods upon which they are based are presented in Table 7.1. The referenced SOPs provided in Attachment 2 of this document are based on the U.S. EPA methodology requirements promulgated in the following guidance documents:

i) "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, U.S.EPA Office of Solid Waste, 3rd Edition and Promulgated Updates, 1986; and

ii) "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, March 1983

The EQLs for the target analytes and compounds for the environmental media to be analyzed as part of the RFI Work Plan are presented in Table 1.2.

7.2.1 LIST OF PROJECT TARGET COMPOUNDS AND LABORATORY DETECTION LIMITS

A complete listing of project target compounds and project EQLs for each analyte group is listed in Table 1.2. MDLs have been experimentally determined by the project laboratory using the procedure identified in 40 CFR, Part 136 Appendix B. The laboratory's MDLs are provided in the laboratory reference data in Attachment 3.

7.2.2 LIST OF ASSOCIATED QC

The laboratory SOPs include a QC section that addresses the minimum QC requirements for the analysis of specific analyte groups. Section 8.0 of this QAPP references a listing of the associated QC samples for every analyte group and matrix.

8.0 INTERNAL QUALITY CONTROL CHECKS

This section presents the internal quality control checks that will be employed for field and laboratory measurements.

8.1 FIELD QUALITY CONTROL

QC procedures for pH/temperature, turbidity and conductivity of water samples and PID screening soil samples will include calibrating the instruments as described in Section 6.1 of this QAPP, measuring duplicate samples and checking the reproducibility of the measurements by taking multiple readings on a single sample or the measurement reference standards. The QC information for field equipment is stated in Section 3.0 of this QAPP. Assessment of field sampling precision and bias will be made by collecting field duplicates and equipment rinsate blanks for laboratory analysis. Collection of the samples will be in accordance with the applicable procedures in Section 4.3 of this QAPP.

8.1.1 EQUIPMENT RINSATE BLANK SAMPLES

Equipment rinsate blank samples will be collected in the field at a frequency of one for every 20 investigative sample. However, at a minimum, one will be collected each day for each type of sampling equipment. Equipment rinsate blanks will be used to assess the efficiency of sampling equipment decontamination. The presence of analytes in the equipment rinsate blank samples will be indicative of potential cross contamination.

8.1.2 TRIP BLANK SAMPLES

Trip blank samples, supplied by the laboratory with the sample containers, will be submitted with each shipping cooler containing samples to be analyzed for VOC . Trip blank samples are used to assess whether contamination of VOC samples has occurred due to contamination migration during sample shipment and storage.

8.1.3 FIELD DUPLICATE SAMPLES

Field duplicate samples are collected in a similar fashion to investigative samples at a minimum frequency of one duplicate per at least one in every 10 or fewer investigative samples by matrix at a minimum, one per sampling event. Field duplicate samples are analyzed by the laboratory to evaluate matrix, sample and analytical reproducibility.

8.2 LABORATORY QUALITY CONTROL CHECKS

The laboratories identified in Section 7.0 of this QAPP have QC programs they use to ensure the reliability and validity of the analysis performed at the laboratory. All analytical procedures are documented in writing as SOPs and each SOP includes a QC section, which addresses the minimum QC requirements for the procedure. The internal quality control checks may vary slightly for each individual procedure but in general will include the following QC requirements:

iii) Calibration Standards;
iv) Instrument Performance Checks – Organics;
v) Initial and Continuing Calibration Checks;
vi) Internal Standard Performance;
vii) Method Blank Samples;
viii) Laboratory Control Samples and QC Check Samples;
ix) Matrix Spike/Matrix Spike Duplicates;
x) Surrogates;
xi) ICP Interference Check Samples (ICS);
xii) ICP Serial Dilution;
xiii) ICP and ICP/MS QC Analyses;
xiv) Reagent Checks; and
xv) Blind Check Samples.

All data will be properly recorded. The data package will include a summary of QC data as presented in Section 9.3.2. Any samples analyzed in nonconformance with QC criteria will be re-analyzed by the laboratory, if sufficient volume is available.

9.0 DATA REDUCTION, VALIDATION AND REPORTING

All data generated through in field activities or by the laboratory operation shall be reduced and validated prior to reporting in accordance with the following procedures:

9.1 DATA REDUCTION

9.1.1 FIELD DATA REDUCTION PROCEDURES

Field data reduction procedures will be minimal in scope compared to those implemented in the laboratory setting. Only direct read instrumentation will be employed in the field. The pH, conductivity, temperature, turbidity and HNU readings collected in the field will be generated from direct read instruments following calibration per manufacturer's recommendations. Such data will be written into field logbooks immediately after measurements are taken and/or recorded on field forms. If errors are made, results will be legibly crossed out, initialed and dated by the field member, and corrected in a space adjacent to the original entry. Later, when the results forms required for this study are being filled out, the Field QA Officer will proof the forms to determine whether any transcription errors have been made by the field crew.

9.1.2 LABORATORY DATA REDUCTION PROCEDURES

For this project, the equations that will be employed in reducing data are found in the appropriate chapters of SW-846, Third Edition. Two of these equations, expressing analytical accuracy and precision, are presented in Section 12 of this QAPP. Such formulae make pertinent allowances for matrix type. All calculations are checked at the conclusion of each operating day. Errors are noted; corrections are made, but the original notations are crossed out legibly. Analytical results for soil samples shall be calculated and reported on a dry weight basis.

Quality control data (e.g., laboratory duplicates, surrogates, matrix spikes, and matrix spike duplicates) will be compared to the method acceptance criteria. Data considered to be acceptable will be entered into the laboratory computer system. Data summaries will be sent to the Laboratory QA Officer for review. If approved, data are logged into the project

database format. Unacceptable data shall be appropriately qualified in the project report. Case narratives will be prepared which will include information concerning data that fell outside acceptance limits and any other anomalous conditions encountered during sample analysis.

9.2 DATA VALIDATION

Data validation procedures shall be performed for both field and laboratory operations as described below:

9.2.1 PROCEDURES USED TO EVALUATE FIELD DATA

Procedures to evaluate field data for this project will include review of field logbooks, field forms and checking for transcription errors. This task will be the responsibility of the Field QA Officer.

9.2.2 PROCEDURES TO VALIDATE LABORATORY DATA

Validation of the analytical data will be performed by the CRA QA Officer or designee using the following documents as guidance for the review process:

"U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review", EPA-540/R-99/008, October 1999 and the "U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review ", EPA-540/R-94-013, February 1994.

The specific data qualifiers used will be as presented and defined in the CLP National Functional Guidelines. Validation will be performed, by qualified chemists at the direction of the CRA QA Officer. Data review and validation will consist of two tiers of assessment that incorporates an approach similar to "Innovative Approaches to Data Validation", U.S. EPA Region III, June 1995.

Tier I data validation will be performed on 100% of the laboratory quality control summary data deliverables and will consist of the following components:

Organic Analysis

i) technical holding times;
ii) GC/MS instrument performance check;
iii) initial and continuing calibration;
iv) internal standard performance
v) method, trip and equipment rinsate blanks;
vi) system monitoring compounds (surrogate spikes);
vii) MS/MSD results;
viii) laboratory control samples; and
ix) field duplicate samples.

Inorganic Analysis

i) technical holding times;
ii) initial calibration;
iii) initial and continuing calibration verification;
iv) calibration and method blanks;
v) ICP interference check samples;
vi) ICP/MS internal standards;
vii) ICP serial dilutions;
viii) laboratory control samples;
ix) MS/MSD results; and
x) field duplicate.

Tier II validation will be completed on 10 percent of the expanded deliverables during the initial investigation activities. The Tier II validation will include the evaluation of all raw data and mass spectra to validate the above components as well as verify sample results.

The completeness of each data package will be evaluated by the Data Validator. Completeness checks will be administered on all data to determine whether deliverables specified in the QAPP are present. At a minimum, deliverables will include sample chain-of-custody forms, analytical results, QC summaries and supporting raw data from instrument printouts. The review will determine whether all required items are present and request copies of missing deliverables.

9.3 DATA REPORTING

Data reporting procedures shall be carried out for field and laboratory operations as indicated below:

9.3.1 FIELD DATA REPORTING

Field data reporting shall be conducted principally through the transmission of report sheets containing tabulated results of all measurements made in the field and documentation of all field calibration activities.

9.3.2 LABORATORY DATA REPORTING

The laboratory data reporting package will be sufficient to perform a data validation in accordance with protocols described in Section 2.3. The task of reporting laboratory data by GM will be initiated after the completion of data validation. The laboratory Project Manager will perform a final review of the QC data summary packages and case narratives to determine whether the report meets the project requirements. In addition to the chain-of-custody record, the final laboratory data report format shall consist of the following.

1. Case Narrative

   i) date of issuance;
   ii) laboratory analysis performed;
   iii) any deviations from intended analytical strategy;
   iv) laboratory batch number;
   v) number of samples and respective matrices;
   vi) cross-reference table of laboratory sample to project sample identification numbers;
   vii) definition of data qualifiers to be used;
   viii) methods of sample preparation and analyses for samples;

ix) QC procedures utilized and also references to the acceptance criteria;
x) laboratory report contents;
xi) project name and number;
xii) condition of samples "as received";
xiii) discussion of whether or not sample holding times were met;
xiiii) discussion of technical problems or other observations which may have created analytical difficulties;
xv) discussion of laboratory QC checks which failed to meet project criteria; and
xvi) signature of the Laboratory QA Officer or his designee.

2. Chemistry Data Package

i) sample results with quantitation (report) limits (RL), reporting MDLs and estimated values between the RL and MDL;
ii) raw data for sample results and laboratory quality control samples;
iii) results of (dated) initial and continuing calibration checks, GC/MS tuning results;
iv) internal standard response and retention time summaries;
v) matrix spike and matrix spike duplicate recoveries, laboratory control samples, method blank results, surrogate recoveries, calibration check compounds and system performance check compound results (organics);
vi) matrix spike recoveries and matrix duplicate relative percent differences, laboratory control sample, serial dilutions, method blank results, and reagent blank results (inorganics);
vii) labeled and dated chromatograms, quantitation reports and mass spectra of sample results and laboratory quality control checks; and
viii) all sample preparation forms and other related instrumental data.

Note: Only the TCL parameter list identified in Table 1.2 shall be included in the laboratory chromatographic quantitation reports.

10.0 PERFORMANCE AND SYSTEM AUDITS

A performance audit is an independently obtained quantitative comparison with data routinely obtained in the field or the laboratory. Performance audits include two separate, independent parts: internal and external audits.

10.1 FIELD PERFORMANCE AND SYSTEM AUDITS

10.1.1 INTERNAL FIELD AUDIT RESPONSIBILITIES

Internal audits of field activities include the review of sampling and field measurements conducted by the Field QA Officer. The audits will verify that all procedures are being followed. Internal field audits will be conducted once during each phase of the sampling and at the conclusion of the project. The audits will include examination of the following:

i) Field sampling records, screening results, instrument operating records
ii) Sample collection
iii) Handling and packaging in compliance with procedures
iv) Maintenance of QA procedures; and
v) Chain-of-custody reports.

Follow up audits will be conducted to correct deficiencies and to verify that procedures are maintained throughout the investigation. The field audit checklist is included in Attachment 4.

10.1.2 EXTERNAL FIELD AUDIT RESPONSIBILITIES

External audits may be conducted by the U.S. EPA Project Coordinator, or designee at any time during the field operations. These audits may or may not be announced and are at the discretion of the U.S. EPA Region 5. The external field audits can include (but are not limited to) the following:

i) Sampling equipment decontamination procedures;
ii) Sample bottle preparation procedures;

iii) Sampling procedures;
iv) Examination of health and safety plans;
v) Procedures for verification of field duplicates; and
vi) Field screening practices.

10.2 LABORATORY PERFORMANCE AND SYSTEM AUDITS

10.2.1 INTERNAL LABORATORY AUDIT RESPONSIBILITIES

The laboratory system audits will be conducted by the CRAQA Officer or designee and performed on an annual basis. Laboratory performance audits will be conducted prior to field activities or on an annual basis, depending on the duration of the project.

The system audit will include an examination of laboratory documentation including: sample receiving logs, sample storage, chain-of-custody procedures, sample preparation and analysis, and instrument operating records. System audits are qualitative and consist of an on-site review of a laboratory's quality assurance system and physical facilities for calibration and measurement.

The performance audits after project startup will include submittal of blind performance evaluation samples along with project samples. The CRA QA Officer will evaluate the analytical results to ensure the lab is maintaining acceptable QC performance.

The audit checklist to be used for laboratory systems is provided in Attachment 5.

At the conclusion of internal or external system audits, reports are provided to the laboratory's operating divisions for appropriate comment and remedial/corrective action where necessary. Written response to internal as well as external audits will be required. Records of audits and corrective actions are maintained by the Laboratory QA Officer.

10.2.2 EXTERNAL LABORATORY AUDIT RESPONSIBILITIES

External audits will be conducted as required, by appropriate QA personnel of the U.S. EPA Region 5 and may be conducted at least once prior to sampling and analysis activities.

External audits may include any of the following:

i) Review of laboratory analytical procedures;
ii) Laboratory on-site visits; and
iii) Submission of performance evaluation samples for analysis.

Failure of any of the above audit procedures can lead to laboratory disqualification, and another suitable laboratory will have to be chosen. An on-site review can consist of:

i) Sample receipt procedures;
ii) Custody, sample security, and log-in procedures;
iii) Review of instrument calibration logs;
iv) Review of QA procedures;
v) Review of log books;
vi) Review of analytical SOPs; and
vii) Personnel interviews.

A review of a data package from samples recently analyzed by the laboratory can include (but not be limited to) the following:

i) Comparison of resulting data to the SOP or method
ii) Verification of initial and continuing calibrations within control limits
iii) Verification of surrogate recoveries and instrument timing results
iv) Review of extended quantitation reports for comparisons of library spectra to instrument spectra, where applicable
v) Assurance that samples are run within holding times.

11.0 PREVENTATIVE MAINTENANCE

11.1 FIELD INSTRUMENT PREVENTATIVE MAINTENANCE

The field equipment preventative maintenance program ensures the effective completion of the sampling effort and is designed to minimize equipment down time. Program implementation is concentrated in three areas:

i) Maintenance responsibilities.
ii) Maintenance schedules.
iii) Inventory of critical spare parts and equipment.

The maintenance responsibilities for field equipment will be assigned to the task leaders in charge of specific field operations. Field personnel will be responsible for daily field checks and calibrations and for reporting any problems with the equipment. The maintenance schedule will follow the manufacturer's recommendations. In addition, the field personnel will be responsible for determining that critical spare parts are included with the field equipment. An adequate inventory of spare parts will be maintained. The inventory will primarily contain parts that are subject to frequent failure, have limited useful lifetimes and/or cannot be obtained in a timely manner.

11.2 LABORATORY INSTRUMENT PREVENTATIVE MAINTENANCE

Analytical instruments at the laboratory will undergo routine and/or preventative maintenance. The extent of the preventative maintenance will be a function of the complexity of the equipment.

Generally, annual preventative maintenance service will involve cleaning, adjusting, inspecting and testing procedures designed to deduce instrument failure and/or extend useful instrument life. Between visits, routine operator maintenance and cleaning will be performed according to manufacturer's specifications by laboratory personnel.

Table 11.1 provides the frequency which compounds of key analytical instruments or equipment will be serviced.

Maintenance records will be placed on file at the laboratory and can be made available upon request.

12.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

The following sections include the procedures and formulae utilized to assess the levels of precision, accuracy and completeness achieved during the associated sample analyses.

12.1 FIELD MEASUREMENTS

Field generated information such as pH, specific conductance, temperature, and dissolved oxygen data will be reviewed for validity. The review will be performed by the Field QA Officer and typically include bound logbooks/forms, data entry and calculation checks. Field data will be assessed by the CRA Field QA Officer who will review the field results for compliance with the established QC criteria that are specified in Section 3.0 of this QAPP. The accuracy of pH and specific conductance will be assessed using daily instrument calibration, calibration check, and blank data. Accuracy will be measured by determining the percent recovery of calibration check standards as defined in Section 12.2.2. Precision of the pH and specific conductance measurements will be assessed on the basis of the reproducibility of duplicate readings of a single sample and will be measured by determining the relative percent difference of the readings as defined in Section 12.2.1. Accuracy and precision of the soil VOC screening will be determined using duplicate readings of calibration checks. Field data completeness will be calculated using the following equation:

$$\text{Completeness} = \frac{\text{Valid (usable) Data Obtained}}{\text{Total Data Planned}} \times 100$$

12.2 LABORATORY DATA

Laboratory results will be assessed for compliance with required precision, accuracy and completeness detailed in the following subsections:

12.2.1 PRECISION

The precision of laboratory analysis will be assessed by comparing the analytical results between MS/MSD analyses. The relative percent difference (RPD) will be calculated for each pair of duplicate analyses (laboratory MS/MSDs and field duplicates).

12.2.2 ACCURACY

The accuracy of laboratory results will be assessed for compliance with the established QC criteria that are described in Sections 3.0 and 8.0 of the QAPP using the analytical results of method blanks, reagent/preparation blank, SRMs or QC check samples and MS/MSD samples. The percent recovery (%R) of matrix spike samples and SRMs will be calculated.

12.2.3 COMPLETENESS

Completeness will be assessed by comparing the number of valid (usable) results (as determined by the CRA QA Officer) to the total possible number of results using the formula presented in Section 12.1.

12.3 STATISTICAL EVALUATIONS

In the examination of data and determination of their precision and accuracy, standard statistical formulae will be used. Further details are provided in the following subsections

12.3.1 PERCENT RECOVERY

The percent recovery of a parameter is calculated by dividing the amount recovered by the true amount added and multiplying by 100. The percent recoveries of spiked samples are evaluated to establish the analytical accuracy of a measurement. Percent recovery is calculated using the following formula:

$$\%R = \frac{SSR - SR}{SA} \times 100$$

where:

SSR = Spiked Sample Result
SR = Sample Result or Background
SA = Spike Added

12.3.2 RELATIVE PERCENT DIFFERENCE

The relative percent difference (RPD) is calculated by dividing the absolute value of the difference between two numbers by their arithmetic mean and multiplying by 100. The RPD is used to evaluate the analytical precision of two replicate measurements (e.g., matrix spike/matrix spike duplicate). RPD is calculated using the following formula:

$$RPD = \frac{(R_1 - R_2)}{R_1 + R_2} \times 100$$

where:

R_1 = value of first result
R_2 = value of second result

13.0 CORRECTIVE ACTION

13.1 FIELD CORRECTIVE ACTION

Corrective action is intended to address problems that arise by identification, recommendation, approval, and implementation of measures that counter unacceptable procedures or deficient quality control performance. The Field QA Officer and CRA QA Officer will be responsible for ensuring the quality of the sampling procedures and environmental data and as such, will be responsible for initiating corrective action when appropriate.

The corrective action procedures will be as follows:

i) Identify/define the problem;
ii) Assign responsibility for investigating the problem;
iii) Investigate/determine the cause of the problem;
iv) Determine an appropriate corrective action to eliminate the problem;
v) Implement the corrective action;
vi) Evaluate the effectiveness of the corrective action;
vii) Verify that the corrective action has eliminated the problem;
viii) Prepare a written record detailing the problem, corrective action utilized, and solution of the problem; and
ix) Submit the Corrective Action Record (CAR) to the Task Coordinator who initiated the corrective action and the Project QA Officer, and Project Manager.

The above procedures may be implemented through the use of the Systems Audit as described previously. Any Field Team member of the project may initiate corrective action procedures by reporting in writing the nature of the suspected problem to the Senior Project Engineer/Advisor, Project Manager or CRA QA Officer. The CRA Project Manager will begin corrective action by relating the problem to appropriate personnel.

13.2 LABORATORY CORRECTIVE ACTION

The following paragraphs define the corrective action decision process relative to possible non-compliant events encountered during laboratory analysis of the project samples.

Corrective actions will be initiated by the laboratory QA personnel and will be implemented by laboratory staff chemists under the oversight of the laboratory QA personnel. As with field corrective actions, the laboratory QA personnel will document the problem, the corrective action undertaken and the resolution of the problem. The corrective actions will be performed prior to release of the data from the laboratory.

Documentation will be provided to the laboratory QA director, CRA QA Officer, CRA Project Manager and GM Project Manager.

13.3 CORRECTIVE ACTION DURING DATA VALIDATION AND DATA ASSESSMENT

The CRA QA Officer may identify the need for corrective action during either the data validation or data assessment processes. Potential types of corrective action may include resampling by the field team or re-injection/reanalysis of samples by the laboratory (if possible).

These actions are dependent upon the ability to mobilize the field team, whether the data to be collected are necessary to meet the required quality assurance objectives (e.g., the holding time for samples is not exceeded). When the CRA QA Officer identifies a corrective action situation, GM's Project Manager will be responsible for approving the implementation of corrective action, including resampling, during data assessment. All corrective actions will be documented by the CRA Project Manager.

14.0 QUALITY ASSURANCE (QA) REPORTS

Critically important to the successful implementation of the QA Plan is a reporting system that provides the means by which the program can be reviewed, problems identified and programmatic changes made to improve the plan.

QA reports to management include:

i) Audit reports, internal and external audits with responses;

ii) Performance evaluation sample results; internal and external sources;

iii) Daily QA/QC exception reports/corrective actions.

QA/QC corrective action reports will be prepared by the CRA QA Officer when appropriate and presented to the project and/or laboratory management personnel so that performance criteria can be monitored for all analyses from each analytical department. The updated trend/QA charts prepared by the laboratory QA personnel will be distributed and reviewed by various levels of the laboratory management.

15.0 REFERENCES

1. United States Environmental Protection Agency, (1999). EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations. EPA QA/R-5 Interim Final, November 1999.

2 United States Environmental Protection Agency (1998). U.S. EPA RCRA QAPP Instructions. U.S. EPA Region 5, Revision, April 1998.

3 United States Environmental Protection Agency (1991). Preparation Aids for the Development of Category I Quality Assurance Project Plans. U.S. EPA/600/8-91/003, Risk Reduction Engineering Laboratory, Office of Research and Development, Cincinnati, Ohio, February 1991.

4. United States Environmental Protection Agency, (1993). Data Quality Objectives Process for Superfund, Interim Final Guidance. U.S. EPA/540/R-93-071, Office of Solid Waste and Emergency Response (OSWER), September 1993.

5. United States Environmental Protection Agency, (1992). Specifications and Guidance for Contaminant-Free Sample Containers. OSWER Directive 9240.0-05A, April 1992.

6. United States Environmental Protection Agency. U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review. U.S. EPA 540/R-99-012.

7. United States Environmental Protection Agency. U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review. U.S. EPA 540/R-94-013.

8. United States Environmental Protection Agency. Test Methods for Evaluating Solid Waste, Office of Solid Waste, U.S. EPA, SW-846, November 1986, with updates.

TABLE 1.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
GM POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Area of Interest (AOI)		*Sample*	*Field*	*Laboratory*	*Investigative*	*Quality Control*	
Number	*Description*	*Matrix*	*Parameters*	*Parameters*	*Samples*	*Field Blanks* [2]	*Field Duplicate*
AOI 1	Former Railroad Operations and Minerals Processing Facility	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Chloride, Total and Amenable Cyanide	21	1/20	1/10
		Surface Water	pH, Temperature, DO, Specific Conductivity, Turbidity	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Chloride, Total and Amenable Cyanide	2	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Chloride, Total and Amenable Cyanide	TBD [7]	1/20	1/10
AOI 2	Waste Storage Area	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Total and Amenable Cyanide	12	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	TBD [7]	1/20	1/10
AOI 3	PCB Storage Areas	Surface Wipes	PID VOC Screen	TCL PCB	3	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL PCB[6]	TBD [7]	1/20	1/10
AOI 4	Former North Disposal Area	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Total and Amenable Cyanide	15	1/20	1/10

TABLE 1.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
GM POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Number	*Area of Interest (AOI) Description*	*Sample Matrix*	*Field Parameters*	*Laboratory Parameters*	*Investigative Samples*	*Quality Control*	
						Field Blanks [2]	*Field Duplicate*
AOI 4	Former North Disposal Area (con't'd)	Sediment	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Total and Amenable Cyanide	3	1/20	1/10
		Surface Water	pH, Temperature, DO, Specific Conductivity, Turbidity	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	3	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	TBD [7]	1/20	1/10
AOI 5	Former East Sand Disposal Area	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Total and Amenable Cyanide	12	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	TBD [7]	1/20	1/10
AOI 6	Former Sludge Disposal and Fire Training Area	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Total and Amenable Cyanide	21	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	TBD [7]	1/20	1/10
AOI 7	Former North Lagoon and Outfall 001	Soil, Bedrock	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Total and Amenable Cyanide	8 [9]	1/20	1/10

TABLE 1.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
GM POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

						Quality Control	
Number	*Area of Interest (AOI) Description*	*Sample Matrix*	*Field Parameters*	*Laboratory Parameters*	*Investigative Samples*	*Field Blanks*[2]	*Field Duplicate*
AOI 7	Former North Lagoon and Outfall 001 (cont'd)	Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	1[7]	1/20	1/10
AOI 8	Former South Lagoons and Outfall 002	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Total and Amenable Cyanide	7	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	1[7]	1/20	1/10
AOI 9	Service Tunnels	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Total and Amenable Cyanide	18	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	TBD[7]	1/20	1/10
AOI 10	Existing Stormwater Lagoon and Outfall 003	Surface Water	pH, Temperature, DO, Specific Conductivity, Turbidity	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	2	1/20	1/10
		Sediment	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Total and Amenable Cyanide	3	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	TBD[7]	1/20	1/10

TABLE 1.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
GM POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Number	*Area of Interest (AOI) Description*	*Sample Matrix*	*Field Parameters*	*Laboratory Parameters*	*Investigative Samples*	*Quality Control*	
						Field Blanks [2]	*Field Duplicate*
AOI 11	Above Ground Storage Tanks Mineral Fuel Release Diesel Oil Release	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Total and Amenable Cyanide	11	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	TBD [7]	1/20	1/10
AOI 12	Area Affected by the Reclaimed Hydraulic Fluid Release	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Total and Amenable Cyanide	2	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	TBD [7]	1/20	1/10
AOI 13	Underground Storage Tanks Oil House USTs Piston Yard USTs						
AOI 14	McBride Cows Disposal Area	Soil	PID VOC Screen	TCL PCB	2	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL PCB[6]	TBD [7]	1/20	1/10
AOI 15	Former Equipment Storage Area	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Total and Amenable Cyanide	6	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	TBD [7]	1/20	1/10

TABLE 1.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
GM POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Area of Interest (AOI)		*Sample*	*Field*	*Laboratory*	*Investigative*	*Quality Control*	
Number	*Description*	*Matrix*	*Parameters*	*Parameters*	*Samples*	*Field Blanks* [2]	*Field Duplicate*
AOI 17	Piston Building Oil Accumulations	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Total and Amenable Cyanide	6	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	TBD [7]	1/20	1/10
AOI 18	Area Affected by the Henry System Discharge	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Total and Amenable Cyanide	2	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	TBD [7]	1/20	1/10
AOI 19	Area Affected by the Paint and Thinner Spill	Soil	PID VOC Screen	TCL VOC, TCL PCB	3	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL PCB[6]	TBD [7]	1/20	1/10
AOI 20	Northern Portion of the Piston Building	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Total and Amenable Cyanide	1	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	TBD [7]	1/20	1/10

TABLE 1.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
GM POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Number	*Area of Interest (AOI) Description*	*Sample Matrix*	*Field Parameters*	*Laboratory Parameters*	*Investigative Samples*	*Quality Control*	
						Field Blanks [2]	*Field Duplicate*
AOI 21	Filled Ravine North of the Die Cast Building	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Total and Amenable Cyanide	12	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	TBD [7]	1/20	1/10
AOI 22	Tool Room Annex Dock Release	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Total and Amenable Cyanide	1	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	TBD [7]	1/20	1/10
AOI 23	Area Affected by the 1996 Wastewater Treatment Filter Cake Release	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5], Total and Amenable Cyanide	1	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	TBD [7]	1/20	1/10
AOI 24	Area Affected by the June 2000 Die Lube 5150 Release	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB	1	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB[6]	TBD [7]	1/20	1/10

TABLE 1.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
GM POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Area of Interest (AOI)		*Sample*	*Field*	*Laboratory*	*Investigative*	*Quality Control*	
Number	*Description*	*Matrix*	*Parameters*	*Parameters*	*Samples*	*Field Blanks*[2]	*Field Duplicate*
Site-Wide Hydrogeological Investigation[7]		Soil	PID VOC Screen	TAL Metals[5], Total and Amenable Cyanide, TOC, Grain Size and Hydraulic Conductivity	16	1/20	1/10
		Groundwater	pH, Temperature, DO, Specific Conductivity, Turbidity, ORP	TCL VOC, TCL SVOC, TCL PCB, TAL Metals[5,6], Total and Amenable Cyanide	18[7]	1/20	1/10
Background Soil Sampling[8]		Soil	PID VOC Screen	TAL Metals[5], Total and Amenable Cyanide, TOC, Grain Size	5	1/20	1/10

Notes:

[1] - A laboratory trip blank will be submitted with each shipping cooler containing samples for TCL VOC analysis.
Field quality control sample quantities will be based on the frequencies detailed above by matrix across all AOIs.

[2] - Field blank samples are equipment rinsate blank samples collected at the frequency detailed above or at a minimum of one per day per type of sampling equipment.
They may not be required if dedicated or disposable sampling equipment is used.

[3] - Matrix Spike / Matrix Spike Duplicate (MS / MSD) analyses are required at a frequency of one per group of twenty (20) or fewer investigative samples by matrix.

[4] - The total will vary depending on the quantity of QC samples collected for each AOI, while meeting the minimum frequencies on a project-wide basis..

[5] - The TAL Metals list is modified eliminating Calcium, Magnesium, Potassium and Sodium.

[6] - The TCL PCB and TAL metal surface water and groundwater samples will be collected as filtered and unfiltered samples for the determination of dissolved and total TCL PCB and TAL metals.

[7] - Based on the results of the Site-Wide Hydrogeological Investigation, AOI-specific monitoring wells may be installed.

[8] - An additional four locations (up to four samples each) are included under the Site-Wide Hydrogeological Investigation.

[9] - Includes bedrock samples.

TCL = Target Compound List
VOC = Volatile Organic Compounds
SVOC = Semi-volatile Organic Compounds
PCB = Polychlorinated Biphenyls
TAL = Target Analyte List
TOC = Total Organic Carbon
DO = Dissolved Oxygen
ORP = Oxidation / Reduction Potential

TABLE 1.2

RFI PARAMETER LIST
GM POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Compound	*Estimated[1] Quantitation Limits*	
	Water (µg/L)	*Soil/Sediment (µg/kg)*
Target Compound List (TCL) Volatile Organic Compounds (VOC)		
Acetone	10	20
Benzene	1	5
Bromodichloromethane	1	5
Bromoform	1	5
Bromomethane	1	10
2-Butanone	10	20
Carbon disulfide	1	5
Carbon tetrachloride	1	5
Chlorobenzene	1	5
Chloroethane	1	10
Chloroform	1	5
Chloromethane	1	10
Cyclohexane	1	10
Dibromochloromethane	1	5
1,2-Dibromo-3-chloropropane	2	10
1,2-Dibromoethane	1	5
1,2-Dichlorobenzene	1	5
1,3-Dichlorobenzene	1	5
1,4-Dichlorobenzene	1	5
Dichlorodifluoromethane	1	10
1,1-Dichloroethane	1	5
1,2-Dichloroethane	1	5
1,1-Dichloroethene	1	5
cis-1,2-Dichloroethene	0.5	2.5
trans-1,2-Dichloroethene	0.5	2.5
1,2-Dichloropropane	1	5
cis-1,3-Dichloropropene	1	5
trans-1,3-Dichloropropene	1	5
Ethylbenzene	1	5
2-Hexanone	10	20
Isopropylbenzene	1	5
Methyl acetate	10	10
Methylene chloride	1	5
Methylcyclohexane	1	10
4-Methyl-2-pentanone	10	20
Methyl tert-butyl ether	5	20

TABLE 1.2

RFI PARAMETER LIST
GM POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Compound	*Estimated[1] Quantitation Limits*	
	Water (µg/L)	*Soil/Sediment (µg/kg)*
TCL VOC (continued)		
1,2,4-Trichlorobenzene	1	5
1,1,1-Trichloroethane	1	5
1,1,2-Trichloroethane	1	5
Trichloroethene	1	5
Trichlorofluoromethane	1	10
1,1,2-Trichloro-1,2,2-trifluoroethane	1	5
Vinyl chloride	1	10
Xylenes (total)	1	5
TCL Semi-Volatile Organic Compounds (SVOC)		
Acenaphthene	10	330
Acenaphthylene	10	330
Acetophenone	10	330
Anthracene	10	330
Atrazine	10	330
Benzaldehyde	10	330
Benzo(a)anthracene	10	330
Benzo(a)pyrene	10	330
Benzo(b)fluoranthene	10	330
Benzo(g,h,i)perylene	10	330
Benzo(k)fluoranthene	10	330
1,1'-Biphenyl	10	330
4-Bromophenylphenyl ether	10	330
Butylbenzylphthalate	10	330
di-n-Butyphthalate	10	330
Caprolactam	10	330
Carbazole	10	330
4-Chloroaniline	10	330
bis(2-Chloroethoxy)methane	10	330
bis(2-Chloroethyl)ether	10	330
bis(2-Chloroisopropyl)ether	10	330
4-Chloro-3-methylphenol	10	330
2-Chloronaphthalene	10	330
2-Chlorophenol	10	330
4-Chlorophenyl phenyl ether	10	330
Chrysene	10	330
Dibenz(a,h)anthracene	10	330
Dibenzofuran	10	330
3,3'-Dichlorobenzidine	50	1,600

TABLE 1.2

RFI PARAMETER LIST
GM POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Compound	*Estimated[1] Quantitation Limits* Water (µg/L)	Soil/Sediment (µg/kg)
TCL Semi-Volatile Organic Compounds (cont'd)		
2,4-Dichorophenol	10	330
Diethylphthalate	10	330
2,4-Dimethylphenol	10	330
Dimethylphthalate	10	330
4,6-Dinitro-2-methylphenol	50	1,600
2,4-Dinitrophenol	50	1,600
2,4-Dinitrotoluene	10	330
2,6-Dinitrotoluene	10	330
bis(2-Ethylhexyl)phthalate	10	330
Fluoranthene	10	330
Fluorene	10	330
Hexachlorobenzene	10	330
Hexachlorobutadiene	10	330
Hexachlorocyclopentadiene	50	1,600
Hexachloroethane	10	330
Indeno(1,2,3-cd)pyrene	10	330
Isophorone	10	330
2-Methylnaphthalene	10	330
2-Methylphenol	10	330
4-Methylphenol	10	330
Naphthalene	10	330
2-Nitroaniline	50	1,600
3-Nitroaniline	50	1,600
4-Nitroaniline	50	1,600
Nitrobenzene	10	330
2-Nitrophenol	10	330
4-Nitrophenol	50	1,600
N-Nitroso-di-n-propylamine	10	330
N-Nitrosodiphenylamine (diphenylamine)	10	330
di-n-Octylphthalate	10	330
Pentachlorophenol	10	330
Phenanthrene	10	330
Phenol	10	330
Pyrene	10	330
2,4,5-Trichlorophenol	10	330
2,4,6-Trichlorophenol	10	330

TABLE 1.2

RFI PARAMETER LIST
GM POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Compound	*Estimated[1] Quantitation Limits* Water (µg/L)	Surface Wipe (µg/sample)	Soil/Sediment (µg/kg)
TCL Polychlorinated Biphenyls (PCB)			
Aroclor 1016	0.2	4	33
Aroclor 1221	0.4	4	33
Aroclor 1232	0.2	4	33
Aroclor 1242	0.2	4	33
Aroclor 1248	0.2	4	33
Aroclor 1254	0.2	4	33
Aroclor 1260	0.2	4	33
	Water (mg/L)		***Soil/Sediment (mg/kg)***
Site-Specific TAL Inorganics			
Aluminum	0.20		20
Antimony	0.06		6
Arsenic	0.01		1
Barium	0.20		20
Beryllium	0.005		0.5
Cadmium	0.005		0.5
Chromium	0.01		1
Cobalt	0.50		5
Copper	0.025		2.5
Iron	0.10		10
Lead	0.003		0.3
Manganese	0.015		1.5
Mercury	0.0002		0.1
Nickel	0.04		4
Selenium	0.005		0.5
Silver	0.01		1
Thallium	0.01		1
Vanadium	0.05		5
Zinc	0.02		2
Cyanide, Total	0.01		0.5
Cyanide, Amenable	0.01		0.5
Additional Parameters			
Chloride	1		10
Total Organic Carbon (TOC)	--		100
Grain Size	--		0.1 percent

Notes:

[1] - Please note that these are estimated quantitation limits and are presented for guidance only. Actual quantitation limits are highly matrix dependent and may be elevated due to matrix effects QA/QC problems and high concentrations of target and non-target analytes.

TABLE 4.1

CONTAINER, PRESERVATION, SHIPPING AND PACKAGING REQUIREMENTS
GM POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Analyses	*Sample Containers* [1]	*Preservation*	*Maximum Holding Time from Sample Collection* [2]	*Volume of Sample*	*Shipping*
WATER (Surface Water & Groundwater)					
VOC	Three 40 mL teflon-lined septum vials	HCl to pH < 2 Iced, 4 ± 2° C	14 days for analysis	Fill completely, no air bubbles	Overnight or Hand Delivei
SVOC & PCB	Two 1 liter amber glass bottles per analysis	Iced, 4 ± 2° C	7 days for extraction 40 days after extraction for analysis	Fill to neck of bottle	Overnight or Hand Delivei
Metals & Hardness	One 500-ml plastic bottle	HNO_3 to pH < 2 Iced, 4 ± 2° C	180 days (mercury-28 days) for analysis	Fill to neck of bottle	Overnight or Hand Delivei
Total and Amenable Cyanide	One 500-ml plastic bottle	NaOH to pH $\geq$ 12 Iced, 4 ± 2° C	14 days for preparation and analysis	Fill to neck of bottle	Overnight or Hand Delivei
Chloride	One 500-ml plastic bottle	Iced, 4 ± 2° C	28 days for analysis	Fill to neck of bottle	Overnight or Hand Delivei
SOLID (Soil & Sediment)					
VOC	Three 5g EnCore Sampler™ per analysis or one 2-ounce glass jar	Iced, 4 ± 2° C	48 hours for preservation 7 days after collection for analysis	Fill completely	Overnight or Hand Delivei
SVOC & PCB	One 4-ounce glass jar	Iced, 4 ± 2° C	14 days for extraction 40 days after extraction for analysis	Fill to shoulder of jar	Overnight or Hand Delivei

TABLE 4.1

CONTAINER, PRESERVATION, SHIPPING AND PACKAGING REQUIREMENTS
GM POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Analyses	*Sample Containers* [1]	*Preservation*	*Maximum Holding Time from Sample Collection* [2]	*Volume of Sample*	*Shipping*
SOLID (Soil & Sediment) Continued					
Metals	One 4-ounce glass jar	Iced, 4 ± 2° C	180 days (mercury 28 days) for analysis	Fill to shoulder of jar	Overnight or Hand Delivei
Total and Amenable Cyanide	One 4-ounce glass jar	Iced, 4 ± 2° C	14 days for preparation and analysis	Fill to shoulder of jar	Overnight or Hand Delivei
TOC, Chloride	One 4-ounce glass jar	Iced, 4 ± 2° C	28 days for analysis	Fill to shoulder of jar	Overnight or Hand Delivei
SURFACE WIPE					
PCB	One 4-ounce glass jar	Hexane, Iced, 4 ± 2° C	7 days for extraction 40 days after extraction for analysis	100 cm^2	Overnight or Hand Delivei

Notes:

[1] - Multiple parameters (non-volatile) on a single soil sample may be combined into one single 8-ounce glass jar.

[2] - These are technical holding times, i.e., are based on time elapsed from time of sample collection.

TABLE 7.1

SUMMARY OF ANALYTICAL METHODS
GM POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Parameter[1]	*Preparation Method*[2]	*Laboratory Preparation SOP*	*Analytical Method*[2]	*Laboratory Analytical SOP*
Surface Water and Groundwater Samples				
VOC	SW-846 5030B	CORP-MS-0002NC	SW-846 8260B	CORP-MS-0002NC
SVOC	SW-846 3520C	CORP-OP-0001NC	SW-846 8270C	CORP-MS-0001NC
PCB	SW-846 3500C	CORP-OP-0001NC	SW-846 8082	CORP-GC-0001NC
Metals				
ICP Metals	SW-846 3010A / 3020A	CORP-IP-0003	SW-846 6010B	CORP-MT-0001NC
mercury	SW-846 7470A	CORP-MT-0005NC	SW-846 7470A	CORP-MT-0005NC
Total and Amenable Cyanide	SW-846 9012A	NC-WC-0032	SW-846 9012A	NC-WC-0031
Chloride	EPA-WW 300.0	NC-WC-0084	EPA-WW 300.0	NC-WC-0084
Soil and Sediment Samples				
VOC	SW-846 5035[3]	CORP-MS-0002NC	SW-846 8260B	CORP-MS-0002NC
SVOC	SW-846 3550B	CORP-OP-0001NC	SW-846 8270C	CORP-MS-0001NC
PCB	SW-846 3550B	CORP-OP-0001NC	SW-846 8082	CORP-GC-0001NC
Metals				
ICP Metals	SW-846 3050A	CORP-IP-0002NC	SW-846 6010B	CORP-MT-0001NC
mercury	SW-846 7471A	CORP-MT-0007NC	SW-846 7471A	CORP-MT-0007NC
Total and Amenable Cyanide	SW-846 9012A	NC-WC-0032	SW-846 9012A	NC-WC-0031
TOC	SW-846 9060	NC-WC-0017	SW-846 9060	NC-WC-0017
Chloride	EPA-WW 300.0	NC-WC-0084	EPA-WW 300.0	NC-WC-0084
Surface wipe Samples				
PCB	SW-846 3500C	CORP-OP-0001NC	SW-846 8082	CORP-GC-0001NC

Notes:

[1] Refer to Tables 1.2 and 1.3 for the compounds / elements of each parameter group.

[2] Method References:

- SW-846 - "Test Methods for Evaluating Solid Wastes, Physical / Chemical Methods ", SW-846, 3rd Edition, and Promulgated Updates, November 1986.
- EPW-WW - "Methods for the Chemical Analysis of Water and Wastewater", EPA / 600 / 4-79-020, Revised March 1983.
- EPA-DW 508 - "Methods for the Determination of Organic Contaminants in Drinking Water", EPA / 600 / 4-88 / 039

TABLE 11.1

ROUTINE MAINTENANCE PROCEDURES AND SCHEDULES
GM POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Instrument/Equipment	*Maintenance Procedures/Schedule*	*Spare Parts in Stock*
Gas Chromatograph / Mass Spectrometer (GC / MS)	1. Replace pump oil as needed. 2. Change septa weekly or as often as needed. 3. Change gas line dryers as needed. 4. Replace electron multiplier as often as needed. 5. Replace gas jet splitter as needed. 6. Replace GC injector glass liner weekly or as often as needed. 7. Replace GC column as needed. 8. Check daily to ensure that gas supply is sufficient for the day's activity, and the delivery pressures are set as described in the SOP. 9. Check daily to ensure the pressure on the primary regulator never runs below 100 psi. 10. Clean source as needed.	1. Syringes 2. Septa 3. Various electronic components 4. Glass jet splitter 5. GC column 6. Glass liners
Gas Chromatograph	1. Change septa weekly or as often as needed. 2. Change gas line dryers as needed. 3. Replace GC injector glass liner weekly or as often as needed. 4. Replace GC column as needed. 5. Clean / replace GC detector as needed. 6. Check daily to ensure that gas supply is sufficient for the day's activity, and the delivery pressures are set as described in the SOP. 7. Check daily to ensure the pressure on the primary regulator never run below 100 psi.	1. Syringes 2. Septa 3. Detectors 4. Glass liner 5. GC column

TABLE 11.1

ROUTINE MAINTENANCE PROCEDURES AND SCHEDULES
GM POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Instrument/Equipment	*Maintenance Procedures/Schedule*	*Spare Parts in Stock*
Purge and Trap Sample Concentrator	1. Replace trap as needed. 2. Decontaminate the system after running high concentration samples or as required by blank analysis. 3. Leak check system daily and as often as needed 4. Check daily to ensure the gas supply is sufficient for the day's activity, and the delivery pressures are set as described in the SOP. 5. Check daily to ensure the pressure on the primary regulator never run below 100 psi.	1. Spare traps 2. Spare sparger vessels 3. Various electronic components / circuits 4. Plumbing supplies - tubing, fittings
Mercury Analyzer	1. Clean tubing and quartz cell weekly or as often as needed. 2. Clean aspirator after each batch samples or as necessary. 3. Check daily to ensure the gas supply is sufficient for the day's activity, and the deliver pressures are set as described in the SOP.	1. Quartz cells 2 Aspirator
Inductively Coupled Plasma Spectrometer (ICP)	1. Clean torch assembly and mixing chamber when discolored or after eight hours of running high dissolved solid samples 2. Clean nebulizer as needed. 3. Check daily to ensure the gas supply is sufficient for the day's activity pressures are set as described in the SOP.	1. Spare torch and mixing chambers 2. Spare nebulizer 3. Spare capillary tubing

TABLE 11.1

ROUTINE MAINTENANCE PROCEDURES AND SCHEDULES
GM POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Instrument/Equipment	*Maintenance Procedures/Schedule*	*Spare Parts in Stock*
ICP / Mass Spectrometer (MS)	1. Change the peristaltic pump tubing 2. Inspect the sampler and skimmer cones for cleanliness, clean if necessary. 3. Check the vacuum system oil levels 4. Rinse nebulizer with 1% HNO3 for five minutes 5. Clean torch assembly and mixing chamber when discolored	1. Peristaltic pump tubing 2. Spare torch and mixing chambers 3. Spare nebulizer 4. Spare capillary tubing
Autoanalyzer / Spectrophotometer	1. Inspect pump tubes after each 8-hour run; replace if discolored or distorted 2. Check daily to ensure the gas supply is sufficient for the day's activity, and the delivery pressures are set as described in the SOP.	1. Pump tubing 2. Colorimeter lamps
pH Meter	1. Check battery (if used in field); and replace if discharged. 2. After use in samples containing free oil, wash the electrode in soap and rinse thoroughly with water. Immerse the lower third of the electrode in diluted HCl (1:9) solution for 10 minutes to remove any film formed. Rinse thoroughly with water. 3. Keep electrode properly filled with appropriate filling electrolyte solution.	1. Standard buffers 2. Electrolyte filling solution 3. Spare electrode
Specific Conductivity Meter	1. Check battery (if used in field); and replace if discharged. 2. After use in samples containing free oil, wash the electrode in soap and rinse thoroughly with water.	1. Standard solution 2. Spare electrodes

TABLE 11.1

ROUTINE MAINTENANCE PROCEDURES AND SCHEDULES
GM POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Instrument/Equipment	*Maintenance Procedures/Schedule*	*Spare Parts in Stock*
Photoionization Detector (HNU)	1. Check battery and recharge when low 2. Clean UV lamp; ion chamber, and fan if calibration cannot be achieved, or if readings are erratic	1. Battery charger 2. Spare lamps
Refrigerators	1. Monitor temperature twice daily.	
Ovens	1. Monitor temperature daily.	

ATTACHMENT 1

FIELD STANDARD OPERATING PROCEDURES (SOP)

ATTACHMENT 1

TABLE OF CONTENTS

1. Example Chain-of-Custody Record	Figure ATT-1.1
2. Laboratory Custody Sequence	Figure ATT-1.2
3. Field SOPs	
a) pH/Temperature	PHT-CRA-98
b) Dissolved Oxygen	DO-WP-93
c) Turbidity	NTU-WP-93
d) Specific Conductance	SC-CRA-98
e) Soil VOC Screening	VOC-CRA-98

CRA
CONESTOGA–ROVERS & ASSOCIATES, INC.
11100 Metro Airport Center Drive – Suite 160
Romulus, MI 48174 (734)942–0909

SHIPPED TO (Laboratory Name):

REFERENCE NUMBER:

PROJECT NAME:

CHAIN OF CUSTODY RECORD

SAMPLER'S SIGNATURE: _____ PRINTED NAME: _____

SEQ. No.	DATE	TIME	SAMPLE No.	SAMPLE TYPE	No. OF CONTAINERS	PARAMETERS	REMARKS
			TOTAL NUMBER OF CONTAINERS				

RELINQUISHED BY: 1. _____	DATE: TIME:	RECEIVED BY: 2. _____	DATE: TIME:
RELINQUISHED BY: 2. _____	DATE: TIME:	RECEIVED BY: 3. _____	DATE: TIME:
RELINQUISHED BY: 3. _____	DATE: TIME:	RECEIVED BY: 4. _____	DATE: TIME:

METHOD OF SHIPMENT: AIR BILL No.

White -Fully Executed Copy Pink -Shipper Copy
Yellow -Receiving Laboratory Copy Goldenrod -Sampler Copy

SAMPLE TEAM:

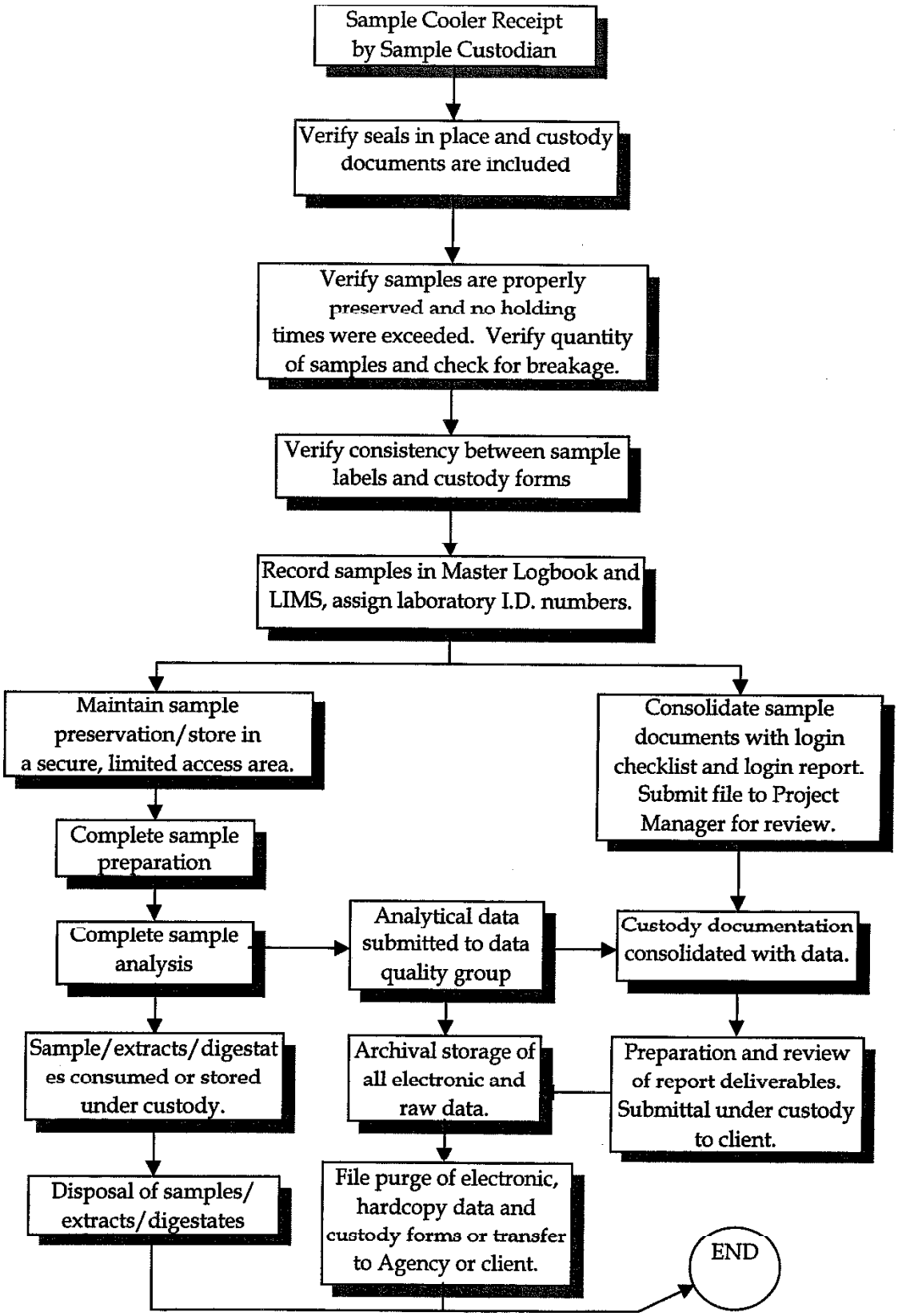
RECEIVED FOR LABORATORY BY:

DATE:_____ TIME:_____

figure ATT - 1.1

TYPICAL CHAIN-OF-CUSTODY RECORD
GMPT - BEDFORD PLANT PRELIMINARY RFI
Bedford, Indiana

FIGURE ATT 1.2
LABORATORY CUSTODY SEQUENCE



13968-9-Fig-Att-1.2.xls

pH/TEMPERATURE

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Potentiometric

Reference: "Methods for Chemical Analysis of Water and Wastes.", EPA-600/4-79-020, revised March 1983, Method 150.1

Sensitivity: 0.01 pH unit; 0.1 °C

Optimum Range: pH 1.00 to 12.00; temperature 0 to 100 °C

Sample Handling: Determined on site

Reagents and Apparatus:

1. Temperature compensated pH meter, Fisher Scientific Accumet Series 1000 or equivalent;
2. Combination pH electrode;
3. Temperature sensor verified against NIST traceable thermometer on an annual basis;
4. pH buffer solutions, pH 4.00, 7.00, and 10.00 (certified buffer solutions);
5. Deionized water in wash bottle.

Calibration:

1. Press On/Off key to turn meter on. If pH indicator in main display area is not on, press pH key to place meter in pH mode.

   Main display will show pH reading. If ATC probe is attached, temperature reading will appear in center display. If meter was previously standardized, buffers used will be shown in lower display.

2. Set pH display resolution by pressing Exp key until desired resolution is shown.

3. Clear current standardization points by pressing Stdz key. Then, within four seconds, press Clear key. Current standardization points will be removed from instrument memory.

4. Immerse electrodes in pH 4.00 buffer. Press Stdz key to begin standardization.

   Nominal (25 °C) value for buffer will begin flashing in lower, Standardization Values display. Main display will track electrode output in millivolts. When electrode output stabilizes, buffer value will cease flashing and remain on.

5. Rinse electrodes and immerse in pH 7.00 buffer. Press Stdz key to begin standardization with this buffer.

   In lower display, nominal (25 °C) value for second buffer will begin flashing, while value for first buffer remains on. Main display will track electrode output in millivolts. When electrode output stabilizes, second buffer value will cease flashing and also remain on.

6. Rinse electrodes and immerse in pH 10.00 buffer. Press Stdz key to begin standardization with this buffer.

   In lower display, nominal (25 °C) value for third buffer will begin flashing, while values for first and second buffers remain on. Main display will track electrode output in millivolts. When electrode output stabilizes, third buffer value will cease flashing and also remain on.

7. Rinse electrode with deionized water and place in pH 7.00 buffer. If meter reading is not 7.00, follow Steps 4 to 7 again.

Procedure:

1. Calibrate meter using calibration procedure.

2. Pour the sample into clean sample jar or plastic cup.

3. If stability bar is not currently on, press Auto key. Otherwise, press pH key. This initiates measurement of first sample pH.

Stability bar will flash and main display will begin to track sample pH. When both electrode output and ATC stabilize, stability bar will remain on with pH reading locked.

4. Record temperature and pH of the sample in the logbook.

5. Repeat steps 3 and 4 for each sample.

6. Recheck calibration with pH 7.00 buffer solution after a minimum of every 10 samples and after the last sample.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates must be ±0.2 pH units.

   If the results are outside of the control limits, rinse electrodes and repeat analysis. If results are still outside of the control limits, recollect samples and repeat analysis. If the results are still outside of the control limits, check calibration and recalibrate if necessary (see item 2, below). If drift is suspected to be the cause of the problem, clean the electrode and recalibrate. If drift is still apparent, replace electrode.

2. Calibration check results must be ±0.10 pH unit of the true value. If the result is outside of ±0.10 pH unit, rinse electrodes and check solution again. If still outside the control limit, recalibrate the meter and reanalyze all samples analyzed since the last in control calibration.

3. All glassware is to be soap and water washed, tap rinsed and deionized water rinsed prior to analyses unless pre-cleaned sample jars are used.

Interferences:

Interferences in pH measurements occur with presence of weak organic and inorganic salts and oil and grease. If oil and grease are visible, note in logbook. Clean electrode with soap and water, followed by 10% HCl and deionized water. Then recalibrate meter before analysis of next sample.

DISSOLVED OXYGEN

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Potentiometric

Reference: "Methods for Chemical Analysis of Water and Wastes:", EPA-600/4-79-020, revised March 1983, Method 360.1

Sensitivity: 0.1 mg/L as O_2

Optimum Range: 0.1 mg/L to 20 mg/L O_2

Sample Handling: Determined on site

Reagents and Apparatus:

1. Temperature compensated dissolved oxygen (DO) meter, Corning Check Mate System;
2. Zero oxygen standard;
3. DO sensor filling solution;
4. DO membrane replacement kit;
5. Distilled or deionized water in wash bottle.

Setting Up DO Sensor:

The sensor is shipped dry and must be filled before use.

1. Unscrew the membrane cap from sensor and fill using DO electrolyte.
2. Tap membrane cap gently to remove air bubbles. Gently screw cap onto probe body allowing surplus electrolyte to run out. (Caution: Do not overtighten)
3. Fit sensor to meter module.

4. Allow 30 minutes for polarization of electrode.

Calibration:

1. Remove wetting cap from tip of sensor. Switch on meter.
2. For first calibration point, place sensor in zero oxygen solution. Allow sufficient time for sensor to stabilize.
3. Move the sensor in a gentle circular motion.
4. Make sure sensor is immersed to a depth of 40 mm to cover the temperature sensing element.
5. Press "CAL" key. CAL 1 is displayed on meter and after endpointing, the display automatically updates to zero.
6. For second calibration point, hold sensor in air. Press "CAL" key. CAL 2 is displayed. After endpointing, the display automatically updates to 100% O_2.
7. To adjust oxygen calibration for salinity and barometric pressure, press "Mode" key. In mg/L O_2 mode, press "CAL" key and 100 is displayed. Use down arrow and up arrow on the keypad to adjust the display according to the salinity and barometric pressure tables contained on the operating instruction leaflet.

Procedure:

1. Calibrate meter using calibration procedure.
2. Pour the sample into clean sample jar or plastic cup.
3. Place sensor in sample. After following the immersion, stirring and stabilization steps referred to during calibration, press "READ" key to obtain sample result.
4. Record result in the field logbook.
5. Repeat steps 2 through 4 for each sample.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates must be within 15%.

   If the results are outside of the control limits, rinse electrode and repeat analysis. If results are still outside of the control limits, recollect samples and repeat analysis. If the results are still outside of the control limits, check calibration and recalibrate if necessary (see item 2, below). If unable to recalibrate, replace sensor membrane.

2. Calibration check results must be within 10% of the true value. If the result is outside of 10%, rinse electrodes and check solution again. If still outside the control limit, recalibrate the meter and reanalyze all samples analyzed since the last in control calibration.

3. All glassware is to be soap and water washed, tap rinsed and distilled or deionized water rinsed prior to analyses unless pre-cleaned sample jars are used.

Interferences:

Interferences in DO measurements generally occur due to membrane coating. Clean probe as specified in the sensor manual.

The presence of other gases such as chlorine, nitrous and nitric oxide, hydrogen sulfide and sulfur dioxide interfere with DO measurements. The sulfur based compounds will tarnish the electrodes resulting in sluggish or erratic measurements. Polishing the electrodes as specified in the operating manual will restore the performance of the meter. Recalibrate meter before analysis of next sample.

TURBIDITY

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Nephalometric

Reference: "Methods for Chemical Analysis of Water and Wastes:", EPA-600/4-79-020, revised March 1983, Method 180.1

Sensitivity: 0.01 Nephalometric Turbidity Unit (NTU)

Optimum Range: 0 - 20; 0 - 200 NTU

Sample Handling: Determined on site

Reagents and Apparatus:

1. Direct reading turbidity meter, HF Scientific Model DRT-15C;
2. Cuvettes with screw tops;
3. Battery charger;
4. 0.02 NTU (nominal) reference standard;
5. Distilled or deionized water in wash bottle.

Calibration Check and Operation

The turbidimeter has been calibrated by the manufacturer and electronic calibration using freshly prepared formazin standards should only be performed if the electronic printed circuit board, the photodetectors or the light source has been replaced. The calibration procedure is presented in pages 5 and 6 of the operating manual (attached).

The procedures for calibration checks and the operation of the meter follows:

1. For accurate measurements in the low range rotate the cuvettes in the well to obtain the minimum reading. Mark the cuvette with one of the adhesive dots provided with the instrument so that orientation of the cuvette will be identical each time it is placed in the instrument.
2. To operate the turbidimeter, switch to the "20" range and place the Reference Standard (0.02 NTU) in the optical well.

3. With the light shield in place over the well, adjust the Reference Adjust knob to cause the meter to read the reference standard value on the scale. The unit is now ready for use in either range.

4. To make a measurement of a sample, clean one of the cuvettes and fill to within approximately 1/2" of top with sample. Place the top on the cuvette and carefully clean the outside surface of the cuvette with a lint free wiper such as KimWipes. Place the sample in the well and place the light shield over the well. Select the appropriate range for best readability. Record results in field logbook.

5. Repeat steps 3 and 4 for each sample.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates must be within ±15%.

   If the results are outside of the control limits, clean cuvettes and repeat analysis. If results are still outside of the control limits, recollect samples and repeat analysis. If the results are still outside of the control limits, check calibration and recalibrate if necessary (see item 2, below).

2. Calibration check results must be ±10% of the true value. If the result is outside of ±10%, clean cuvettes and check solution again. If still outside the control limit, recalibrate the meter and reanalyze all samples analyzed since the last in-control calibration.

3. All glassware is to be soap and water washed, tap rinsed and distilled or deionized water rinsed prior to analyses.

Interferences:

Interferences in turbidity measurements are generally due to dirty or scratched cuvettes. Handle only the top one-third of the cuvettes and wipe clean using a lint-free wiper (KimWipes or equivalent).

SPECIFIC CONDUCTANCE

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Specific Conductance

Reference: "Methods for Chemical Analysis of Water and Wastes" EPA-600/4-79-020, revised March 1983, Method 120.1

Sensitivity: 10 µmhos/cm

Optimum Range: 10-20,000 µmhos/cm

Sample Handling: Determine on site

Reagents and Apparatus:

1. Conductivity meter - Cambridge Scientific Industries Model 301353 or equivalent;
2. Deionized water;
3. Conductivity standard, 1,413 µmhos/cm @ 77 °F (0.01 M KCl).

Notes:

The conductivity meter is factory calibrated. The calibration is checked using a solution of known conductance and recalibrated, if necessary.

Recalibration:

To recalibrate conductance, remove black plug revealing the adjustment potentiometer screw. Add standard solution to cup, discard and refill. Repeat procedure until the digital display indicates the same value twice in a row. Adjust the potentiometer until the digital display indicates the known value of conductance. To increase the digital display reading, turn the adjustment potentiometer screw counterclockwise (clockwise to decrease).

Procedure:

1. Rinse the inside of sample cup with liquid to be measured. (This is especially important if samples with a wide range of conductivity are to be measured.)

2. Fill sample cup.

3. Fill sample cup at least 2/3 full. If the sample is hot boiler water, allow to cool to 160 ^{o}F or below.

4. Slide the right hand function switch to "TEMP" and push the "READ" button. If temperature reading is not stable, empty and refill cup several times to bring cup and sample to the same temperature.

5. Read the temperature on the digital display panel and adjust both temperature compensation knobs accordingly.

6. If the approximate conductance is known, slide the left hand range selector switch to the proper range.

   Example: If you expect the sample to be around 2,000 µmhos, slide the left hand selector switch to x 1,000.

7. Slide the right hand function switch to "COND" and push the "READ" button.

8. Multiply the digital display reading by the factor indicated by the position of the left hand range switch to determine conductance.

   Example: A display reading of 1.00 with the left hand range selector switch indicating x 1,000 is:

   1.00 x 1,000 or 1,000 µmhos/cm

   Note: If a single "1" appears on the left hand side of the digital display, the sample conductance is higher than the selected range. Slide the left hand (range) selector switch in one step intervals until a 3 or 4 digit display appears.

   Conversely, if a decimal display appears (such as 0.11) move the range selector switch to the left until a 3 or 4 digit number, 1.00 or larger, appears on the display. This puts the unit in a range affording the best accuracy. Caution: A single "1" always means that the conductance is higher than the selected range.

9. Repeat steps 1 through 8 for remaining samples.

Quality Control:

1. The quality control calibration check standard must be analyzed initially, after a minimum of every 10 samples and after the last sample. If less than 10 samples are analyzed, the calibration standards are still required. The standards must be within ±10 percent of the true value or the samples run after the last acceptable check standard are to be reanalyzed. Record the calibration standard in the field logbook.

2. Duplicate a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicate values are to be within ±15% of each other. If outside of this range, reanalyze the samples. If still outside the acceptance range, recollect sample and reanalyze. If still out, replace meter.

SOIL VOC SCREENING

Scope and Application: This method is applicable to screening VOCs in the headspace of soil samples.

Method: Photoionization

Sensitivity: Approximately 0.5 ppm depending on background

Optimum Range: Background to 2,000 ppm

Sample Handling: Determined on site

Reagents and Apparatus:

1. HNu Model PI-101 Photoionizer or Photovac Microtip Photoionization Detector;
2. Calibration gas;
3. Calibration apparatus and tubing;
4. Battery rechargers.

Calibration:

A) Calibration of the HNu

1. Connect the analyzer to the regulator and calibration gas cylinder with a short segment (butt connection) of tubing. The calibration gas consists of a mixture of isobutylene and zero air. Isobutylene is non toxic and safe to use in confined areas. There are no listed exposure levels at any concentration.

   It is important that the tubing be clean since contaminated tubing will affect the calibration reading. A cylinder containing less than 30 psig pressure will not be used as readings below that level can deviate up to ten percent from the rated value.

   Turn the function switch to the STANDBY position. Turn the zero knob until the meter needle rests at 0.

2. With the SPAN setting and the function switch at the same positions as listed in the Application Data Sheet or Calibration Report, open the valve on the cylinder until a steady reading is obtained.

3. If the reading is the same as the recorded data, the analyzer calibration for the original species of interest is still correct.

4. If the reading has changed, adjust the SPAN setting until the reading is the same.

5. Shut off the cylinder as soon as the reading is established.

6. Record and maintain this new SPAN setting.

7. Background organic vapor readings should be measured prior to any soil screening.

B) Calibration of MicroTip

1. Connect the supplied regulator to the span gas cylinder. Hand tighten the fittings. Observe proper handling techniques for all gases.

2. Open the valve on the gas bag by turning the valve stem fully counterclockwise.

3. Attach the nut to the regulator. Hand tighten the fittings.

4. Turn the regulator knob counterclockwise about half a turn to start the flow of gas.

5. Fill the gas bag about half full and then close the regulator fully clockwise to turn off the flow of gas.

6. Disconnect the bag from the adapter and empty it. Flush the bag a few times with the span gas and then fill it.

7. Close the gas bag by turning the valve clockwise.

8. Press SETUP and select the desired Cal Memory with the arrow keys and press ENTER. Press EXIT to leave Setup.

9. Press CAL and enter the desired response factor. Use Table 2 from the manual to find the correct response factor. If the compound is not in

Table 2 or you are not looking specifically for one compound then enter 1.00.

The concentration detected by MicroTIP will be multiplied by the response factor before it is displayed and logged.

10. Expose MicroTIP to zero air. Press ENTER and MicroTIP sets its zero point.

11. MicroTIP then asks for the span gas concentration. Enter the known span gas concentration and then connect the span gas bag adapter to the inlet.

12. Press ENTER and MicroTIP sets its sensitivity.

13. When MicroTIP's display reverts to normal, MicroTIP is calibrated and ready for use. Remove the span gas bag from the inlet.

Procedure:

1. Calibrate meter using the correct calibration procedure for the meter used.

2. The samples for VOC headspace screening will be prepared in the field by filling a 4-ounce soil jar to one-half its volume and sealing with a teflon-lined closure. The remaining sample will be placed in the appropriate jars for the analyses required.

3. Allow the sample for VOC headspace screening to remain at ambient temperature for a minimum of 10 minutes. This will allow for VOCs in the soil to reach equilibrium in the headspace of the jar.

4. Remove the lid of the soil jar slightly and insert the probe of the meter into the headspace.

5. Take the highest reading from the meter or readout of the instrument.

6. Record the reading in the field logbook.

7. Recheck calibration with calibration gas after a minimum of every 10 samples and after the last sample.

Quality Control:

1. Calibration check results must be ±10 percent of the true value. If the result is outside of ±10 percent, recalibrate the meter as specified above.

2. Duplicate samples are not analyzed since the headspace VOC readings will vary considerably as the soil VOCs volatilize.

Interferences and Limitations:

Humid conditions will cause a negative bias to the reading. The photoionization detection principle used by the instruments will not detect all VOCs. The instrument is most sensitive to aromatic, alkene and alkyne VOCs.

ATTACHMENT 2

LABORATORY STANDARD OPERATING PROCEDURES (SOP)

ATTACHMENT 2

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iii)	Nonconformance and Corrective Action	CORP-QA-0010
iv)	Total Solids, Percent Moisture, Ash and Total Volatile Solids	NC-WC-0004
B.	Organics Preparation and Analysis	
i)	Determination of Volatile Organics by GC/MS Based on Methods 8260B, 624, and 524.2.	CORP-MS-0002NC
ii)	Extraction and Cleanup of Organic Compounds from Waters and Soils, Based on SW-846 3500 Series, 3600 Series, 8151A and 600 Series Methods.	CORP-OP-0001NC
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C.	Inorganics Preparation and Analysis	
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ATTACHMENT 2

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vi)	Total Organic Carbon (TOC) and Total Inorganic Carbon (TIC)	NC-WC-0017
vii)	Cyanide Preparation Method	NC-WC-0032
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STL STANDARD OPERATING PROCEDURE

TITLE: SAMPLE RECEIVING AND SAMPLE CONTROL

(SUPERSEDES: REVISION 6, 09/02/99)

Prepared by:		6/27/01 Date
Reviewed by:	Technology Specialist	6-27-01 Date
Approved by:	Quality Assurance Manager	6/28/01 Date
Approved by:	Environmental Health and Safety Coordinator	6-28-01 Date
Approved by:	Laboratory Director	7-2-01 Date

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1 SCOPE AND APPLICATION

1.1 It is the responsibility of Sample Receiving and Control personnel to perform the procedures described herein in full compliance with this SOP.

1.2 It is the responsibility of the Laboratory Director, QA Manager, and departmental Supervisor of the facility to assure that the procedures described are performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable personnel to perform this SOP correctly.

1.3 Analyst

1.3.1 It is the responsibility of the analyst to provide the correct request(s) for bottles to the sample custodian by using the QuantIMS program, PSR024.01 (as described in section 5.3) and to return all bottles to the custodian.

1.4 Sample Custodian

1.4.1 It is the responsibility of the sample custodian or designee to fill all bottle requests in a timely fashion and document the transfers on the request/return forms (Appendix 17.1.6).

1.4.2 It is the responsibility of the sample custodian or designee to correctly document the bottle information required (Appendix 17.1.6).

1.4.3 It is the responsibility of the sample custodian or designee to ensure that the returned bottle is the same as the one relinquished and to return it to the proper storage area.

1.4.4 This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2 SUMMARY OF METHOD

2.1 Not applicable.

3 DEFINITIONS

3.1 Sample Custodian refers to sample control personnel or designee.

3.2 Refer to the glossary in the Laboratory Quality Manual (LQM), latest version

4 INTERFERENCES

4.1 Not applicable.

5 SAFETY

5.1 Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

5.2 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately. {VITON gloves may be worn when halogenated solvents are used for extraction or sample preparation, nitrile gloves may be used when other solvents are handled. [Note: VITON is readily degraded by acetone; all solvents will readily pass through disposable latex rubber gloves.]}

5.3 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. {The following specific hazards are known} The following specific hazards are known:

5.3.1 The following materials are known to be corrosive: Sulfuric Acid, Nitric Acid, Hydrochloric Acid, Sodium Hydroxide.

5.4 Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5 The preparation of standards and reagents {and glassware cleaning procedures that involve solvents such as methylene chloride} will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6 All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported immediately to a laboratory supervisor.

5.7 This document accurately reflects current standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

6 EQUIPMENT AND SUPPLIES

6.1 Thermometers

6.2 PPE such as gloves, lab coats, safety glasses, etc.

6.3 Utility knives

6.4 PH paper

6.5 Copier, printer, computer and label generator

6.6 Carts

7 REAGENTS AND STANDARDS

7.1 Not applicable.

8 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Not applicable.

9 QUALITY CONTROL

9.1 Nonconformance and Corrective Action

9.1.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10 CALIBRATION AND STANDARDIZATION

10.1 Not applicable.

11 PROCEDURE

11.1 Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.

11.2 The procedures listed in this document describe the responsibilities of Sample Control personnel in ensuring that data is transmitted correctly from the client samples to all personnel involved with sample analysis and review.

11.3 The intent of the sample custodian maintenance program is to show custody of individual bottles based on workorder number as well as bottle control numbers. This program acts as an internal chain of custody.

11.4 The sample control group opens each cooler and removes the enclosed sample documents. Coolers are prioritized based on rush status and expirable tests. If a COC (chain-of-custody) is marked rush, the following occurs:

24 hour TAT – flag with red folder
48 hour TAT – flag with blue folder
72 hour TAT – flag with yellow folder
1 week TAT – flag with green folder

11.5 If tests on the COC are expirable, the cooler is marked with a manila folder.

11.6 Rush and expirable coolers are to be unpacked first and logged as soon as possible. The lab groups are to be notified with any rush 72 hours or less.

11.7 The following information on the Cooler Receipt/ Narrative Form (Appendix 17.2.1).

11.7.1 Presence of the custody seals on the outside of the cooler

11.7.2 Presence of the custody papers inside the cooler

11.7.3 The custody papers were properly filled out (ink, signed, match labels)

11.7.4 The custody papers were signed in the appropriate place

11.7.5 Presence of the shipper's packing slip

11.7.6 Presence of packing material information: if yes, type of packing material

11.7.7 Conditions of samples at receipt (chilled, etc.).

11.7.7.1 If temp vial is present, it is used to take the temperature. The temperature of the temp vial is taken as soon as it is removed from the cooler. If a temp vial is not present, proceed to section 11.7.7.2.

11.7.7.2 When a temp vial is not present the temp is taken by placing the thermometer probe between the coolant and the sample(s). If a coolant is not present, proceed to section 11.7.7.3.

11.7.7.3 If no coolant is in the cooler, the thermometer probe is placed between two sample bottles and the temperature recorded.

11.7.7.4 If the temperature is outside 4°C ± 2°C, the anomaly is recorded on the cooler receipt form. The project manager is contacted when the temperature is >6°C.

11.7.8 Bottles sealed in separate plastic bags

11.7.9 Condition of bottles upon receipt (good condition, broken, etc.)

11.7.10 Complete bottle labels (date, time, client ID)

11.7.11 Information on bottle labels and tags agree with custody papers

11.7.12 Correct bottles used for the tests indicated

11.7.13 VOA bottles were checked for the presence of air bubbles. Any bubbles exceeding 6 mm in diameter are narrated and the PM is contacted.

11.7.14 Sufficient amount of sample sent in each bottle

11.7.15 Samples were received via overnight courier, client drop off, or other means

11.7.16 pH's are taken, on all preserved samples less Volatiles, TOC, and TOX by removing sample lids and using a droplet of sample from in the lid to test the pH. The pH's are then recorded on the cooler receipt form. The pH paper strips are then discarded.

11.7.16.1 In determining if the sample is preserved to the correct pH for Navy samples, the sample custodian or receiving personnel must take an aliquot of sample out of the sample container either by pouring out a small aliquot or using a disposable pasteur pipette and place a drop of the sample on a pH paper strip.

11.7.17 Purchased prepared vials of preservatives are used if samples are not at the correct pH. The pH is adjusted by adding the appropriate preservative in 5 mL increments up to a maximum of 20 mL or unless there is a reaction. Sulfides are preserved with 5 mL Sodium hydroxide and 1 mL Zinc acetate. The pH adjustment is noted on the cooler receipt form.

11.7.18 The concentrations of the preservatives used:

4N Sodium Hydroxide
1N Zn Acetate
1:1 HCL (18%)
1:4 $HN0_3$ (18%)
1:2 H_2SO_4 (33%)

11.7.18.1 If the pH of Navy samples are adjusted, the sample custodian or receiving personnel must record on the cooler receipt form the laboratory assigned reagent ID number.

11.8 The Sample Control person is to remove all sample containers. Any broken, leaking, or dirty sample containers are to be placed inside the fume hood. Dirty sample containers are to be cleaned appropriately, so as not to contaminate the sample storage area. The Sample Control person is to wear disposable latex gloves, safety glasses, and a lab coat while handling any samples.

11.9 Any volatile sample(s) suspected (e.g., odor) or known (client information or site history) to be high in volatile concentration, the volatiles department will be contacted for pick-up and segregation of sample(s).

11.10 The Sample Control person is to examine all documents and compare information from sample container labels and Chain-of-Custody Records to insure that there is no discrepancy between documents, ensuring that all documents are properly completed and signed.

11.11 If any problems or discrepancies are noted during the sample receiving process that compromise sample integrity, such as limited sample volume, sample identification cannot be determined from the COC, incorrect pH levels (or preservatives if known), or broken, leaking samples, the Project Manager is immediately notified. They in turn will contact the client in an attempt to solve discrepancies.

11.12 Expirable tests (hold time 48 hours or less) must be written on the top of the bottles from which they are to be analyzed. If more than one method exists for the analysis, the method must also be written. These bottles designated for expirable tests will be given directly to the wet chem lab group.

11.13 All solid /soil samples received are split off for the total solid analysis. Splitting these samples is the responsibility of the receiving group. A small portion (approx. 5-10 grams) from each sample is put into a small plastic snap-top container designated for the TS analysis. If the TS container is not labeled with a QuantIMS label at the time of splitting off, the TS container must be labeled with handwritten client ID. Containers designed for VOC analysis must not be opened/used for TS aliquot. When only one solid container is received for VOC analysis, receiving group must **not** split off because of possible contamination or possible lost volatiles. An empty TS container with a QuantIMS label is given to the VOC analysis group for each solid. The VOC group will aliquot for the TS when they open container for analysis. TS plastic containers are placed into baggies by lot (project) and the baggies are put into a box inside the walk-in cooler door.

11.14 If all samples recorded on the Chain-of-Custody Record were received by the laboratory and there are no problems observed with the sample shipment, the Sample Control person will sign the Chain-of-Custody Record in the "Received for Laboratory by:" box on the document. If problems are noted, sign for shipment and note the problems. All discrepancies are recorded on Cooler Receipt Form.

11.15 A quote must reflect what is on the chain-of-custody. Any discrepancies must be resolved by the project manager. Likewise, if there is not an associated quote in QuantIMs, the samples are placed on hold until a quote is completed by the project manager.

11.16 In the event that a project is on hold until the next day or longer, all associated paperwork is placed into a black folder and all samples and black folder are put into cold storage. Any project that is on hold must be recorded on the dry-erase board posted on walk-in cooler door.

11.17 The Sample Control person will enter each sample into the laboratory computer (QuantIMS), where a unique lot number is assigned to each project received, and sequential sample numbers are designated for each client identification within the lot.

11.17.1 Lot Numbers: The lot number is nine characters in length and is based on the date of receipt. Lot number A9J010121 is described as follows:

A - STL location where the samples were received.

(A = North Canton, B = Tampa, C = Pittsburgh, etc.)

9 - Last digit of the year (i.e. 1999).

J - Month (i.e. A = January, B = February, J = October, etc.)

01 - The next 2 numeric characters identify the day of the month, in this case, the first day of the month.

0121 - The next 4 numeric characters are the sequential assignment of numbers specific to each lot received. Each day the first lot logged in receives the number "0101", the second lot receives the number "0102", etc..

For example:

If four bottles were submitted under Client ID numbers AB100-AB103 and the laboratory identification number generated by the computer is A9K100101, then the assigned laboratory number recorded on the Sample Log-In Sheet would be as follows.

Client ID Sample Number	Assigned Laboratory Number
AB 100	A9K100101-001
AB 101	A9K100101-002
AB 102	A9K100101-003
AB 103	A9K100101-004

11.17.2 Sample Numbers: The samples in each lot are assigned a sample number that is attached to the lot number and are reset at each new lot. For example: the first and second samples in the lot above are labeled A9J010121-001 and A9J010121-002.

11.17.3 Sample Suffixes: Each sample also has a 1 character field (which is not a required field for all samples) called the suffix which identifies the sample as specified below.

Client Sample	no suffix
Method Blank	B
Laboratory Control Sample	C
Laboratory Control Sample Duplicate	L
Matrix Spike	S
Matrix Spike Duplicate	D
Sample Duplicate	X
Serial Dilution	P
Sample Confirmation	Y
Post Digestion Spike	Z
Re-analysis	I

Example: A9J010121-001X is a sample duplicate for sample A9J010121-001.

11.17.4 Work Order Numbers: Each test requested by the client for an individual sample receives an individual 8 digit work order number assigned by QuantIMS. Work order number A5WE1-2-1C is described as follows:

A5WE1 - In addition to the three digit sample identification described in 4.7.2 (i.e. - 001 and - 002), the first 5 characters of the work order number also identifies each unique sample. This identification is generated in QuantIMS using a sequential logic that is beyond the scope of this SOP to describe.

2 - The "modifier" indicates the type of run. In this case this is the second time the sample had to be run. If it needs reprepped and run again, the number would indicate a "3". The original analysis work order number assigns "1" to the modifier position.

1C - The "suffix" is the identification of the specific test for that sample. The suffix in this case is not always sequential, but is unique to the test to be performed on the sample.

Example: A5WE1-2-1C is the assigned 8 digit work order number for the reanalysis of the chloride test on the sample A5WE1. A5WE1-1-05 could be the 8 digit work order number for the analysis of SW846 8270 on sample A5WE1.

11.17.5 Each sample container with the same client ID has a unique number. Each container will be labeled with the same 5 digit work order number and then will contain a suffix beginning with – 001, -002, -003, etc. For example, if 5 containers are submitted from the same sample point and the LIMS-generated work order number is CREE4, LIMS will generate 5 labels: CREE4-001, CREE4-002, CREE4-003, CREE4-004, CREE4-005. The specific bottle number is the number used for sample request/removal paperwork.

11.17.6 Expirable tests must be given to the lab groups the day they are received. The expirable test/method is written on top of the bottle and the bottle must be put in the red bin designated for expirables. The work order or sample ID must be recorded on the expirable logsheet – also must record the test to be run, special method if necessary and the initials of the person relinquishing the sample. The wet chem lab group checks this bin throughout the day and is responsible for signing out the sample container when they take it.

11.17.7 Once all sample containers have been properly labeled and all the information has been recorded by the Sample Lot Summary, the Sample Control person will place the samples into the proper storage locations. These locations are as follows:

11.17.8 Organic extractable samples (Semivolatiles, Pesticides/PCBs) are to be placed into the walk-in refrigerators located in Sample Receiving.

11.17.9 Volatile samples are to be stored in the double-door refrigerator located in the Sample Custodian area.

11.17.9.1 Samples known or suspected to be of high concentration are not stored in these refrigerators. The applicable volatile group is contacted to obtain the samples for segregation.

11.17.10 Inorganic samples are to be placed into the walk-in refrigerators located in Sample Receiving.

11.18 Preserved metal samples are placed in a non-refrigerated room located in the Sample Custodian area. Metals samples that need to be lab filtered and preserved are stored in the walk-in cooler. All metals for Navy projects are stored in the walk-in cooler.

11.19 One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.20 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.21 For clients who request a show of sample transfer from sample receipt to storage, a sample control record is completed (see Appendix 17.2.4 or Appendix 17.2.5). STL LIMS will generate this record for Expanded Deliverable and CLP designated samples. This record is also referred to as an internal chain of custody (COC). The form is completed as follows:

11.21.1 Laboratory sample number - record/list the individual five digit work order number in this column. List all pertaining samples in the project lot. Use an additional sheet if necessary. Do not record multiple lots on a sample control record.

11.21.2 Transferred by - record name of person making the transfer

11.21.3 Date - record date of sample transfer

11.21.4 Entered - "X" or "√" since samples are already logged into the LIMs.

11.21.5 Removed - leave blank since samples are not being removed

11.21.6 Reason - record "storage"

11.21.7 Date returned - leave blank

11.22 The completed sample control record is attached to the summary package. The sample control record can be manually printed using the SAM S31 command in QuantIMS. Note: The report package for the lot must be "D" (Expanded Deliverable) or "C" (CLP) for the report to print.

11.23 Samples are requested by an analyst through the QuantIMS program PSR024.01. This program is accessed from the Sample Receiving Menu (SAM) by selecting option S24 (Sample Removal Requests).

Bottles are requested based on method code, workorder number, prep code, QA batch number, lot number, or method group.

11.24 Each bottle has an associated lot number, sample workorder number, and bottle suffix number (Appendix 17.2.9).

11.25 When an analyst requests bottles and exits the request program, a requisition (Appendix 17.2.6) prints in the custodian's work area. This requisition identifies the requestor and the method/parameter requested. The sample custodian fills the requisition, recording each bottle number on the form. The bottle(s) is/are signed out to the custody of the requestor by indicating the name and the date of transfer. The request is relinquished with the custodian's initials and accepted by the requestor's initials.

11.26 When bottles are returned, the sample custodian records the return on the original requisition form and re-enters custody of the bottles to the sample control area. The custodian initials and dates the return on the form (Appendix 17.2.6).

11.27 When sample bottles are consumed in the analysis process, the empty container is returned to the custodian. The custodian marks a "C" by the appropriate sample on the request form to indicate a consumed sample (Appendix 17.2.6).

11.28 Samples returned after the custodian has left at the end of the day are placed in the walk-in cooler by a custodian designee and recorded as received by the custodian the next working day.

11.28.1 All request forms remain in the sample custodian area while samples are being analyzed. The final form is also kept in the sample custodian area after samples have been analyzed and reported.

11.28.2 When a sample custodian is not available, a designee gets the samples, and all request paperwork is completed properly.

11.29 Subcontracting of samples

11.29.1 Samples that are logged but not analyzed at the laboratory are subcontracted to different laboratories for analysis including other STL facilities.

11.29.2 The LIMs system will automatically print a Sample Analysis Requisition for these samples upon completion of the log-in process (Appendix 17.2.7).

11.29.3 This form contains information necessary for sample analysis. The original form is sent to the subcontracted laboratory and a copy is attached to the summary package. The Sample Analysis Requisition form must have a relinquished signature with a date and time. Any additional information necessary for sample analysis must be handwritten on the form (e.g. list of compounds, homogenizing of samples, limited quantity, etc.). In order to track subcontracted samples, the lab purchase order number on the Sample Analysis Requisition form must be recorded in the subcontracted sample PO book located in the receiving log-in area.

11.29.4 For samples sent to a non QuantIMS lab a chain of custody form is filled out and accompanies the samples.

11.29.5 A sample analysis request (SAR) can be printed from LIMS by entering the Lot Number. The SAR form can be found on Main Screen on receiving computer terminals.

12 DATA ANALYSIS AND CALCULATIONS

12.1 Not applicable.

13 METHOD PERFORMANCE

13.1 Training Qualifications:

13.1.1 The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.1.2 The only personnel authorized to execute this SOP are the Sample Log-In persons.

14 POLLUTION PREVENTION

14.1 This method does not contain any specific modifications that serve to minimize or prevent pollution.

15 WASTE MANAGEMENT

15.1 All samples may be disposed of 30 days after the report date except for those samples associated with special client retention. Shelves are purged in chronological order. All lots on a specific shelf must be noted. If a lot can be disposed of, a disposal date must be recorded.

15.2 Disposal dates are recorded on a print-out of lots according to a storage location. When clearing samples that cannot yet be disposed of because of special client retention, samples are boxed or stored on carts. Stored samples must have a specific date listed in which samples can be disposed of, or a note that indicates "SAVE" and client name or reason.

15.3 All lots assigned to a certain shelf can be obtained from QuantIMS.

15.3.1 UTL – "Enter"

15.3.2 Type "wrk" – "Enter"

15.3.3 Type "2" by option

15.3.4 Choose one query name and "Enter"

15.3.5 SLOIS for metals shelves

15.3.6 CLOIS for cooler shelves C1 – C272

15.3.7 WLOIS for cooler shelves W1-163

15.3.8 Type "1" next to "select records"

15.3.9 Enter the shelf location 3x where prompted (appears on two different screens). Then hit "Enter".

15.3.10 Dates need to be updated every four to six months so shelf locations only print most recent lots assigned.

15.3.11 "F3" to run query and "Enter".

15.3.12 At save definition type "Y" and run option "2" and "Enter".

15.4 Solid samples that can be disposed of are boxed and labeled "SOLIDS NOW."

15.5 Liquid wastes that can be disposed of are boxed and labeled "LIQUID NOW." These boxes are taken to the facility waste building and drummed by the Health and Safety Coordinator or designee.

15.6 Water samples that can be disposed of are placed on carts and purged down the drain in one of the following areas: sample receiving, wet chemistry, or extractions.

15.7 Solvent waste must be disposed of in clearly labeled waste cans.

15.8 Acid waste must be collected in clearly labeled acid waste containers. Solid materials (gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.9 Refer to the Laboratory Sample and Waste Disposal plan.

15.10 Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16 REFERENCES

16.1 STL Quality Management Plan (QMP), current version.

16.1.1 STL Laboratory Quality Manual (LQM), current version.

16.2 Associated SOPs and Policies, latest version

16.2.1 QA Policy, QA-003

16.2.2 Navy/Army SOP, NC-QA-0016

17 MISCELLANEOUS

17.1 Wherever "Sample Control" is mentioned in all SOPs, it is assumed to include the sample custodian or any alternate that is designated by the Sample Control Coordinator.

17.2 Appendix:

17.2.1 Cooler Receipt Form/Narrative

17.2.2 Preservative Preparation

17.2.3 Preservative Requirements

17.2.4 Internal Chain of Custody – CLP

17.2.5 Internal Chain of Custody – Regular Production Organic LIMS generated

17.2.6 Sample Custodian Removal Request

17.2.7 Sample Analysis Requisition

17.2.8 Subcontracted PO Logbook

17.2.9 Laboratory Generated Bottle Label

17.1.1

Example

Cooler Receipt/Narrative Form

STL Cooler Receipt Form/Narrative

North Canton Facility

Client:_____ Project:_____ Quote#:_____

Cooler Received on:_____ Opened on:_____ by: _____

(Signature)

Fedx ☐ Client Drop Off ☐ UPS ☐ Airborne ☐ Other:_____

Cooler ☐ Safe ☐ Foam Box ☐ Client Cooler ☐ Other:_____

STL Shipper No#:_____

1. Were custody seals on the outside of the cooler and intact? Yes ☐ No ☐

   If YES, Quantity_____ Location_____

   Were the custody seals signed and dated? Yes ☐ No ☐ NA ☐

2. Shipper's packing slip attached to this form? Yes ☐ No ☐

3. Were custody papers included inside the cooler and relinquished? Yes ☐ No ☐

4. Did you sign the custody papers in the appropriate place? Yes ☐ No ☐

5. Packing material used:

Peanuts ☐ Bubble Wrap ☐ Vermiculite ☐ Foam ☐ None ☐ Other :_____

6. Cooler temperature upon receipt _____ °C (see back of form for multiple coolers/temp)

METHOD: Temperature Vial ☐ Between Coolant & Sample Container ☐ Against Bottles ☐

COOLANT: Wet Ice ☐ Blue Ice ☐ Dry Ice ☐ Water ☐ None ☐

7. Were all the bottles sealed in separate plastic bags? Yes ☐ No ☐

8. Did all bottles arrive in good condition (Unbroken)? Yes ☐ No ☐

9. Did all bottle labels and tags agree with the custody papers? Yes ☐ No ☐

10. Were samples at the correct pH? Yes ☐ No ☐ NA ☐

11. Were correct bottles used for the tests indicated? Yes ☐ No ☐

12. Were air bubbles >6 mm in any VOA vials? Yes ☐ No ☐ NA ☐

13. Was a sufficient amount of sample sent in each bottle? Yes ☐ No ☐

Contacted PM _____ Date:_____ by:_____ via Voice Mail ☐ Verbal ☐ Other ☐

Concerning:_____

√	MACRO	MACRO
1. CHAIN OF CUSTODY		
	SR1A	Samples were received under proper custody procedures and without discrepancies.
	SR1B	The chain of custody and sample bottles did not agree. The following discrepancies occurred_____
2. SAMPLE CONDITION		
	SR2A	Sample(s)_____ were received or requested after the recommended holding time had expired.
	SR2B	Sample(s) _____ were received with insufficient volume
	SR2C	Sample(s)_____ were received in a broken container.
3. SAMPLE PRESERVATION		
	SR3A	Sample(s) _____ were further preserved in sample receiving to meet recommended pH level(s).
	SR3B	Sample(s)_____were received with bubble > 6 mm in diameter (cc: PM)
4. NCM		
	SR4A	NCM has been generated. Refer to Clouseau for details
5. Other Anomalies (see below or back)		

STL Cooler Receipt Form/Narrative

North Canton Facility

Client ID	pH	Date	Initials

Discrepancies Cont.

Macro Name:

Macro Name:

Macro Name:

Macro Name:

Other Anomalies:

17.1.2

Preservative Preparation

Preservative Preparation

(If purchased preservative solution vials are not used)

1:1 Hydrochloric Acid (18%): Slowly add 1000 mL concentrated HCl to 1000 mL reagent water and mix. Store in a well labeled plastic coated acid bottle.

1:2 Sulfuric Acid (33%): In a 2000 mL beaker, SLOWLY and CAREFULLY add 500 mL concentrated H_2SO_4 to 1000 mL reagent water and mix. A cool water bath may be needed to cool the solution and beaker. Store in a well labeled plastic acid bottle.

NOTE: *All preparations must be performed in a hood and proper personal protective equipment must be worn. All reagents and final preservative solution must be documented in applicable reagent logbooks.*

17.1.3

Preservative Requirements

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative[1,2]	Volume	Parameter	Container	Preservative [1,2]	Volume
Asbestos	P	None	250 mL	Radiological Alpha, Beta, Radium	P	HNO_3	4 L
Acidity	P	None	250 mL	Hardness	P	HNO_3	250 mL
Alkalinity (Sep)	P	None	250 mL	Metals	P	HNO_3	1 L
BOD	P	None	250 mL	Dissolved Metals*	P	HNO_3	1 L
Carbonaceous BOD	P	None	250 mL	Total Organic Carbon (TOC)	G	HCl	2 x40 mL
Bromide (Br)	P	None	250 mL	Chemical Oxygen Demand	P	H_2SO_4	250 mL
Chloride (Cl)	P	None	250 mL	Total Organic Halogens	G	H_2SO_4	250 mL
Chromium, $^{6+}$	P	None	250 mL	COD	P	H_2SO_4	250 mL
R. Chlorine	P	None	100 mL	Ammonia Nitrogen (NH_3)	P	H_2SO_4	500 mL
Color	P	None	50 mL	TKN	P	H_2SO_4	1L
Conductivity	P	None	250 mL	Nitrate/Nitrite	P	H_2SO_4	250 mL
Corrosivity	P	None	250 mL	Oil & Grease	G	H_2SO_4	1 L
Dissolved Oxygen	G	None	300 mL	Phenols	G	H_2SO_4	1 L
Fecal Coliform	P	None	125 mL	Total Phosphorus	P	H_2SO_4	250 mL
Flashpoint	G	None	100 mL	TON	P	H_2SO_4	1 L
Fluoride	P	None	250 mL				
Nitrate	P	None	250 mL	TRPH - IR 418.1	G	HCl	2 L
Nitrite	P	None	250 mL	VOC 601	G	HCl	3x40 mL
pH	P	None	50 mL	VOC 8010	G	HCl	3x40 mL

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative[1,2]	Volume	Parameter	Container	Preservative [1,2]	Volume
Elemental PO_4	G	None	250 mL	VOC 624	G	HCl	8x40 mL
Orthophosphate	P	None	250 mL	BTEX 8020	G	HCl	3x40 mL
TDS	P	None	250 mL	VOC 8240	G	HCl	3x40 mL
TSS	P	None	250 mL	THM/502.2	G	HCl	2x40 mL
Total Solids	P	None	250 mL	502.2	G	HCl & Asc. Acid	2x40 mL
TVS	P	None	250 mL	VOC 624	G	HCl	3x40 mL
T. Coliform	P	None	125 mL	VOC 602	G	HCl	3x40 mL
Settleable Solids	P	None	1L	465 C & D	G	HCl	4x40 mL
Silica	P	None	250 mL	BTEX 8021	G	HCl	3x40 mL
Sulfate	P	None	250 mL	VOC	G	HCl	3x40 mL
Sulfite	P	None	250 mL	VOC 8260	G	HCl	3x40 mL
Surfactants (MBAS)	P	None	250 mL	VOC and VOA	G	HCl	3x40 mL
Turbidity	P	None	250 mL	VOC 8010/8020	G	HCl	3x40 mL
TPH-GC	G	None	2 L				
				Total Cyanide	P	NaOH[3]	250 mL
BNAs	G	None	2 L	Amenable Cyanide	P	NaOH	250 mL
BNA + Dioxin	G	None	2 L	Free Cyanide	P	NaOH	250 mL
PNA/PAH	G	None	2 L	Sulfide	P	Zn Acetate & NaOH	1 L
Pesticides	G	None	2 L	Formaldehyde	G	None	500 mL
Reactive Cyanide	P	None	1 L	Carbonate	P	None	250 mL
Reactive Sulfide	P	None	1 L	Bicarbonate	P	None	250 mL
PCB	G	None	2 L	TPH - Diesel (Ext.)	G	None	2 L

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative[1,2]	Volume	Parameter	Container	Preservative [1,2]	Volume
Pesticides + PCBs	G	None	2 L	TPH - Gasoline (P&T)	G	HCl	2x40 mL
Herbicides	G	None	2 L	Glycols 8015	G	None	2x40 mL
OPPs	G	None	2 L	BTEX & MTBE	G	HCl	3x40 mL
				601/602	G	HCl	3x40 mL

* Filtered in field

[1] HCl, HNO_3, and H_2SO_4 to pH < 2. NaOH to pH > 12

[2] Temperature = 4°C ± 2°C except for aqueous metals

[3] Samples to be analyzed for Cyanide should be field-filtered for Residual Chlorine. If Residual Chlorine is detected, ascorbic acid (0.6 g) should be added.

17.1.4

Example

Internal Chain of Custody - CLP

STL North Canton

Sample Control Record

Client: _____ Case Number/SDG: _____

Storage Location: _____

Laboratory Sample Number	Transferred By	Date	Entered	Removed	Reason	Date Returned
1.						
2.						
3.						
4.						
5.						
6.						
7.						
8.						
9.						
10.						

17.1.5

Internal Chain of Custody

Regular Production-Organic

STL North Canton

Sample Control Record

Client: _____ Case Number/SDG: _____

Storage Location: _____

Laboratory Sample Number	Transferred By	Date	Entered	Removed	Reason	Date Returned
11.						
12.						
13.						
14.						
15.						
16.						
17.						
18.						
19.						
20.						

17.1.6

Example

Sample Custodian Removal Request Form

(LIMS Generated)

17.1.7

Example Sample Analysis Requisition

17.1.8

Example Subcontracted PO Logbook

17.1.9

Laboratory Generated Bottle Label

STL North Canton
STANDARD OPERATION PROCEDURE

TITLE: GLASSWARE WASHING

(SUPERSEDES: REVISION 3, DATED 01/11/01)

Prepared by: _____ 7/03/01
Date

Reviewed by: Patrick O'Meara 7/11/01
Technology Specialist Date

Approved by: _____ 7/3/01
Quality Assurance Manager Date

Approved by: _____ 7-10-01
Environmental Health and Safety Coordinator Date

Approved by: _____ 7-6-01
Laboratory Director Date

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1. PURPOSE

1.1. The procedure listed in this document will describe the standard operating procedures for washing glassware in the laboratories.

1.2. This document accurately reflects current standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

2. RESPONSIBILITIES

2.1. It is the responsibility of the analyst to perform the analysis described herein in full compliance with this SOP.

2.2. It is the responsibility of the Laboratory Director, QA Manager, and departmental Supervisor of the facility to assure that the analysis described is performed in full compliance with this SOP. It is also their responsibility to supply adequate training, materials, and equipment to enable the analyst to perform this SOP correctly.

3. SAFETY

3.1. Procedures shall be carried out in a manner that protects the health and safety of all STL North Canton associates.

3.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately. {VITON gloves may be worn when halogenated solvents are used for extraction or sample preparation, nitrile gloves may be used when other solvents are handled. [Note: VITON is readily degraded by acetone; all solvents will readily pass through disposable latex rubber gloves.]}

3.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

3.3.1. The following materials are known to be corrosive: **Nitric Acid, Hydrochloric Acid, Chromic Acid**

3.3.2. The following material is known to be flammable: **Acetone**.

3.4. Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

3.5. The glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operation will permit.

3.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported immediately to a laboratory supervisor.

3.7. Glassware in contact with chemicals used in analytical procedures may be toxic or carcinogenic. Therefore, each piece of glassware should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals should be reduced to the lowest possible level.

3.8. Hands must not be placed in the glassware while washing. An appropriate scrub brush must be used to clean the inside of glassware. This will prevent the breakage of glassware while trying to force hands in or out of apparatus.

4. PROCEDURES

4.1. Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.

4.2. Metals

4.2.1. Dirty glassware is taken to a central location and thoroughly rinsed. Any ink on the outside of the glassware is removed with acetone.

4.2.2. The glassware is immersed in a hot, soapy solution of water and laboratory detergent. An appropriate scrub brush or pad is used to scrub the glassware.

4.2.3. Rinse the glassware thoroughly three times with hot tap water and then rinse once with 1:1 nitric acid. Appropriate protective wear should be worn. If the glassware is still visibly dirty, or if spotting or beading occurs, repeat 4.2.2.

4.2.4. Rinse the glassware three times with analyte-free water and place it in a clean drying area.

4.2.5. After air drying, the glassware should be spot and stain free. If it is not, then repeat the entire procedure. Seldom used items should be stored to minimize contamination.

4.3. Wet Chemistry

4.3.1. After using a piece of glassware, it is thoroughly rinsed with hot tap water and carried to the dirty glassware area. If it is visibly dirty, it should be washed immediately.

4.3.2. The glassware is immersed in a hot, soapy solution of water and laboratory detergent. An appropriate scrub brush or pad is used to scrub the glassware. A mechanical, laboratory dishwasher equipped with a DI water rinse is also approved for glassware cleaning.

4.3.3. Rinse the glassware thoroughly three times with hot tap water. If the glassware is still visibly dirty, or if spotting or beading occurs, repeat 4.3.2.

4.3.4. After air drying, the glassware should be spot and stain free. If it is not, then repeat the entire procedure.

4.3.5. Place the glassware in a designated clean storage location free from interferences or contamination.

4.4. Semi-volatile Organics

4.4.1. If high level contamination is suspected perform the appropriate high level cleaning before proceeding with the standard cleaning process described in Section 4.4.2 and following.

4.4.1.1. Non-polar organics: if the glassware was most recently *wet* with a solvent, then rinse 3 times with that solvent, if *wet* with water, then rinse 3 times with acetone. Collect rinses in the appropriate solvent waste container. Allow the remaining solvent to evaporate from the glassware in a ventilation hood to reduce analyst exposure to the solvent.

4.4.1.2. Organic acids (e.g. phenols, carboxylic acids such as some herbicides): rinse 3 times with 1 M NaOH. Collect rinses in the water sample waste container.

4.4.1.3. Organic bases (e.g. amines [fish smell]): rinse 3 times with 1 M H_2SO_4. Collect rinses in the water sample waste container.

4.4.2. After using a piece of glassware, it is thoroughly rinsed with hot tap water and carried to the dirty glassware area. If it is visibly dirty, it should be washed or soaked immediately rather than allow the residue to harden and become more difficult to wash.

4.4.3. The glassware is immersed in a hot, soapy solution of water and laboratory detergent (recommended pH > 10). An appropriate scrub brush is used to scrub the glassware. Change the wash water (when it becomes cold, visibly dirty or is used on glassware with oil or sediment residue) by emptying, rinsing the inside of the sink with hot water and refill with hot water and detergent.

4.4.4. Rinse the glassware three times vigorously with hot tap water. If the glassware is still visibly dirty, or if spotting or beading occurs, rinse with 1:1 HCl and repeat 4.4.3.

4.4.4.1. If the HCl rinse is not effective, rinse or soak the item in chromic acid. Return as much chromic acid to the storage bottle as practical. Rinse the item with reagent water, collecting the rinses in an acid waste container. Chromic acid is very corrosive and must be handled with great care. Chromic acid can be reused until it begins to turn green. Spent chromic acid and chromic acid rinse water are transferred to the metals waste container.

4.4.5. After completing the hot tap water rinse, rinse the glassware three times with deionized water and place it in a clean drying area.

4.4.6. Place cleaned glassware used for semi-volatiles in a muffle oven for approximately one hour at 400°C. Do not heat glassware with visible residue. Baked on residue is much more difficult to remove.

4.4.7. Clean glassware is inverted (where applicable) and stored prior to use in a designated clean storage location.

4.4.8. Before use, glassware is to be pre-rinsed with the solvent inherent to a given procedure if stored more than one week.

4.5. Volatile organics

4.5.1. Rinse the glassware vigorously with hot tap water. If necessary, use a scrub brush to remove any remaining residue.

4.5.2. Place the cleaned glassware in the drying oven until dry or overnight.

4.5.3. Allow the cleaned and dry glassware to cool completely before use.

4.6. All Laboratory Groups

4.6.1. All glassware washing brushes must be hung on hooks when not in use to prevent contamination and ensure proper drying.

5. DEFINITIONS

5.1. Refer to the glossary in the Laboratory Quality Manual (LQM), latest version.

6. APPENDICES

6.1. See Table 1, Glassware Washing

6.2. Reagents

6.2.1. 1 M NaOH - 40±5 g sodium hydroxide dissolved in 1 L of reagent water.

6.2.2. 1 M H_2SO_4 - 55±5 mL concentrated sulfuric acid dissolved in 1 L of reagent water

6.2.3. Chromic acid – add a few milliliters of water to 40 g of potassium dichromate to make a paste, slowly add 600 mL of concentrated sulfuric acid while stirring.

Table 1 Glassware Washing

	Metals	Organics	Wet Chemistry
Wash	Hot Water, Detergent Solution	Hot Water, Detergent Solution	Hot Water Detergent Solution
Rinse	3 Times Tap Water 1 Time 1:1 Nitric Acid 3x Reagent Water	3 Times Tap Water 1 Time 1:1 Hydrochloric Acid* 3x Reagent Water	3 Times Tap Water 3x Reagent Water
Dry	Air	Muffle at 400°C for 1 hour	Air
Storage	Designated Cabinets & Shelves	Designated Cabinets & Shelves	Designated Cabinets & Shelves

*If the glassware is visibly dirty after washing in a hot detergent solution, the glassware is rinsed with 1:1 HCl and rewashed with a hot detergent solution.

Controlled Copy No. _____
Implementation Date _____

SOP No. CORP-QA-0010
Revision No. 2
Revision Date: 06/15/99
Page: 1 of 23

QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: NONCONFORMANCE AND CORRECTIVE ACTION SYSTEM

(SUPERSEDES: REVISION NO. 1)

Prepared by: Quanterra® QA Managers

Approved by: _____
Director of Quality Assurance

Approved by: _____
Director of Environmental Health and Safety

Approved by: _____
Vice President Operation Services

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1. PURPOSE

1.1. The purpose of this document is to establish procedures for the identification and documentation of nonconformances and the corrective actions taken as a result of these events. The Quanterra® Quality Assurance Management Plan (QAMP) requires documentation of instances of deviations from established control limits, approved standard operating procedure (SOPs), or client-specified requirements. The Nonconformance Memo (NCM) described in this procedure is used to document deviations from Quanterra® policies and procedures and documented client specifications including root causes and corrective actions taken to remedy the nonconformance. The NCM may be stored in electronic form in a database, or maintained using defined printed forms.

1.2. This document applies to procedures, services, analytical data, reports, or materials purchased by the laboratory or supplied by the laboratory to its clients. Nonconformances related to sample receiving activities may be documented separately from the NCM process by use of the Condition Upon Receipt (CUR) Form as described in a facility-specific SOP. Regardless of the type of system used to handle the sample receiving and client-related issues, including holding time violations (HTVs), the system must include and emphasize the immediate notification of the Project Manager (PM). This will allow the PM to initiate immediate client notification and resolution of how to proceed. See Section 5, Definitions, for further clarification of application.

1.3. Nonconformances can be identified by Quanterra® laboratory employees in the course of their daily operations or by external parties (i.e., customers and representatives of customers) through reviews of records, audit, or proficiency testing.

2. RESPONSIBILITIES

2.1. **Laboratory Associate:** During the course of their work, all employees are responsible for identifying and documenting problems, using a Nonconformance Memo, that might affect the quality of Quanterra's® product. They should also identify or attempt to seek out possible measures to correct the problem. By signing or initialing laboratory notebooks, forms, bench sheets, data reports, and other quality-related documents, associates are verifying that procedures have been followed. Any deviation that might render a measurement suspect shall be documented.

2.2. **Group Leader/Team Leader/Supervisor** (for purposes of this SOP, the term "Supervisor" will be used): Each supervisor is responsible for the review of NCMs to ensure that problems which might affect quality are adequately described and that personnel are assigned to correct them. Supervisors review hardcopy or electronic

versions of NCMs and forward them to the appropriate project manager. Together with project managers and Quality Assurance personnel, supervisors are responsible for determining the appropriateness of planned corrective actions.

2.3. **Project Manager (PM):** The project manager is responsible for relaying project requirements to staff so that special project requirements are understood and nonconformances recognized. The project manager communicates conformance problems to clients and documents decisions made with clients. The project manager ensures that short-term corrective actions for routine analytical QC failures are completed. An example would be making sure that repreparation and analysis of a sample was done. The project manager may withhold final reports to clients until corrective actions agreed to with the client have been completed.

2.4. **QA Manager:** The Quality Assurance manager or his or her designee is responsible for reviewing all initiated NCMs to ensure that actions taken are appropriate, and assisting in resolving QA/QC discrepancies. The QA staff will maintain a nonconformance tracking system to guarantee that each nonconformance is brought to closure. The system will also be used to monitor for trends that might indicate long-term quality problems. Systematic problems are investigated, NCMs issued and reviewed, and spot audits conducted to ensure that long-term corrective actions have been successfully completed. If review of an area reveals a significant problem with data quality, the Quality Assurance manager has the authority and responsibility to stop production in that laboratory area.

2.5. **Operations/Systems Manager :** The operations/systems manager shall ensure that corrective actions are correct and have been implemented. The operations/systems manager shall document this review and concurrence by signing the NCM as the responsible manager, if QA-required, for a specific corrective action. Along with the laboratory manager, the operations/systems manager shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work.

2.6. **Laboratory Manager:** The laboratory manager shall emphasize the importance of quality requirements and require all employees to report any problem that might adversely affect the quality of work. The laboratory manager is also responsible for the implementation of the NCM system in the laboratory.

2.7. **Corporate QA Director:** The Quanterra® Quality Assurance Director should be notified of any continuing nonconformances that are not properly addressed by operations or where the root cause cannot be identified.

3. SAFETY

3.1. Normal office dependent safety precautions must be taken in performing this SOP. If personnel are required to perform any portion of the procedure in laboratory areas, appropriate personal protective equipment and precautions must be utilized.

3.2. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra® associates.

3.3. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra® associate. The situation must be reported **immediately** to a laboratory supervisor.

4. PROCEDURE

4.1. When to Initiate a Nonconformance Memo

4.1.1. Lab associates are to prepare a hard copy or initiate an electronic nonconformance memo (NCM) whenever procedures, services, data, reports, electronic disk deliverables (EDDs), or standard materials deviate from established specifications. All nonconformances with the exception of matrix-related failures require an NCM (see definitions of nonconformance, anomalies, and deficiencies in Section 5 and Section 1.2 for exceptions). ALL holding time violations (HTVs) require an NCM.

4.1.2. All standard operating procedures (SOPs) shall be followed. By signing or initialing laboratory notebooks, bench sheets, data reports, and other quality-related documents, employees are verifying that the SOPs have been followed with the exceptions of the pre-approved deviations (as described in QAPjPs Quality Assurance Summaries, or equivalent systems). Any intentional deviation from an SOP must be pre-approved by the QA manager and Supervisor. Any deviation from an SOP or client requirement not previously approved must be documented using the NCM process.

4.1.3. An NCM is to be completed for each instance of a nonconformance. A single NCM can be used for a single event affecting multiple lot numbers and samples, but normally a separate NCM would be initiated for different nonconformance issues. If the nonconformance involves projects for multiple project managers, then the NCM will need to be routed to each project manager.

4.2. Determining Which NCM Process to Use

4.2.1. Laboratories equipped with Quanterra's® standard Laboratory Information Management System (QuantIMS) shall use the NCM database application developed for this purpose, **Clouseau.**

4.2.2. Laboratories not equipped with QuantIMS shall use a paper-based NCM process or an equivalent electronic system.

4.3. How to Process the NCM using Clouseau

4.3.1. Initiating the NCM

4.3.1.1. While properly logged into a PC where Clouseau has been installed, start the Clouseau program.

4.3.1.2. At the Main Menu, select Create NCM.

4.3.1.3. Enter the information required at the top of the form. At a minimum select the Production Area, Classification (anomaly or deficiency), NCM Type, and NCM Description.

4.3.1.4. For NCMs that affected samples, indicate the relevant batch numbers, lots, samples, and tests by using the Tests/Samples option within Clouseau.

4.3.1.5. Complete the Comments/Details and Corrective Action sections of the Create NCM Form. These sections may be filled out jointly with the supervisor. Consult the project manager or the operations/systems manager and QA manager if the supervisor and associate are uncertain of corrective actions. Be objective and specific but brief. Include enough information that decisions to approve the NCM can be made easily (include pertinent QC information).

4.3.1.5.1. Design corrective actions to correct the immediate problem (short-term corrective actions) and to minimize the possibility of its recurrence (long-term corrective actions). Examples of corrective actions are modifications to nonconforming procedures, repair or replacement of deficient equipment, training personnel, and reanalysis of any affected samples.

4.3.1.5.2. Where operational corrective actions are required, they shall be supported with reference to recovery data, control charts, or other documentation.

4.3.1.6. If the corrective action involved retraining, the training must be documented with the signatures of the trainees and submitted to the QA staff before the NCM is considered closed.

4.3.1.7. Save the NCM using the appropriate command in Clouseau. When presented with the Send E-Mail form, the supervisor of the affected Production Area will be listed in the "Notify Now" box, while affected Project Managers and QA staff will be listed in the "Notify Later" box. Verify that these names are correct. If any personnel should be informed that are not listed, add their names to the "Notify Now" box by double-clicking on their names in the Personnel box.

4.3.1.8. Initiate the NCM notification process by selecting SEND EMAIL.

4.3.2. Supervisory Review and Approval

4.3.2.1. The supervisor will receive notification of the NCM via e-mail. The supervisor must log into a computer workstation where Clouseau has been installed, and run Clouseau.

4.3.2.2. Using the Review NCM form, select the NCM to be reviewed and click on OPEN.

4.3.2.3. With the NCM on screen, review the information provided. Add necessary comments or corrective actions to the appropriate areas. If the corrective action has not been determined, the situation must be referred to the project manager and the operations/systems manager for resolution to ensure client requirements can be satisfied. The QA staff should be consulted if there are questions as to how to proceed. If the above input was not needed, the operations/systems manager does not need to have every NCM routed to him or her. If, upon receipt and review of the NCM by the QA staff, it is felt the operations/systems manager needs to be made aware of the issues, the QA staff will notify the operations/systems manager using the Under Review/Send Email options of Clouseau.

4.3.2.4. If the nonconformance is hardware/equipment related, the item shall be nonconformance tagged and segregated, if possible, to ensure that it is not used until repaired. Refer to Section 4.4.4.8.

4.3.2.5. The supervisor will be responsible for the completion of the corrective action unless otherwise indicated. Enter the name of the person responsible for performing the corrective action if other than the supervisor. This is Operations' commitment to rectify the problem. The supervisor selects the "Approved" option on the Review NCM form and selects SAVE. This initiates the NCM routing process, which will now route the NCM to the project manager. The project manager must receive the NCM in a timely manner, generally within 48 hours. If the NCM is for a holding time violation, a project manager ***must*** be notified ***immediately***.

4.3.3. Project Manager Review, Client Notification, and Project Documentation

4.3.3.1. The project manager shall determine if client notification is required to either assist in the definition of corrective action or to notify the client of problems related to sample analysis. The project manager shall indicate using the Client Notification Form in Clouseau the date and method of notification and client's response .

4.3.3.2. With the NCM on screen, review the information provided. Add necessary comments or corrective actions to the appropriate areas. Notify the supervisor of any changes made to the corrective action plan.

4.3.3.3. The project manager selects the "Approved" option on the Review NCM form and selects SAVE. This initiates the NCM routing to the QA office. The QA office then must review the NCM in a timely manner, generally within 72 hours.

4.3.3.4. If the nonconformance involves analytical work in process, the **final** report cannot be released until a project manager has approved the NCM. If it is found that erroneous analytical data (e.g., from data validation comments or phone requests, etc.; inaccurate chromatograms, spectra, calculations, or final reports) have been released by the laboratory, this fact must be documented on an NCM and forwarded to the QA office. Prior to making the corrections, proper documentation shall be filled out and turned into the QA staff if corrections are needed in the database (QuantIMS). The original data shall be marked as unusable and maintained for historical purposes. The corrective action shall include prompt client notification and issuance of amended reports.

4.3.4. Quality Assurance Review and Trending

4.3.4.1. The QA staff shall review all NCMs for conformance with standard laboratory practices and Quanterra® policies.

4.3.4.2. NCMs will be reviewed to ensure that the corrective action was completed and effective in addressing the root cause of the nonconformity to prevent recurrence.

4.3.4.3. Clouseau's reporting and tracking system will be used to monitor for repetitive failures that might indicate systematic problems. Tracking records would (when applicable) include:

- NCM log number
- Date initiated
- Project number
- Lab sample ID numbers
- Method or parameter
- Nonconformance description
- Corrective action required
- Characterization as an anomaly or deficiency
- Closure of NCM

4.3.4.4. The QA staff shall identify repetitive quality issues that may be systematic in nature and may require corrective actions to prevent recurrence. Recurrent technical or Information Technology problems shall be referred to the appropriate technical group for corrective actions. Correction of systematic problems could take the form of modifications of nonconforming procedures, repair or replacement of deficient equipment, training or replacement of personnel. Findings and corrective actions from these investigations or audits shall also be documented. Resolution of corrective actions for systematic problems must be documented by the responsible laboratory area along with supporting evidence.

4.3.4.5. The QA staff shall conduct follow-up assessments to confirm that correction of systematic problems is successful. These assessments can be done as part of the monthly spot assessment program at the laboratory.

4.3.4.6. The approval of the QA manager or designee is required in Clouseau to indicate that the NCM has been closed.

4.3.4.7. The QA office may maintain a central file/record of all closed NCMs.

4.4. How to Process NCMs (Non-QuantIMS laboratories)

4.4.1. Initiate the NCM

4.4.1.1. Enter the information required at the top of the form (see Example Form in Attachment A; printed forms are available from QA). Names or initials must be uniquely identified by using three initials or by spelling out the last name. Use specific test codes (when available) rather than generic codes such as "VOAs" or "Anions." At a minimum enter Project ID, QuantIMS batch number or LIMS QC Lot number if appropriate, in addition to sample numbers. The laboratory generating the NCM is identified as part of the log number (e.g., AUS-97-123). Therefore, separate laboratory location identification is not required on the form.

4.4.1.1.1. If the NCM is for a holding time violation, the project manager ***must*** be notified immediately.

4.4.1.1.2. An instrument tag-out does not require the completion of the NCM if there are no client samples involved (e.g., instrument out of service to perform routine maintenance.) See section 4.2.5.

4.4.1.2. Check appropriate NCM boxes. If box 31—"Other"—is checked, supply a brief description of the problem. Attach further information as necessary.

4.4.1.3. Complete the reverse side of the form concerning root cause and corrective actions. These sections may be filled out jointly with the supervisor. Consult the project manager or the operations/systems manager and QA manager if the supervisor and associate are uncertain of corrective actions. Be objective and specific but brief.

Include enough information that decisions to approve the NCM can be made easily (include pertinent QC information).

4.4.1.3.1. Design corrective actions to correct the immediate problem (short-term corrective actions) and to minimize the possibility of its recurrence (long-term corrective actions). Examples of corrective actions are modifications to nonconforming procedures, repair or replacement of deficient equipment, training personnel, and reanalysis of any affected samples. Additional sheets documenting corrective action may be attached to the NCM. Reference the attachment on the NCM.

4.4.1.3.2. Where operational corrective actions are required, they shall be supported with reference to recovery data, control charts, or other documentation.

4.4.1.4. If the corrective action involved retraining, the training must be documented with the signatures of the trainees and submitted to the QA staff.

4.4.1.5. Instrument/Equipment Nonconformance Tag

4.4.1.5.1. Instruments and equipment which habitually fail to meet calibration criteria or are out of service due to needed repair or other reasons must be marked with a tag indicating the nonconforming condition. (See Example in Attachment B.)

4.4.1.5.2. If the reason for the nonconformance tag caused sample data to be impacted, initiate an NCM and mark box 30. Identify the instrument by name and/or identification number and briefly describe the problem. This information can be recorded on page 2 under "Problem Description/Root Cause". Submit the NCM to the group supervisor.

4.4.1.5.3. Upon receipt of the NCM, QA assigns the NCM number and provides the number to the analytical group so that the nonconformance tracking number can be recorded in the instrument's maintenance log.

4.4.1.5.4. The corrective action will be to either permanently remove the instrument from service or to have the instrument repaired. If an instrument is repaired, its reliability must be

demonstrated through successful recalibration before the nonconformance can be closed. The nonconformance tag remains in effect during the demonstration period. Record this information in the instrument maintenance logbook. Reference the successful calibration on the tag and return the tag to the QA staff for closure of the NCM.

4.4.1.6. Submit the form to the supervisor responsible for the area indicated at the top of the form.

4.4.2. Supervisory Review, Approval, and Signature

4.4.2.1. Review the information provided. Complete or amend the descriptions of root cause and corrective action. If the corrective action has not been determined, the situation must be referred to the project manager and the operations/systems manager for resolution to ensure client requirements can be satisfied. The QA staff should be consulted if there are questions as to how to proceed. If the above input was not needed, the operations/systems manager does not need to have every NCM routed to him or her. If, upon receipt and review of the NCM by the QA staff, it is felt the operations/systems manager needs to be made aware of the issues, the QA staff will notify and document the notification of the operations/systems manager on the form.

4.4.2.2. If the nonconformance is hardware/equipment related, the item shall be nonconformance tagged and segregated, if possible, to ensure that it is not used until repaired.

4.4.2.3. The supervisor will be responsible for the completion of the corrective action unless otherwise indicated. Enter the name of the person responsible for performing the corrective action if other than the supervisor. This is Operations' commitment to rectify the problem. This will be verified by the QA staff and/or the operations/systems managers.

4.4.2.4. The supervisor signs and dates the "Corrective Action Approved By" line and forwards the original form to the project manager. The project manager must receive the NCM in a timely manner, generally within 48 hours. If the NCM is for a holding time violation, a project manager ***must*** be notified ***immediately***.

4.4.3. Project Manager Review, Client Notification, and Project Documentation

4.4.3.1. The project manager shall determine if client notification is required to either assist in the definition of corrective action or to notify the client of problems related to sample analysis. The project manager shall indicate on the NCM whether client notification is required or not.

4.4.3.2. Record the result of the client contact or leave blank if client contact was not required. Sign and date the NCM form. This must be done whether client notification is completed or not. The signature documents the project manager as having read and reviewed the NCM.

4.4.3.3. Document any changes to the corrective action plan, and notify the supervisor. The project manager's signature on the front of the NCM serves as documentation that the PM has read and reviewed the NCM and is aware of the corrective action plan.

4.4.3.4. The project manager will forward the original to the QA office within 72 hours for review and closure. The PM may keep a copy for his or her own personal use, but the original must be forwarded to the QA office.

4.4.3.5. If the nonconformance involves analytical work in process, the **final** report cannot be released until a project manager has signed the NCM.

4.4.3.6. If it is found that erroneous analytical data (e.g., from data validation comments or phone requests, etc.; inaccurate chromatograms, spectra, calculations, or final reports) have been released by the laboratory, this fact must be documented on an NCM (items 25 and 26 on NCM form) and forwarded to the QA office. Prior to making the corrections, proper documentation shall be filled out and turned in to the QA staff if corrections are needed in the database (LIMS or QuantIMS). The original data shall be marked as unusable and maintained for historical purposes. The corrective action shall include prompt client notification and issuance of amended reports.

4.4.3.7. After the QA staff has closed the NCM, the original of the closed NCM will be filed either in the project file or QA files per laboratory procedure.

4.4.4. Quality Assurance Review and Tracking

4.4.4.1. The QA staff shall review all NCMs for conformance with standard laboratory practices.

4.4.4.2. NCMs will be reviewed to ensure that the corrective action was completed.

4.4.4.3. A tracking system will also be used to monitor for repetitive failures that might indicate systematic problems. Tracking records would (when applicable) include:

- NCM log number
- Date initiated
- Project number
- Lab sample ID numbers
- Method or parameter
- Nonconformance description and NCM box number
- Corrective action required
- Characterization as an anomaly or deficiency
- Closure of NCM

4.4.4.4. The QA staff shall identify repetitive quality issues that may be systematic in nature and may require corrective actions to prevent recurrence. Recurrent technical or Information Technology problems shall be referred to the appropriate technical group for corrective actions. Correction of systematic problems could take the form of modifications of nonconforming procedures, repair or replacement of deficient equipment, training or replacement of personnel. Findings and corrective actions from these investigations or audits shall also be documented. Resolution of corrective actions for systematic problems must be documented by the responsible laboratory area along with supporting evidence.

4.4.4.5. The QA staff shall conduct follow-up assessments to confirm that correction of systematic problems is successful. These assessments can be done as part of the monthly spot assessment program at the laboratory.

4.4.4.6. The signature of the QA manager or designee is required for corrective actions to indicate that the issue has been closed.

4.4.4.7. The QA office shall maintain a central file/record of all NCMs.

4.4.4.8. Instrument/Equipment Nonconformance Tag

4.4.4.8.1. Instruments and equipment which habitually fail to meet calibration criteria or are out of service due to needed repair or other reasons must be marked with a clearly visible tag or sign indicating the nonconforming condition. (See Example in Attachment B.)

4.4.4.8.2. If the reason for the nonconformance tag caused sample data to be impacted, initiate an NCM (electronic or hard copy). Identify the instrument by name and/or identification number and briefly describe the problem.

4.4.4.8.3. Upon saving the NCM, the NCM number indicated by Clouseau shall be recorded in the instrument's maintenance log.

4.4.4.8.4. The corrective action will be to either permanently remove the instrument from service or to have the instrument repaired. If an instrument is repaired, its reliability must be demonstrated through successful recalibration before the nonconformance can be closed. The nonconformance tag remains in effect during the demonstration period. Record the back to control information in the instrument maintenance logbook. Reference the successful calibration on the tag and return the tag to the QA staff for closure of the NCM.

5. DEFINITIONS

5.1. ***Nonconformance:*** an unplanned deviation from an established protocol or plan. The deviation may be the result of Quanterra's® actions, then termed a **deficiency**, or the result of events beyond the control of Quanterra®, then termed an **anomaly**.

A nonconformance exists when:

5.1.1. Any laboratory QC sample (e.g., method blank, laboratory control sample, duplicate laboratory control sample, matrix spike, matrix spike duplicate, and surrogate spike) component result is outside established control limits and demonstrate a **systematic** deficiency. Any matrix spike or matrix spike duplicate or sample related QC outside of established control limits attributed

to matrix effects must be documented, but an NCM is not required. A procedure is not performed as described in the applicable SOP or QA Policy, ***except*** in cases where the procedure has been performed according to a client-specified document Quanterra® has agreed to follow (e.g., EPA SOWs and QAPjPs).

5.1.2. A practice or procedure is not performed as described according to a client or project document that Quanterra® has agreed to follow.

5.1.3. Purchased materials or services are determined to be defective and their use would affect data quality.

5.1.4. ANY holding time violations (HTVs) occurs regardless of what or whose actions caused them including ACTS of GOD.

5.1.5. A formal NCM is not required for routine minor instrument maintenance, malfunctions, and power failures which can be documented in instrument maintenance logbooks.

5.2. ***Corrective action:*** Measures taken to rectify conditions adverse to quality and, where possible, to prevent their reoccurrence.

5.2.1. Corrective actions may vary from reporting the data as is—with appropriate documentation—to a complete reevaluation and restructure of a system.

5.2.2. Many corrective actions can be implemented immediately; however, some will take time to implement.

6. MISCELLANEOUS

6.1. Associated Reference Documents

6.1.1. Quanterra® Quality Assurance Management Plan (QAMP), current revision.

6.1.2. ANSI/ASME NQA-1, Chapter II, Basic Requirement 15 "Control of Nonconforming Items." Supplement 15S-1 "Supplementary Requirements for the Control of Nonconforming Items."

6.1.3. ANSI/ASQC Q94-1987, "Quality Management and Quality System Elements - Guidelines," Section 14.0 "Nonconformity" and Section 15.0 "Corrective Action."

6.1.4. Clouseau Program Documentation.

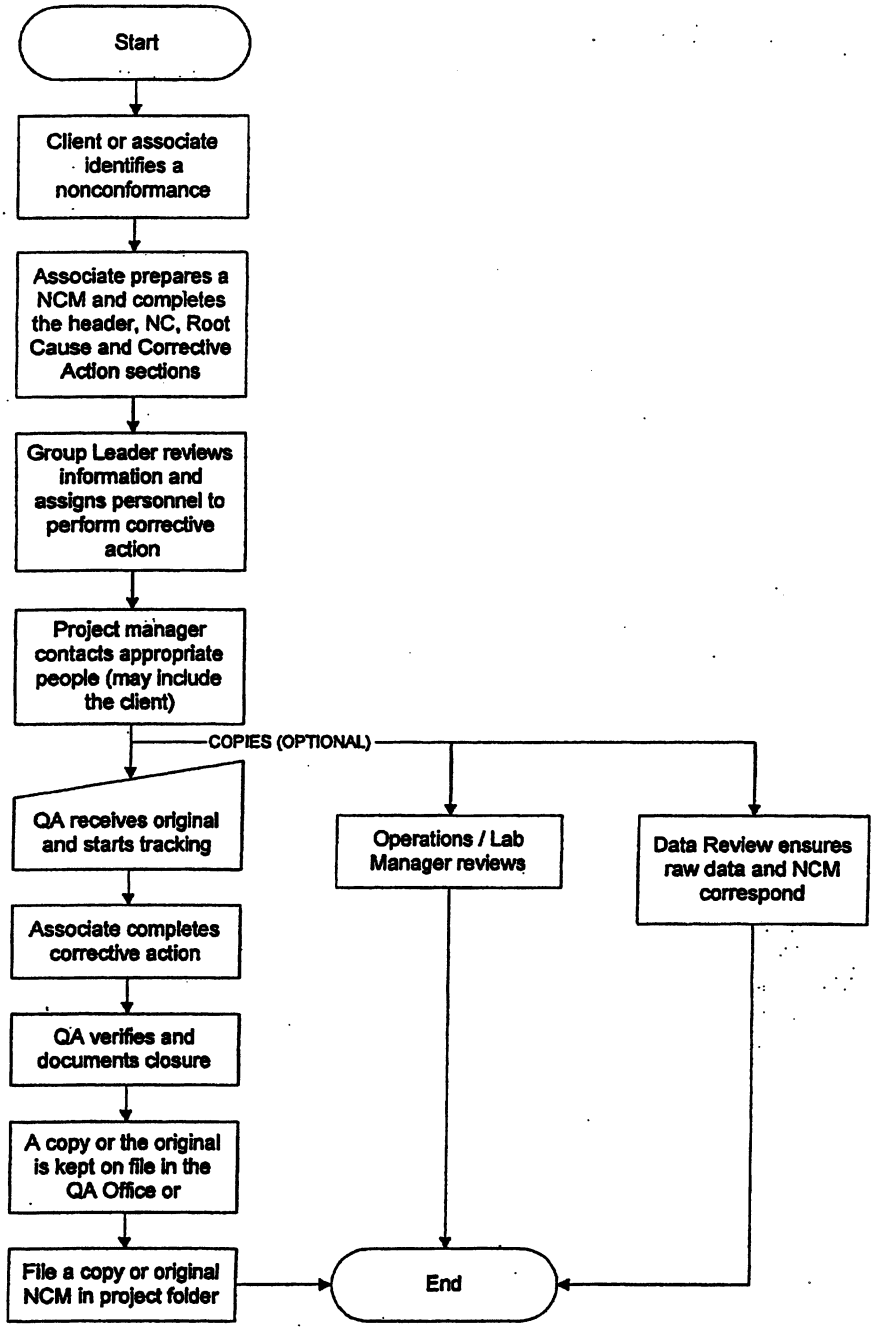
6.2. Appendices

6.2.1. Attachment A: Laboratory Nonconformance Memo (NCM).

6.2.2. Attachment B: Instrument/Equipment Nonconformance Tag Form

6.2.3. Attachment C: Nonconformance Memo Generated from Clouseau

6.3. Flow Chart for Internal NCM



ATTACHMENT A LOG #: _____

LABORATORY NONCONFORMANCE MEMO (NCM)

Quanterra® Incorporated

Project ID/Client: _____

NCM Initiated by/Date: _____ Project Manager: _____

Sample Numbers/QC batch or lot numbers _____

Tests: _____

Analytical Area (check appropriate area):

- ☐ Sample control
- ☐ Organic preparation
- ☐ Inorganic preparation
- ☐ GC
- ☐ HPLC
- ☐ GC/MS
- ☐ Wet chemistry
- ☐ Metals
- ☐ Reporting
- ☐ Data review
- ☐ Radiochemistry
- ☐ _____

Nonconformance (check appropriate area): *To be completed by analyst*

Holding Time Violations (exceeded by _____ days)

Category I: Laboratory Independent

- ☐ 1. Holding time expired in transit
- ☐ 2. Sample rec'd > 48 hrs after sampling. or ½ holding time has expired
- ☐ 3. Test added by client after expiration

Category II: Laboratory Dependent

- ☐ 4. Instrument failure
- ☐ 5. Analyst error
- ☐ 6. Log-in error
- ☐ 7. Miscommunication
- ☐ 8. Other (explanation required)_____

_____

Category III: Analysis Reruns (QA/QC)

- ☐ 9. Surrogates
- ☐ 10. Internal standards
- ☐ 11. Spike recoveries
- ☐ 12. Blank contamination

Category IV: Analysis Reruns (Confirmation)

- ☐ 13. Second column
- ☐ 14. Contamination check
- ☐ 15. Confirmation of matrix effects
- ☐ 16. Other (explanation required)_____

Category V: Analysis Reruns (Dilution)

- ☐ 17. Over calibration
- ☐ 18. Under calibration
- ☐ 19. Other (explanation required)_____

Quality Assurance/Quality Control

- ☐ 20. QC data reported outside of controls
- ☐ 21. Incorrect procedure used
- ☐ 22. SOP intentionally modified with QA and tech approval
- ☐ 23. Invalid instrument calibration
- ☐ 24. Received insufficient sample for proper analysis

Incorrect or Incomplete Client Deliverable

- ☐ 25. Hardcopy deliverable error
- ☐ 26. Electronic deliverable error

Reported Detection Limits Elevated Due to:

- ☐ 27. Sample matrix: Does not include high analyte content
- ☐ 28. Insufficient sample volume
- ☐ 29. Other (explanation required)

Miscellaneous

- ☐ 30. Instrument/equipment Tag-out
- ☐ 31.Other (explanation required)

_____

_____

_____

_____

Notification (check appropriate area): ☐ **Required** ☐ **Not Required** *To be completed by project manager*

Client notified by (name and date): _____

- ☐ In writing
- ☐ By facsimile
- ☐ By telephone
- ☐ Other (explain)

Client's name and response: _____

- ☐ Process "as is"
- ☐ Re-sample
- ☐ On hold until _____
- ☐ Other (explain)

Project manager (signature and date): _____

QUANTERRA® LABORATORY NONCONFORMANCE MEMO LOG #: _____

Corrective Action: *To be completed and reviewed by all associates involved*

Problem Description/Root Cause Author's initials and date: _____

Corrective Actions (Short Term) Author's initials and date: _____

Corrective Actions to Prevent Reoccurrence (Long Term) _____

Corrective Action approved by (Supervisor/Group Leader) and date: _____

Additional Comments: _____

Corrective Action to be completed by (if other than Supervisor/Group Leader): _____

Date Corrective Action is to be completed: _____

❑ Notified Ops/Sys Manager (Initials) _____

The Office of Quality Assurance maintains a copy or the original of this NCM indicating its final status.

ATTACHMENT B

EXAMPLE

INSTRUMENT/EQUIPMENT NONCONFORMANCE TAG FORM

QUANTERRA®

CAUTION

DO NOT USE

NONCONFORMING ITEM

NCM NUMBER _____

AFFECTED ITEM _____

_____

_____

_____ _____

ANALYST DATE

WORK MAY NOT PROCEED ON THIS ITEM UNTIL SUCCESSFUL CALIBRATION IS DOCUMENTED.

ATTACHMENT C

EXAMPLE

LABORATORY NONCONFORMANCE MEMO (GENERATED BY CLOUSEAU)

Quanterra

Clouseau

Nonconformance Memo

NCM #: E00053
NCM Initiated By: Kevin Freitag
Date Opened: 03/10/99
Date Closed: 03/18/99

Classification: Anomaly
Status: CLOSED
Production Area: Sample Control
Tests: 8260B
Lot #'s (Sample #'s): E9C100234 (9)
QC Batch: None.

Nonconformance: Sample receiving issues
Subcategory: Samples received did not match the COC

Problem Description / Root Cause

Name	Date	Description
Kevin Freitag	03/10/99	Sample was received but was not listed on COC. Sample was logged in as E9C100234-009.

Corrective Action

Name	Date	Corrective Action

Quality Assurance Verification

Verified By	Due Date	Status	Notes:
Sevdá Aleckson	N/A	Client approved, verification not required	

Client Notification Summary

Client	Project Manager	Date Notified	Response Date	How Notified
ARCADIS GERAGHTY & MILLER	Diane Suzuki	03/18/99	03/18/99	in writing

Response: No response saved
Response Details:

Approval History

Name	Date Approved:	Position
Kevin Freitag	03/10/99	Chemist
Linnea Coffey	03/18/99	Project Manager
Diane Suzuki	03/18/99	Project Manager
Sevdá Aleckson	03/18/99	Quality Assurance

Date Printed: 06/07/99

Page 1 of 1

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Implementation Date: 08/24/00

SOP No: NC-WC-0004
Revision No: 3
Revision Date: 08/04/00

STL STANDARD OPERATING PROCEDURE

TITLE: TOTAL SOLIDS, PERCENT MOISTURE, ASH AND TOTAL VOLATILE SOLIDS

(SUPERSEDES: REVISION 2.0)

Prepared by:		8-2-00 Date
Reviewed by:	Technology Specialist	8-7-00 Date
Approved by:	Quality Assurance Manager	8-4-00 Date
Approved by:	Environmental Health and Safety Coordinator	8-13-00 Date
Approved by:	Laboratory Director	8-17-00 Date

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Total Solids, Total Volatile Solids, Percent Moisture, and Ash in waters, wastewaters, sludges, and solids. It is based on EPA 160.3, EPA 160.4, ASTM D2216-90, ASTM D1553-83, SM 2540E, USEPA CLP Statements of Work OLM01.9, OLM03.1, ILM04.0 and ILM03.0. The approximate working range for Total Solids is 10 - 20,000 mg/L for waters and 0.5 to 100% for non-waters.

1.2. The applicable method codes are SM (160.3), AN (160.4) , NJ (D2216-90), ES (OLM03.1), DS (ILM03.0), WN (OLM01.9), OV (ILM04.0) and H9 (SM2540E). The associated preparation code is 88.

1.3. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

2. SUMMARY OF METHOD

2.1. A homogenous sample is dried at 104 °C ± 1°C and the difference in the weight loss of the sample (non-water) or the weight gain of the vessel (water) represents the Total Solids.

3. DEFINITIONS

3.1. Refer to the glossary in the Quality Assurance Management Plan (QAMP).

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Samples high in concentrations of minerals must be dried longer, desiccated, and weighed quickly to prevent any excess weight added due to absorption of water from the atmosphere.

4.3. Non-homogeneous samples may give erratic results.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Analytical balance: capable of accurately weighing ± 0.0001 g

6.2. Drying Oven

6.3. Desiccators

6.4. Evaporating dishes: various

6.5. Tongue blades

6.6. Graduated cylinders: various

6.7. Sample containers

6.8. Top loading balance: capable of accurately weighing ± 0.01 g

6.9. Analytical balance: capable of accurately weighing ±0.0001 g

6.10. Aluminum pans

6.11. Beakers: glass, various

6.12. Muffle furnace

6.13. Conductivity meter

6.14. Volumetric flasks: various

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Reagent water

7.2. Standards

7.2.1. Sodium Chloride (NaCl): reagent grade

7.2.2. Total Solid Standard, 2,500 mg/L: Add 2.5 g sodium chloride (NaCl) to a 1000 mL volumetric flask and dilute to volume with reagent water. Mix well. Replace after three months or as needed.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored in plastic or glass containers at 4°C ± 2°C.

8.3. The holding time is seven days from sampling to the completion of analysis for water samples.

8.4. There is no recommended holding time for non-water samples.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.2. Corrective Action for Blanks

9.2.2.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.2.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS) (Water Samples Only)

9.3.1. One aqueous LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. A LCS consisting of 25 mL of the 2500 mg/L TS standard is analyzed with each analytical batch of samples.

9.3.3. Corrective Action for LCS

9.3.3.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.3.2. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Duplicates

9.4.1. Sample duplicates are performed at a frequency of 10% and must meet laboratory-specific limits for precision.

10. CALIBRATION AND STANDARDIZATION

10.1. Not Applicable

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Vessel Preparation - Water Samples

11.3.1. Place glass beakers or evaporating dishes in the drying oven (103°C - 105°C) for at least one hour and in the desiccator for at least one hour.

11.4. Sample Analysis - Water Samples

11.4.1. Label and accurately weigh the beaker or evaporating dish on an analytical balance to the nearest 0.1 mg. Record weight on the analytical logsheet.

11.4.2. Tare the vessel and add approximately 100 mL of the homogenized sample. Record the volume of the sample on the analytical logsheet.

NOTE: If the sample is known to contain > 2000 mg/L dissolved solids, it should be diluted. A prescreening may be performed using a conductivity meter in which any results >2000 umhos should require a dilution.

11.5. To determine proper dilutions:

$$\frac{2000 \times 100}{Conductivity} = mL\ Sample\ to\ Use$$

11.5.1. Place the vessel with sample in the drying oven (103° - 105°C) overnight or until dry and then place the vessel in the desiccator for at least one hour.

11.5.2. If the sample was not dried overnight, place the dish in the oven for one hour, the desiccator for one hour, and reweigh. Continue this process until a constant weight is reached, until loss of weight is less than 4% of the previous weight, or 0.5 mg, whichever is less.

11.5.3. If a TVS or Ash is required, go to Section 11.7

11.6. Non-water Samples

11.6.1.. For liquid, sludge, TVS and ash samples, label and weigh an evaporating dish on a top loading balance. Record the weight on the analytical logsheet. For solid samples (TS), the tare weight is 5.652 For solid samples, sample receiving will divide the soil sample into a pre-tared container with a label. Weigh and record the wet weight on the analytical logsheet or the computer spreadsheet. For liquids and sludges, transfer approximately 50 mL to the tared evaporating dish then weigh and record the actual weight on the analytical logsheet.

11.6.2. Place dishes with sample in the drying oven (103° - 105°C) until dry (minimum of 12 hours). Document the time samples were placed in the oven on the logsheet or spreadsheet.

11.6.3. Remove the samples from the drying oven when they are dry. Document the time samples were removed from the oven on the logsheet or spreadsheet.

11.6.4. Place the dishes in the desiccator for at least one hour. Weigh the sample and dish on the top loading balance. Record the weight on the analytical logsheet.

NOTE: If the drying time was not overnight, it may be necessary to repeat the drying, desiccating, and weighing cycle until a constant weight is achieved as described in Section 11.5.2.

11.6.5. Method Blank for solid samples (TS), use a new container with a label. Weigh and record the weight on the analytical logsheet or computer spreadsheet.

11.6.6. Sample Duplicate for solid samples (TS), sample receiving will supply the sample duplicate. If the sample duplicate is not supplied from receiving, split some sample off from another container into a new container with a label. Weigh and record the weight on the analytical logsheet.

11.6.7. If a Total Volatile Solid or Ash is required, go to Section 11.7. All other samples may be properly disposed in the solid hazardous waste container.

11.7. Total Volatile Solids (TVS) and Ash Samples

11.7.1. Follow Section 11.6 for non-water samples

11.7.2. After the final dried weight has been taken, place the evaporating dish and dried sample in the muffle furnace. Muffle the sample for one hour at 550°C ± 50°C.

NOTE: Open the oven slowly and allow to cool for at least one hour before removing the samples.

11.7.3. Remove the dish with sample and place it in the desiccator for at least one hour, then weigh it on the top loading balance. Record the weight on the analytical logsheet or computer spreadsheet.

11.8. Percent Moisture

11.8.1. Follow Section 11.6

11.9. Analytical Documentation

11.9.1. Record all analytical information in the analytical logbook/logsheet or computer spreadsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.9.2. All standards are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.9.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.9.4. Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Total Solids

$$\textit{Total Solids, mg/L (waters)} = \frac{(A - B) \times 1000 \times 1000}{C}$$

Where: ***A =*** ***Final weight of dried sample and dish, g***

B = ***Initial weight of dish, g***

C = ***Sample volume, mL***

D = ***Sample weight, g***

$$\textit{Total Solids, \% (non-waters)} = \frac{(A - B) \times 100}{(C - B)}$$

Where: ***A = Final weight of dried sample and dish, g***

B = ***Initial weight of dish, g***

C = Inital weight of wet sample and dish, g

12.2. Ash

$$\textit{Ash, mg / L (waters)} = \frac{(D - B) \times 1000 \times 1000}{\textit{Sample Volume, mL}}$$

$$\textit{Ash, \% (non - waters)} = \frac{(D - B) \times 100}{A - B}$$

Where: ***A = Weight of dried sample and dish (after Total Solid), g***

B = Weight of dish only, g

C = Weight of wet sample and dish, g

D = Weight of muffled sample and dish, g

12.3. Total Volatile Solids (TVS)

$$\frac{(A - D) \times 1000 \times 1000}{\text{Sample Volume, mL}} = \text{TVS, mg / L (waters)}$$

$$\frac{(A - D) \times 100}{(A - B)} = \text{TVS, \% (non - waters)}$$

Where: *A = Weight of dried sample and dish (after Total Solid), g*

B = Weight of dish only, g

C = Weight of wet sample and dish, g

D = Weight of muffled sample and dish, g

12.4. LCS % Recovery

$$\text{LCS \% Recovery} = \frac{\text{mg / L}}{\text{2,500 (true)}} \times 100$$

12.5. Dry Weight

$$\text{Dry Weight} = \frac{\text{Sample Test Result} \times 100}{\text{(\%) Total Solid Results}} = \text{Dry Weight}$$

12.6. Percent Moisture

$$\text{\%Moisture} = 100 - \text{\%TS (Section 12.1)}$$

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Solid materials (gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.2. Refer to the Laboratory Sample and Waste Disposal plan.

15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1. References

16.1.1. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition, Solids, Method 2540E.

16.1.2. EPA 600, 1984, Total Residual Solids, Method 160.3

16.1.3. EPA 600, Total Volatile Residue, Method 160.4

16.1.4. Annual Book of ASTM Standards, Volume 04.08, 1990

16.1.5. USEPA Contract Laboratory Program, Statement of Work for Inorganics Analysis, Multi-media Multi-concentration, ILM02.1, ILM03.0, and ILM04.0

16.1.6. USEPA Contract Laboratory Program, Statement of Work for Organics Analysis, Multi-media Multi-concentration, OLM01.9 and OLM03.1

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Reporting limits

17.1.1. The lower reporting limit (RL) for Total Solids, TVS, and Ash is 10 mg/L for water and 0.5% for non-waters. The reporting limit for Method 160.3 is 10%.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

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Implementation Date: 06/05/01

SOP No. CORP-MS-0002NC
Revision No. 2.3
Revision Date: 05/23/01

STL STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF VOLATILE ORGANICS BY GC/MS BASED ON METHOD 8260B, 8260A, AND 624

(SUPERSEDES: REVISION 2.2, DATED 11/28/00)

Prepared by: Thomas E. Stallin 5/25/01
Date

Approved by: Laura Evans 5/25/01
Technology Specialist Date

Approved by: [signature] 5/25/01
Quality Assurance Manager Date

Approved by: [signature] 5-29-01
Environmental Health and Safety Coordinator Date

Approved by: [signature] 5-25-01
Lab Manager Date

Approved by: [signature] 6/4/01
Corporate Technology and/or Corporate Quality Assurance Date

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ANALYSIS OF VOLATILE ORGANICS BY METHOD 624 55

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1.0 SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Volatile Organic Compounds in waters, wastewater, soils, sludges and other solid matrices. Standard analytes are listed in Tables 5 and 6.

1.2. This SOP is applicable to method 8260B. It may also be used for analysis following method 8260A. Appendix A presents modifications to the procedures in the main SOP that are necessary for analysis of wastewater by method 624. The associated LIMS method codes are QK (8260B), DN (624), and MZ (8260A). Ohio VAP projects are distinguished by Program Code 2J. The following Prep Codes are used: 15 (5 mL purge), 25 (25 mL purge), 4B (Methanol preservation, EnCore™), 4D (Sodium Bisulfate preservation, EnCore™), 4P (Frozen, EnCore™), and 73 (5030A Methanol Prep).

1.3. This method can be used to quantify most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4. The method is based upon a purge and trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 5 to 200 µg/L for 5 mL waters, 1 to 40 µg/L for 25 mL purge waters, 5 to 200 µg/kg for low-level soils, and 250 to 25,000 µg/kg for medium-level soils. Reporting limits are listed in Tables 1 and 3.

1.5. Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates, and laboratory control spike samples.

2. SUMMARY OF METHOD

2.1. Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2. Aqueous samples are purged directly. Generally, soils are preserved by extracting the volatile analytes into methanol. If especially low detection limits are required, soil samples may be preserved with sodium bisulfate and purged directly.

2.3. In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature or at 40°C (40°C required for low level soils) and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbant

column where the volatile components are trapped. After purging is completed, the sorbant column (trap) is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components which are detected with a mass spectrometer.

2.4. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3. DEFINITIONS

3.1. Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. Using this method, each BFB analysis will normally start a new batch. Batches for medium level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort should be made to keep the samples together.

3.1.1. The Quality Control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. Refer to the STL QC Program document (QA-003) for further details of the batch definition.

3.2. Method Blank

3.2.1. A method blank consisting of all reagents added to the samples must be analyzed with each batch of samples. The method blank is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

3.3. Laboratory Control Sample (LCS)

3.3.1. Laboratory Control Samples are well characterized, laboratory generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

3.4. Surrogates

3.4.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Each sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

3.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

3.5.1. A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second aliquot of the same sample which is prepared and analyzed along with the sample and matrix spike. Matrix spikes and duplicates are used to evaluate accuracy and precision in the actual sample matrix.

3.6. Calibration Check Compound (CCC)

3.6.1. CCCs are a representative group of compounds which are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and % drift for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.7. System Performance Check Compounds (SPCC)

SPCCs are compounds which are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, pre-purged purified reagent water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants. In extreme cases the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

4.3. Matrix interferences may be caused by non-target contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.5. Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered an antifoaming agent (e.g., J.T. Baker's Antifoam B silicone emulsion) can be used. A blank spiked with this agent must be analyzed with the sample because of the non-target interferences associated with the agent.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

5.2. The Chemical Hygiene Plan (CHP) gives details about the specific health and safety practices which are to be followed in the laboratory area. Personnel must receive training in the CHP, including the written Hazard Communication plan, prior to working in the laboratory. Consult the CHP, the STL Health and Safety Policies and Procedures Manual, and available Material Safety Data Sheets (MSDS) prior to using the chemicals in the method.

5.3. Consult the STL Health and Safety Policies and Procedures Manual for information on Personal Protective Equipment. Eye protection that protects against splash and a laboratory coat must be worn in the lab. Appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately. Disposable gloves shall not be reused.

5.4. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined, therefore each chemical compound should be treated as a potential health hazard. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

5.4.1. Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include: Acrylonitrile, benzene, carbon tetrachloride, chloroform, 1,2-dibromo-3-chloropropane, 1,4-dichlorobenzene, and vinyl chloride.

5.4.2. Chemicals known to be flammable are: **Methanol.**

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor.

5.8. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the STL Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed by an annual refresher training.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes: 10 µL and larger, 0.006 inch ID needle.

6.2. Syringe: 5 or 25 mL glass with luerlok tip, if applicable to the purging device.

6.3. Balance: Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g

6.4. Glassware:

6.4.1. Vials: 20 mL with screw caps and Teflon liners.

6.4.2. Volumetric flasks: 10 mL and 100 mL, class A with ground-glass stoppers.

6.5. Spatula: Stainless steel.

6.6. Disposable pipets: Pasteur.

6.7. pH paper: Wide range.

6.8. Gases:

6.8.1. Helium: Ultra high purity, gr. 5, 99.999%.

6.8.2. Nitrogen: Ultra high purity, from cylinders of gas generators, may be used as an alternative to helium for purge gas.

6.8.3. Compressed air: Used for instrument pneumatics.

6.8.4. Liquid nitrogen: Used for cryogenic cooling if necessary.

6.9. Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.

6.9.1. Sample Purger: The recommended purging chamber is designed to accept 5 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low level soils are purged directly from a VOA vial.

6.9.2. Trap: A variety of traps may be used, depending on the target analytes required. For most purposes the Vocarb 3000 trap is suitable. Other traps, such as Vocarb 4000, or Tenax / Silica gel / Charcoal may be used if the Quality Control criteria are met.

6.9.3. Desorber: The desorber should be capable of rapidly heating the trap to 180°C. Many such devices are commercially available.

6.9.4. Sample Heater: A heater capable of maintaining the purge device at 40°C is necessary for low level soil analysis.

6.10. Gas Chromatograph/Mass Spectrometer System:

6.10.1. Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming.

6.10.2. Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:

6.10.2.1. Column 1: 105m x 0.53 ID Rtx-624 with 3 µm film thickness.

6.10.2.2. Column 2: 75 m x 0.53 ID DB-624 widebore with 3 µm film thickness.

6.10.2.3. Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 AMU every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-Bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.10.3. GC/MS interface: In general glass jet separators are used but any interface (including direct introduction to the mass spectrometer) that achieves all acceptance criteria may be used.

6.10.4. Data System: A computer system that allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as

the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

6.10.5. Cryogenic Cooling: Some columns require the use of liquid nitrogen to achieve the subambient temperature required for the proper separation of the gases.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Methanol: Purge and Trap Grade, High Purity

7.1.2. Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See section 9.4) Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable.

7.2. Standards

7.2.1. Calibration Standard

7.2.1.1. Stock Solutions: Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at -10° to -20°C.

7.2.1.2. Working standards: A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer or as recommended by the manufacturer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by more than 20% then corrective action is necessary. This may include steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem then a new initial calibration must be performed.

7.2.1.3. Aqueous Calibration Standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

7.2.1.4. If stock or secondary dilution standards are purchased in sealed ampoules they may be used up to the manufacturers expiration date.

7.2.2. Internal Standards: Internal standards are added to all samples, standards, and blank analyses. Refer to Table 7 for internal standard components.

7.2.3. Surrogate Standards: Refer to Table 8 for surrogate standard components and spiking levels.

7.2.4. Laboratory Control Sample Spiking Solutions: Refer to Table 9 for LCS components and spiking levels.

7.2.5. Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Table 9.

7.2.6. Tuning Standard: A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration of 25 ng/µL of 4-Bromofluorobenzene in methanol is prepared as described in Sections 7.2.1.1 and 7.2.1.2.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample collection.

8.2. Water samples are normally preserved at pH $\leq$ 2 with 1:1 hydrochloric acid. If residual chlorine is present, 2 drops of 10% sodium thiosulfate are added.

8.3. Solid samples are field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. At specific client request, unpreserved soil samples may be accepted.

8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take an EnCore™ sample. (The 5 g or 25 g sampler can be used, depending on client preference). Following shipment back to the lab the soil is preserved in methanol. This is the medium level procedure. If very low detection limits are needed (< 50 µg/kg for most analytes) then it will be necessary to use two additional 5 g EnCore™ samplers or to use field preservation.

8.5. Sample collection for medium level analysis using EnCore™ samplers.

8.5.1. Ship one 5 g (or 25 g) EnCore™ sampler per field sample position.

8.5.2. An additional bottle must be shipped for percent moisture determination.

8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25 g) sample into a tared VOA vial containing 5 mL methanol (25 mL methanol for the 25 g sampler). Obtain the weight of the soil added to the vial and note on the label.

8.5.4. Add the correct amount of surrogate spiking mixture. (Add 25 µL of 2500 µg/mL solution for a nominal 25 g sample, 5 µL for a nominal 5 g sample.) Refer to Section 17.8 for Michigan project criteria.

8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 500 µL of 50 µg/mL solution for a nominal 25 g sample, 100 µL for a nominal 5 g sample.) Reduce the volume of methanol added to ensure the final volume is 25 mL for nominal 25 g sample or 5 mL methanol for a nominal 5 g sample. Refer to Section 17.8 for Michigan project criteria.

8.5.6. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (50 µL of spike to 25 mL methanol or 10 µL spike to 5 mL methanol). Refer to Section 17.8 for Michigan project criteria.

8.5.7. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.5.8. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4 ±2°C until analysis.

8.6. Sample collection for medium level analysis using field methanol preservation

8.6.1. Prepare a 2 oz sample container by adding 25 mL purge and trap grade methanol. (If a 5 g sample is to be used, add 5 mL methanol to a 2 oz container or VOA vial).

8.6.2. Seal the bottle and attach a label.

8.6.3. Weigh the bottle to the nearest 0.01g and note the weight on the label.

8.6.4. Ship with appropriate sampling instructions.

8.6.5. Each sample will require an additional bottle with no preservative for percent moisture determination.

8.6.6. At client request, the methanol addition and weighing may also be performed in the field.

8.6.7. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.6.8. Add the correct amount of surrogate spiking mixture. (Add 25 µL of 2500 µg/mL solution for a nominal 25 g sample, 5 µL for a nominal 5 g sample.) Refer to Section 17.8 for Michigan project criteria.

8.6.9. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 25 µL of 50 µg/mL solution for a nominal 25 g sample, 100 µL for a nominal 5 g sample.) Reduce the volume of methanol added to ensure the final volume is 25 mL for nominal 25 g sample or 5 mL methanol for a nominal 5 g sample. Refer to Section 17.8 for Michigan project criteria.

8.6.10. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (500 µL of spike to 25 mL methanol or 100 µL spike to 5 mL methanol). Refer to Section 17.8 for Michigan project criteria.

8.6.11. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.6.12. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4+2°C until analysis.

8.7. Low level procedure

8.7.1. If low detection limits are required (typically < 50 µg/kg) sodium bisulfate preservation must be used. However, it is also necessary to take a sample for the medium level (field methanol preserved or using the EnCore™ sampler) procedure, in case the concentration of analytes in the soil is above the calibration range of the low level procedure.

8.7.2. A purge and trap autosampler capable of sampling from a sealed vial is required for analysis of samples collected using this method. (Varian Archon or O.I. 4552).

8.7.3. The soil sample is taken using a 5g EnCore™ sampling device and returned to the lab. It is recommended that two EnCore™ samplers be used for each field sample position, to allow for any reruns than may be necessary. A separate sample for % moisture determination is also necessary.

8.7.4. Prepare VOA vials by adding a magnetic stir bar, approximately 1 g of sodium bisulfate and 5 mL of reagent water.

8.7.5. Seal and label the vial. It is strongly recommended that the vial is labeled with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction. The label absolutely must not cover the neck of the vial or the autosampler will malfunction.

8.7.6. Weigh the vial to the nearest 0.1g and note the weight on the label.

8.7.7. Extrude the soil sample from the EnCore™ sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil and note on the label.

Note: Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case at a specific site, add 5 mL of water instead, and freeze at <-10°C within 48 hours, analyzed within 12 days after preserving with water, and stored at a 45 degree angle in the freezer.

Note: Freezing is not allowed for Ohio VAP soil samples.

8.7.9. Alternatively the sodium bisulfate preservation may be performed in the field. This is not recommended because of the many problems that can occur in the field setting. Ship at least two vials per sample. The field samplers must determine the weight of soil sampled. Each sample will require an additional bottle with no preservative for percent moisture determination, and an additional bottle preserved with methanol for the medium level procedure. Depending on the type of soil it may also be necessary to ship vials with no or extra preservative.

8.8. Unpreserved soils

8.8.1. At specific client request unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the lab. This is the old procedure based on method 5030A and method 8260A. It is no longer included in SW846 and is likely to generate results that are biased low, possibly be more than an order of magnitude.

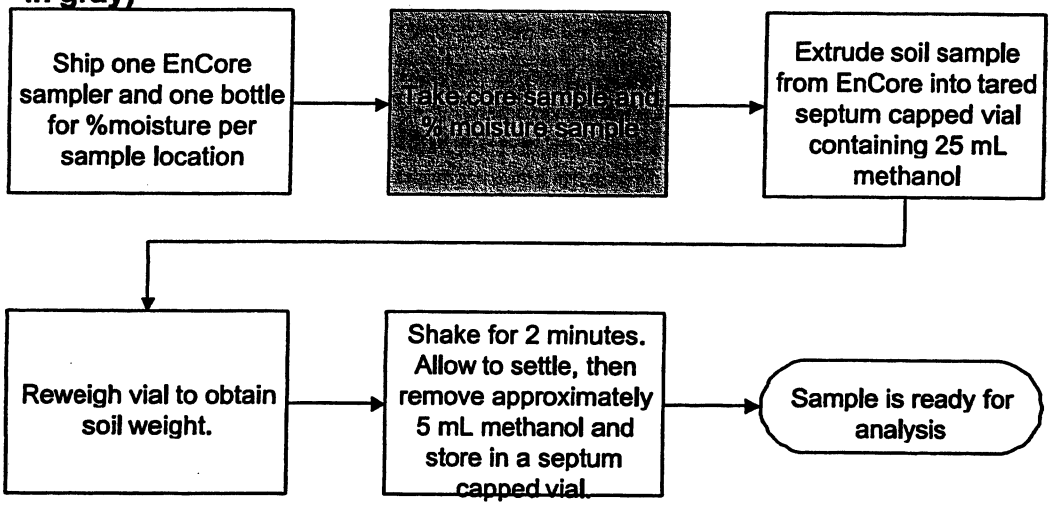
8.9. Aqueous samples are stored in glass containers with Teflon lined septa at 4°C +/- 2°C, with minimum headspace.

8.10. Medium level solid extracts are aliquoted into 2 - 5 mL glass vials with Teflon lined caps and stored at 4°C +/- 2°C. The extracts are stored with minimum headspace.

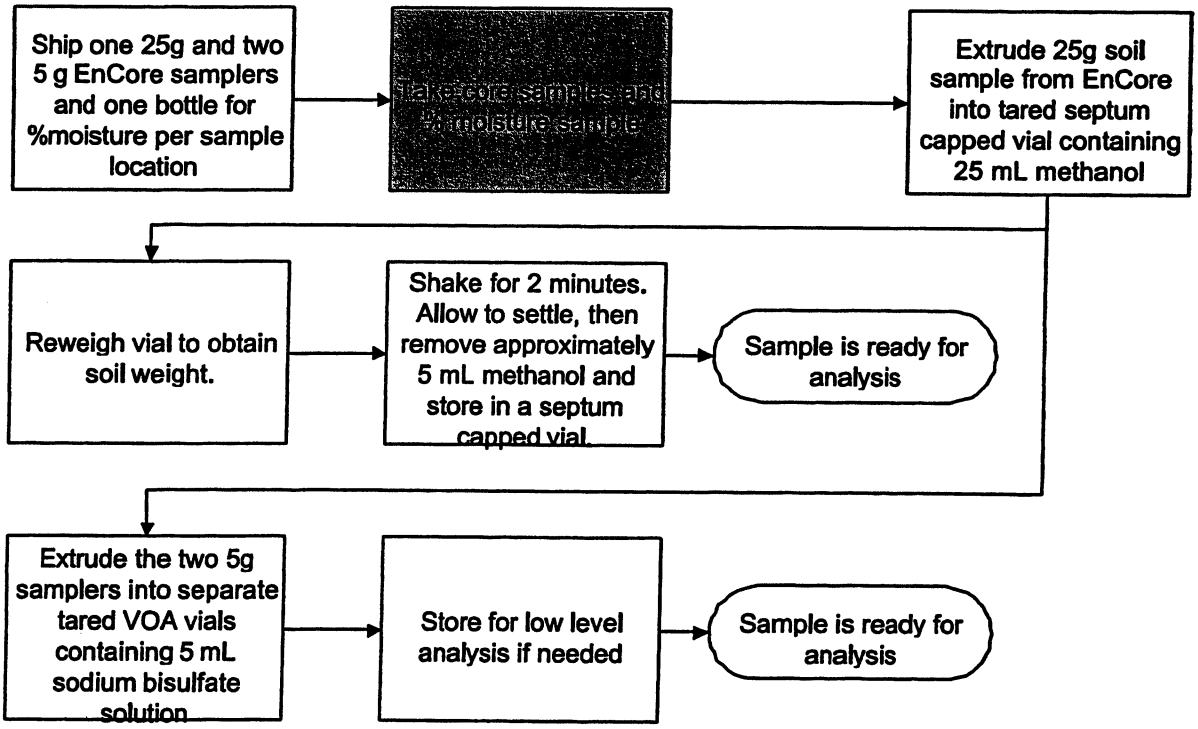
8.11. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14 day holding time. However they should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative). Maximum holding time for the EnCore™ sampler (before the sample is added to methanol or sodium bisulfate) is 48 hours.

8.12. A holding blank is stored with the samples. This is analyzed and replaced if any of the trip blanks show any contamination. Otherwise it is replaced every 14 days.

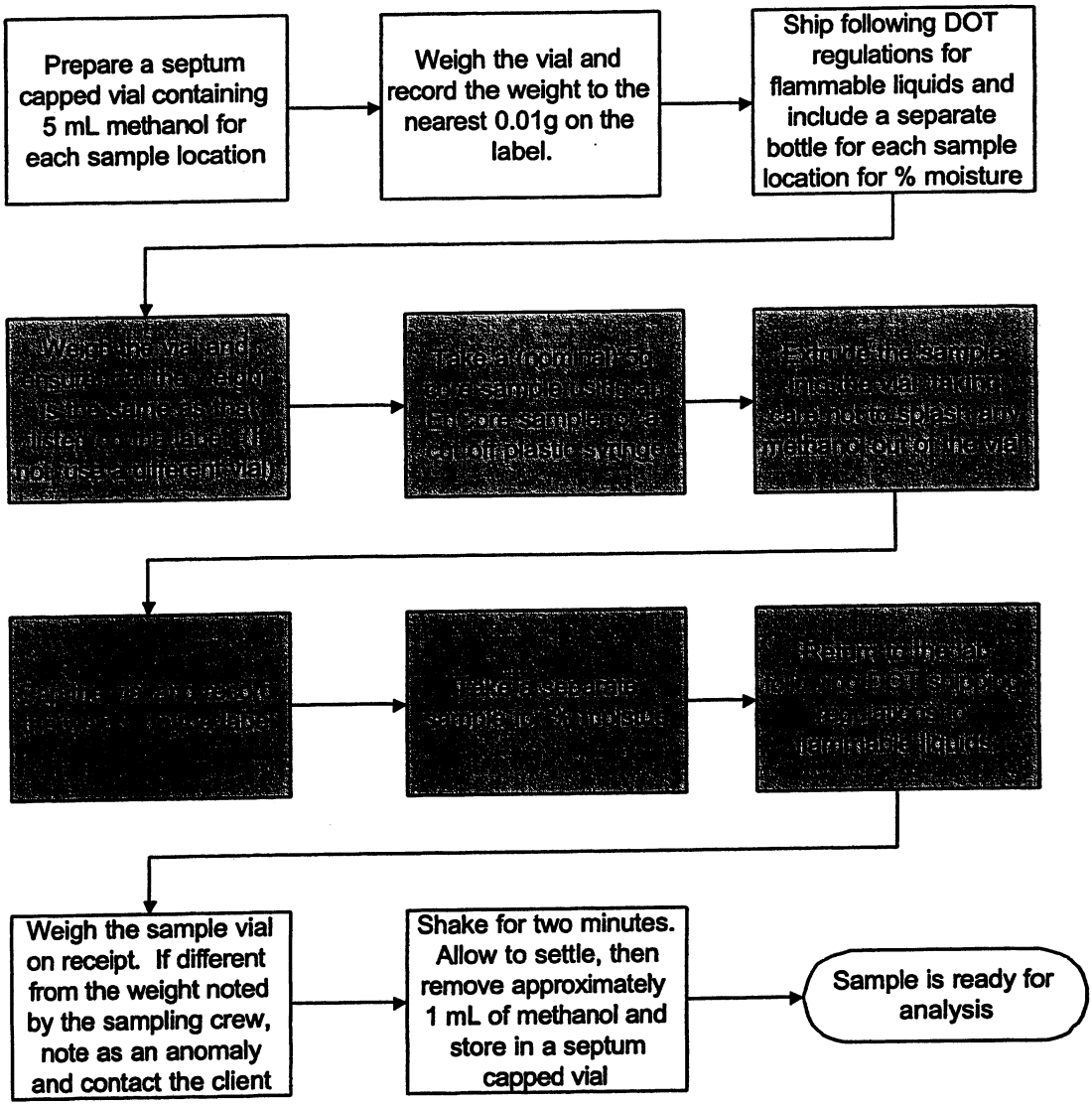
EnCore procedure when low level is not required (field steps in gray)



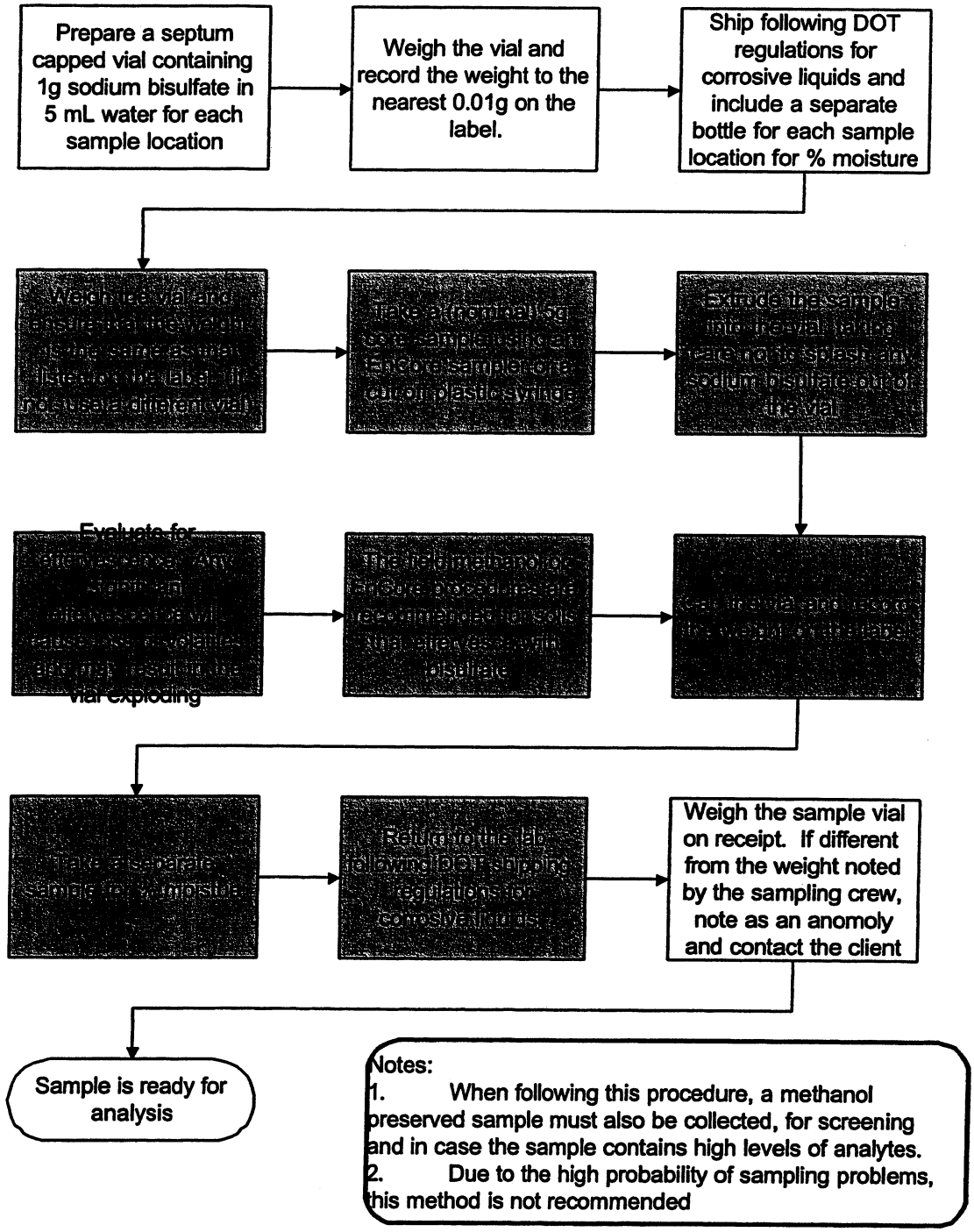
EnCore procedure when low level is required



Field methanol extraction procedure (field steps in gray)



Field bisulfate preservation procedure (field steps in gray)



9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration described in Section 13 and method detection limit (MDL) studies must be acceptable before analysis of samples may begin. MDLs should be analyzed for low and medium soils and aqueous samples.

9.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of a standard at the reporting limit and a single point calibration.

9.2. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery +/- 3 standard deviations for surrogates, matrix spikes and LCS. Precision limits for matrix spikes / matrix spike duplicates are 0 to mean relative percent difference + 3 standard deviations.

9.2.1. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.2. Refer to the QC Program document (QA-003) for further details of control limits.

9.3. Surrogates

Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Table 8. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.

- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.

- Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare/reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.3.1. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.

9.3.2. Refer to the STL QC Program document (QA-003) for further details of the corrective actions.

9.4. Method Blanks

9.4.1. For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. For low-level volatiles, the method blank consists of reagent water. For medium-level volatiles, the method blank consists of 25.0 mL of methanol. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

- If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

- Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be done in consultation with the client.

9.4.2. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the

purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.4.3. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B," and appropriate comments may be made in a narrative to provide further documentation.

9.4.4. Refer to the STL QC Program document (QA-003) for further details of the corrective actions.

9.5. Laboratory Control Samples (LCS)

9.5.1. For each batch of samples, analyze a LCS. The LCS is analyzed after the calibration standard, and normally before any samples. The LCS contains a representative subset of the analytes of interest (See Table 9), and must contain the same analytes as the matrix spike. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (Examples of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS.)

- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5.2. Refer to the STL QC Program document (QA-003) for further details of the corrective action.

9.5.3. If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be expected statistically. These requirements should be negotiated with the client. Refer to Section 17.5 for Ohio VAP specific analytes.

9.6. Matrix Spikes

9.6.1. For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 9. Compare the percent recovery and relative percent difference

(RPD) to that in the laboratory specific historically generated limits. See Section 17.5 for Ohio VAP specific analytes.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

- If the recovery for any component is outside QC limits for both the matrix spike/ spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include reanalysis of the batch.

- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.

- The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7. Nonconformance and Corrective Action

9.7.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.8. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.9. STL QC Program

Further details of QC and corrective action guidelines are presented in the STL QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. Prior to the analysis of samples and blanks, each GC/MS system must be tuned and calibrated. Hardware tuning is checked through the analysis of the 4-Bromofluorobenzene (BFB) to establish that a given GC/MS system meets the standard mass spectral abundance criteria. The

GC/MS system must be calibrated initially at a minimum of five concentrations (analyzed under the same BFB tune), to determine the linearity of the response utilizing target calibration standards. Once the system has been calibrated, the calibration must be verified each twelve hour time period for each GC/MS system.

10.2.1. General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 AMU
Scan Time:	to give at least 5 scans/peak, but not to exceed 2 second/scan
Injector Temperature:	200–250°C
Source Temperature:	According to manufacturer's specifications
Transfer Line	Temperature: 250–300°C
Purge Flow:	40 mL/minute
Carrier Gas	Flow: 15 mL/minute
Make-up Gas Flow:	25–30 mL/minute

10.2.2. Gas chromatograph suggested temperature program

10.2.2.1. BFB Analysis

Isothermal:	170°C

10.2.2.2. Sample Analysis

Initial Temperature:	40°C
Initial Hold Time:	4 minutes
Temperature Program:	8°C/minute
Final Temperature:	184°C
Second Temperature	Program: 40°C/minute
Final Temperature:	240°C
Final Hold Time:	2.6 minutes

10.3. Instrument Tuning

10.3.1. Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 10 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB.

10.4. Initial Calibration

10.4.1. A series of five initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Six standards must be used for a quadratic least squares calibration. Suggested calibration levels for a 5 mL purge are: 5, 20, 50, 100, and 200 µg/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Suggested calibration levels for a 25 mL purge are 1, 5, 10, 20, and 40 µg/L. Again, some analytes are prepared at higher levels. Tables 2 and 4 list the calibration levels for each analyte. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument. (For example, adequate sensitivity can be obtained on the Agilent 5973 instruments to use a 5 mL purge volume to reach the same reporting limits that once required a 25 mL purge. The calibration levels will still be the same 1, 5, 10, 20, 40µg/L.) However, the same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

10.4.2. It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for same tests. For example, the Appendix IX list requires the Primary standard (Table 5) and the Appendix IX standard (Table 6). If acceptable analytical performance can be obtained the primary and appendix IX standards may be analyzed together.

10.4.3. Internal standard calibration is used. The internal standards are listed in Table 7. Target compounds should reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See equation 1, Section 12, for calculation of response factor.

10.4.4. The % RSD of the calibration check compounds (CCC) must be less than 30%. Refer to Table 12 for the CCCs.

10.4.4.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.4.5. The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 11 for the SPCC compounds and required minimum response factors.

10.4.6. If the average of all the %RSDs in the calibration is $\leq$ 15%, then all analytes may use average response factor for calibration.

10.4.6.1. If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve then the appropriate curve should be used for quantitation. If Relative Standard Error (RSE) is used to evaluate the curve it must be better than 15%. Otherwise the correlation coefficient (coefficient of determination for non-linear curves) must be $\geq$ 0.990.

10.4.6.2. If the average of all the %RSDs in the calibration is > 15% then calibration on a curve must be used for all analytes with %RSD > 15%. Linear or quadratic curve fits may be used. The analyst should consider instrument maintenance to improve the linearity of response. If Relative Standard Error (RSE) is used to evaluate the curve it must be better than 15%. If the % RSD is > 15%, the analyst may drop the low or high in the ICAL , as long as a minimum of 5 points are maintained and the quantitation range is adjusted accordingly. Otherwise the correlation coefficient, (coefficient of determination, r^2 for non-linear curves) must be $\geq$ 0.990. If the correlation coefficient is < 0.990, then any hit for these compounds must be flagged as estimated.

10.4.6.3. Refer to Section 17.5 for specific Ohio VAP criteria.

10.4.7. Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called 1/X^2 weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4.8. If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.9. The calibration standards for the initial 5-point calibration for low level soils that are not preserved in sodium bisulfate (i.e. are preserved by freezing, or not preserved) must be heated to 40°C for purging. Using this calibration curve for water samples is acceptable as long as all calibration, QC, and samples are also heated to 40°C. A separate five point calibration must be prepared for analysis of low level soils that are preserved with sodium bisulfate. Low level soils analysis requires the use of a closed vial autosampler such as the Varian Archon, O.I. 4552 or Tekmar Precept. Each standard for analysis of sodium

bisulfate preserved samples is prepared by spiking the methanolic standard solution through the septum of a VOA vial containing 5 mL of water and 1 g sodium bisulfate. The standards are heated to 40°C for purging. All low-level soil samples, standards, and blanks must also be heated to 40°C for purging. Medium soil extracts should be analyzed using the water (unheated or optionally heated) calibration curve as long as all calibration standards, samples, and QC samples are purged at the same temperature.

10.4.10. Non-standard analytes are sometimes requested. For these analytes, it is acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. If the analyte is detected in any of the samples, a five point initial calibration must be generated and the sample(s) reanalyzed for quantitation. However, if the analyte is not detected, the non-detect may be reported and no further action is necessary.

Note: This procedure is may not be used for Ohio VAP samples.

10.5. Continuing Calibration: The initial calibration must be verified every twelve hours.

10.5.1. Continuing calibration begins with analysis of BFB as described in Section 10.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. The level 3 calibration standard is used as the continuing calibration.

10.5.2. The RF data from the standards are compared with the average RF from the initial five-point calibration to determine the percent drift of the CCC compounds. The calculation is given in equation 4, Section 12.3.4.

10.5.3. The % drift of the CCCs must be $\leq$ 20% for the continuing calibration to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 11. In addition, the % drift of all analytes must be $\leq$ 50% with allowance for up to six target analytes to have % drift > 50%.

10.5.3.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.5.3.2. Cyclohexanone, one of the components of the Appendix IX standard, is unstable in the calibration solution, forming 1,1-dimethoxycyclohexane. No calibration criteria are applied to cyclohexanone and quantitation is tentative. Cyclohexanone is included on the Universal Treatment Standard and FO-39 regulatory lists (but not on Appendix IX).

10.5.3.3. Refer to Table 12 for specific Ohio VAP analytes.

10.5.4. If the CCCs and or the SPCCs do not meet the criteria in Sections 10.5.3 and 10.5.4, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis begins. Extensive corrective action such as a different type of column will require a new initial calibration.

10.5.5. Once the above criteria have been met, sample analysis may begin. **Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs.** Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample *desorbed* less than or equal to 12 hours after the BFB is acceptable.)

11. PROCEDURE

11.1. Procedural Variations

11.1.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation shall be completely documented using a Nonconformance Memo and approved by a Supervisor or group leader and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.1.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.2. Preliminary Evaluation

11.2.1. Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories.

11.2.2. Dilutions should be done just prior to the GC/MS analysis of the sample. Dilutions are made in volumetric flasks or in a Luerlok syringe. Calculate the volume of reagent water required for the dilution. Fill the syringe with reagent water, compress the water to vent any residual air and adjust the water volume to the desired amount. Adjust the plunger to the mark and inject the proper aliquot of sample into the syringe. If the dilution required would use less than 1 µL of sample then serial dilutions must be made in volumetric flasks.

11.2.2.1. The diluted concentration is to be estimated to be in the upper half of the calibration range.

11.3. Sample Analysis Procedure

11.3.1. All analysis conditions for samples must be the same as for the continuing calibration standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.3.2. All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain a MS/MSD, a LCS, and a method blank.

11.3.2.1. If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next tune period. However, if any re-tuning of the instrument is necessary, or if a period of greater than 24 hours from the preceding BFB tune has passed, a new batch must be started. For medium level soils the batch is defined at the sample preparation stage.

11.3.2.2. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

11.3.2.3. It is not necessary to reanalyze batch QC with reanalyses of samples. However, any reruns must be as part of a valid batch.

11.4. Water Samples

11.4.1. All samples and standard solutions must be at ambient temperature before analysis.

11.4.2. Fill a syringe with the sample. If a dilution is necessary it may be made in the syringe if the sample aliquot is $\geq$ 5 µL. Check and document the pH of the remaining sample.

11.4.3. Add 250 ng of each internal and surrogate standard (10 µL of a 25 µg/mL solution, refer to Tables 7, 8 and 16). The internal standards and the surrogate standards may be mixed and added as one spiking solution (this results in a 50 µg/L solution for a 5 mL sample, and a 10 µg/L solution for a 25 mL sample). Inject the sample into the purging chamber.

11.4.3.1. For TCLP samples use 0.5 mL of TCLP leachate with 4.5 mL reagent water and spike with 10 µL of the 25 µg/mL TCLP spiking solution. (Note that TCLP reporting limits will be 10 times higher than the corresponding aqueous limits).

11.4.4. Purge the sample for eleven minutes (the trap must be below 35°C).

11.4.5. After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for approximately 3-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.4.6. Desorb and bake time and temperature are optimized for the type of trap in use. The same conditions must be used for samples and standards.

11.5. Methanol Extract Soils

11.5.1. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe. Refer to Section 17.8 for Michigan project requirements.

11.6. Liquid wastes that are soluble in methanol and insoluble in water.

11.6.1. Pipet 2 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1 gram.

11.6.2. Quickly add 7 mL of methanol, then add 1 mL of surrogate spiking solution to bring the final volume to 10 mL. Cap the vial and shake for 2 minutes to mix thoroughly. For a MS/MSD or LCS, 6 mL of methanol, 1 mL of surrogate solution, and 1 mL of matrix spike solution is used.

11.6.3. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe.

11.7. Aqueous and Low level Soil Sample Analysis (Purge and Trap units that sample directly from the VOA vial)

11.7.1. Units which sample from the VOA vial should be equipped with a module which automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

11.7.2. If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise the internal and surrogate standards must be added to the vial. *Note:* Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to analyzed as soils.

11.7.3. Soil samples must be quantitated against a curve prepared with standards containing about the same amount of sodium bisulfate as the samples (1 g in 5 mL).

11.7.4. Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.7.5. For aqueous samples, check the pH of the sample remaining in the VOA vial after analysis is completed.

11.8. Low-Level Solids Analysis using discrete autosamplers, Method 8260A, 5030A.

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

This method is based on purging a heated soil/sediment sample mixed with reagent water containing the surrogates and internal standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.8.1. Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

11.8.2. Weigh out 5 g (or other appropriate aliquot) of sample into a disposable culture tube or other purge vessel. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 1.0 g. If the sample is contaminated with analytes such that a purge amount less than 1.0 g is appropriate, use the medium level method. For the medium level method, add 4g soil to 10 mL methanol containing the surrogates, mix for two minutes, allow to settle then remove a portion of the methanol and store in a clean Teflon capped vial at 4°C until analysis. Analyze as described in section 11.5.

11.8.3. Connect the purge vessel to the purge and trap device.

11.8.4. Rinse a 5 mL gas-tight syringe with organic free water, and fill. Compress to 5 mL. Add surrogate/internal standard (and matrix spike solutions if required.). Add directly to the sample from 11.5.2.

11.8.5. The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

11.8.6. Add the heater jacket or other heating device and start the purge and trap unit.

11.8.7. Soil samples that have low IS recovery when analyzed (<50%) should be reanalyzed once to confirm matrix effect.

11.9. Medium-Level Soil/Sediment and Waste Samples

11.9.5. Sediments/soils and waste that are insoluble in methanol.

11.9.5.1. Sediments/soils and waste that are insoluble in methanol.

11.9.5.1.1. Gently mix the contents of the sample container with a narrow metal or wood spatula. Weigh 4 g (wet weight) into a tared vial. Use a top-loading balance. Record the weight to 0.1 gram. Do not discard any supernatant liquids.

11.9.5.1.2. Quickly add 9 mL of methanol, and 1 mL of surrogate spiking solution to bring the final volume of methanol to 10 mL. For an LCS or MS/MSD sample add 8 mL of methanol, 1 mL of surrogate spike solution, and 1 mL of matrix spike solution. Cap the vial and vortex to mix thoroughly.

NOTE: Sections 11.9.5.1.1 and 11.9.5.1.2 must be performed rapidly and without interruption to avoid the loss of volatile organics.

11.10. Initial review and corrective actions

11.10.1. If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minutes from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.10.2. If the internal standard response in the continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.10.2.1. Any samples that do not meet the internal standard criteria for the continuing calibration must be evaluated for validity. If the change in sensitivity is a matrix effect confined to an individual sample reanalysis is not necessary. If the change in sensitivity is

due to instrumental problems all affected samples must be reanalyzed after the problem is corrected.

11.10.3. The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis may not be necessary. Out of control surrogate standard response may be a matrix effect. It is only necessary to reanalyze a sample once to demonstrate matrix effect, but reanalysis at a dilution should be considered.

11.11. Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.10.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgement.

11.10.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component

characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)

12.1.1. If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

12.2. Tentatively Identified Compounds (TICs)

12.2.1. If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. Guidelines are:

12.2.1.1. Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

12.2.1.2. The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

12.2.1.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.1.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

12.2.1.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the spectrum because of background contamination or coeluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

12.2.1.6. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst assign a tentative identification.

12.3. Calculations.

12.3.1. Response factor (RF):

Equation 1

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_{is} = Concentration of the specific internal standard, ng

C_x = Concentration of the compound being measured, ng

12.3.2. Standard deviation (SD):

Equation 2

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(Xi - X)^2}{N - 1}}$$

X_i = Value of X at i through N

N = Number of points

X = Average value of X_i

12.3.3. Percent relative standard deviation (%RSD):

Equation 3

$$\%RSD = \frac{\text{Standard Deviation}}{\overline{RF_i}} \times 100$$

$\overline{RF_i}$ = Mean of RF values in the curve

12.3.4. Percent drift between the initial calibration and the continuing calibration:

Equation 4

$$\% \text{ Drift} = \frac{C_{expected} - C_{found}}{C_{expected}} \times 100$$

Where

$C_{expected}$ = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.3.5. Target compound and surrogate concentrations:

Concentrations in the sample may be determined from linear or second order (quadratic) curve fitted to the initial calibration points, or from the average response factor of the initial calibration points. Average response factor may only be used when the % RSD of the response factors in the initial calibration is $\leq$ 15%.

12.3.5.1. Calculation of concentration using Average Response Factors

Equation 5

$$\text{Concentration } \mu g / L = \frac{x}{RF}$$

12.3.5.2. Calculation of concentration using Linear fit

Equation 6

$$\text{Concentration } \mu g / L = A + Bx$$

12.3.5.3. Calculation of concentration using Quadratic fit

Equation 7

$$\text{Concentration } \mu g / L = A + Bx + Cx^2$$

x is defined in equations 8, 9 and 10

A is a constant defined by the intercept

B is the slope of the curve

C is the curvature

12.3.5.4. Calculation of x for Water and water-miscible waste:

Equation 8

$$x = \frac{(A_x)(I_s)(D_f)}{(A_{is})(V_o)}$$

Where:

A_x = Area of characteristic ion for the compound being measured (secondary ion quantitation is allowed only when there are sample interferences with the primary ion)

A_{is} = Area of the characteristic ion for the internal standard

I_s = Amount of internal standard added in ng

$$\text{Dilution Factor} = D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

V_o = Volume of water purged, mL

12.3.5.5. Calculation of x for Medium level soils:

Equation 9

$$x = \frac{(A_x)(I_s)(V_t)(1000)(D_f)}{(A_{is})(V_a)(W_s)(D)}$$

Where:

A_x, I_s, D_f, A_{is}, same as for water.

V_t = Volume of total extract, mL (Typically 25 mL)

V_a = Volume of extract added for purging, µL

W_s = Weight of sample extracted, g

$$D = \frac{100 - \%\text{moisture}}{100}$$

12.3.5.6. Calculation of x for Low level soils:

Equation 10

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$

Where:

A_x, I_s, A_{is}, same as for water.

D is as for medium level soils

W_s = Weight of sample added to the purge vessel, g

12.3.5.7. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x = Area in the total ion chromatogram for the compound being measured

A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

In other words, the concentration is equal to x as defined in equations 8, 9 and 10.

12.3.6. MS/MSD Recovery

Equation 11

$$\textbf{Matrix Spike Recovery, \%} = \frac{SSR - SR}{SA} \times 100$$

SSR = Spike sample result
SR = Sample result
SA = Spike added

12.3.7. Relative % Difference calculation for the MS/MSD

Equation 12

$$RPD = \frac{|MSR - MSDR|}{\frac{1}{2}(MSR + MSDR)} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result

MSDR = Matrix spike duplicate result

13. METHOD PERFORMANCE

13.1. Method Detection Limit

13.2. Generally, each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: QA-005. When non-standard compounds are analyzed at client request, lesser requirements are possible with client agreement. At a minimum, a standard at the reporting limit must be analyzed to demonstrate the capability of the method.

13.3. Initial Demonstration

13.4. Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest. The QC check sample is made up at 20 µg/L. (Some compounds will be at higher levels, refer to the calibration standard levels for guidance.)

13.4.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.4.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. The %RSD should be $\leq$ 15% for each analyte, and the % recovery should be within 80-120%.

13.4.3. If any analyte does not meet the acceptance criteria, check the acceptance limits in the reference methods (Table 6 of method 8240B, paragraph 8.3.5 of method 8260A). If the recovery or precision is outside the limits in the reference methods, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.4.4. Training Qualification

13.4.4.1. The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260B, Update III, December 1996

16.2. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260A, Update II, September 1994.

17. MISCELLANEOUS

17.1. Modifications from the reference method

17.1.1. Ion 119 is used as the quantitation ion for chlorobenzene-d5 for 25 mL purge tests.

17.1.2. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.3. The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.1.4. Method 8260A recommends that the purge vessel is run through an additional purge cycle after 25 mL sample analysis to remove carryover. Instead, purge vessels are oven baked between analyses or disposable vessels are used one time only.

17.1.5. SW-846 recommends that a curve be used for any analytes with %RSD of the response factors > 15%. However, some industry standard data systems and forms generation software cannot report this data with the necessary information for data validation. In

addition most software available does not allow weighting of the curve. Unweighted curves may exhibit serious errors in quantitation at the low end, resulting in possible false positives or false negatives. Therefore, this SOP allows used of average response factors if the average %RSD for all compounds is ≤ 15%.

17.2. Modifications from previous revision

This SOP has been substantially revised to reflect the changes included in Update III to SW-846. Directions for method 524.2 and method 624 have also been added.

17.3. Facility specific SOPs

Each facility shall attach a list of facility-specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4. Flow diagrams

17.4.1. Initial Demonstration and MDL

17.5. The following are protocols that must be followed when analyzing OhioVAP samples:

- Sections 9.5 and 9.6: n-Hexane must be spiked and reported for both the LCS and MS/MSD.
- Sections 10.4.6: All analytes must have a %RSDs ≤ 15%. Corrective action must be completed for any compounds failing the <15% requirement.
- Section 11.1 and 17.1.5 (Method deviations) are not to be performed.
- Section 11.9.2: For OhioVAP projects, the laboratory will reanalyze any sample where the internal standard fails and there is no evidence of matrix interference.

17.6. The following are protocols that must be followed when analyzing BP Oil – Lima Refinery RFI work plan.

- Section 8.1 STL will continue to follow the 14 day holding time specified in the Corporate SOP.
- Delete for this project Section 8.3 At specific client request, unpreserved soil samples may be accepted.

- Delete for this project Section 8.8.1 At specific client request unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the lab. This is the old procedure based on method 5030A. It is no longer included and is likely to generate results that are biased low, possibly by more than an order of magnitude.

- Modify Section 8.5.8 For the purpose of this project, the soil/methanol mixture may be stored for two days prior to analysis.

- Modify Section 8.6.12 For the purpose of this project, the soil/methanol mixture may be stored for two days prior to analysis.

- Modify (per discussion with Region V representative) to Section 10.4.6.2 Compounds with %RSD >15% are to be calibrated using an alternate calibration technique (e.g. linear or quadratic calibration curve). For poor responders, the alternate calibration technique requirements may not be met either. This sentence is added for those cases.If the correlation coefficient is < 0.990, then any hit for these compounds must be flagged as estimated.

- Modify Section 10.4.2 It is necessary to analyze the Appendix IX standard separately from the primary standard due to the presence of xylene solvent in the Appendix IX standard. Alternatively, STL will purchase the Appendix IX standard in a solvent other than xylene.

- Modify Section 10.4.9 For this project, this section will be modified to comply with the requirement of adding methanol to the calibration standards so that those standards contain the same amount of methanol as the diluted soil extracts.

- Modify Table 6

- For the project specific SOP, acetonitrile will be removed from table 6, page 49 and appended onto table 5, page 48. Acetonitrile will be calibrated as part of the STL primary standard, using a separate acetonitrile standard. This will ensure that the calibration curve for acetonitrile will be done free from any interference from allyl chloride.

17.7. The following are protocols that must be followed when analyzing South Carolina Projects only.

- **Delete** from Section 10.4.7 In a linear *or quadratic* calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve.

- **Delete** from Section 12.3.5 Concentrations in the sample may be determined from linear *or second order (quadratic)* curve fitted to the initial calibration points, or from the average response factor of the initial calibration points.

- **Delete** from Section 12.3.5.1

- Calculation of concentration using Quadratic fit

Equation 13

Concentration $\mu g / L = A + Bx + Cx^2$

x is defined in equations 8, 9 and 10

A is a constant defined by the intercept

B is the slope of the curve

C is the curvature

- **Change** Section 9.3 The compounds included in the surrogate spiking solutions are listed in Tables 8 *and 9*.

17.8. The following are protocols that must be followed to achieve the lower reporting limits required when analyzing Michigan projects.

17.8.1. Modify Section 8.5.4 and 8.6.8 (add 5 uL of 2500 ug/mL surrogate solution for a nominal 25 g sample).

17.8.2. Modify Section 8.5.5 and 8.6.9 (add 100 uL of 50 ug/mL spike solution for a nominal 25 g sample).

17.8.3. Modify Section 8.5.6 and 8.6.10 (add 100 uL of 50 ug/mL spike solution for a nominal 25g sample).

17.8.4. Michigan reporting limits for methanol preserved soils are achieved by injecting 100 uL of the methanol extract in a 5 mL purge. The instrument is calibrated using the recommended calibration levels in water of 1 ug/L, 2 ug/L (if a quadratic calibration is to be used), 5 ug/L, 10 ug/L, 20 ug/L and 40 ug/L. Some analytes are prepared at higher concentrations.

17.8.5. Samples for Michigan projects frequently require calibration for 2-Methylnaphthalene. Recommended calibration levels for this compounds are 2 ug/L, 10 ug/L, 20 ug/L, 40 ug/L and 80 ug/L.

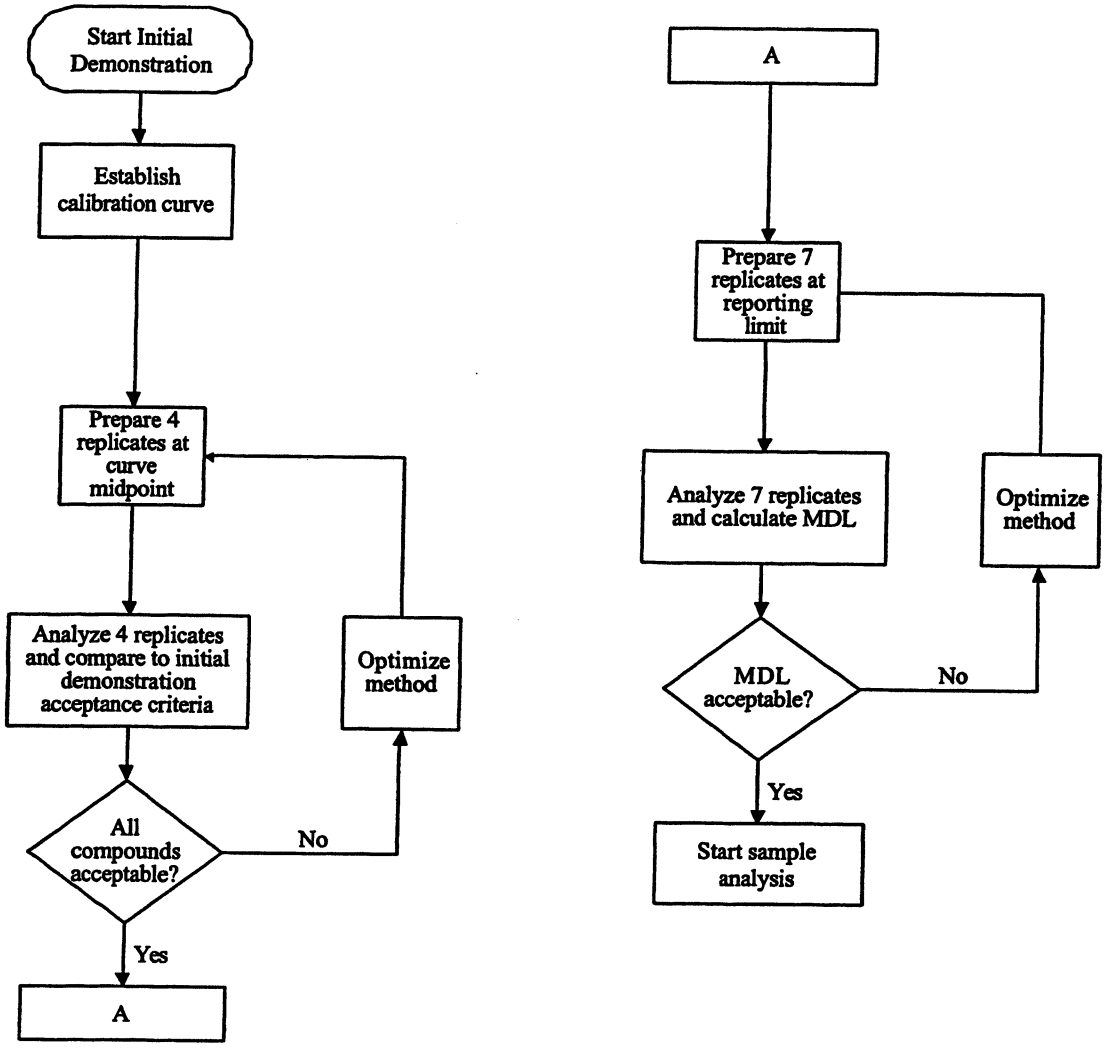

Start Initial Demonstration
Establish calibration curve
Prepare 4 replicates at curve midpoint
Analyze 4 replicates and compare to initial demonstration acceptance criteria
Optimize method
All compounds acceptable?
No
Yes
A
A
Prepare 7 replicates at reporting limit
Analyze 7 replicates and calculate MDL
Optimize method
MDL acceptable?
No
Yes
Start sample analysis

Table 1

STL Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	25 mL[3] water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
Dichlorodifluoromethane	75-71-8	10	2	10	250	1200
Chloromethane	74-87-3	10	2	10	250	1200
Bromomethane	74-83-9	10	2	10	250	1200
Vinyl chloride	75-01-4	10	2	10	250	1200
Chloroethane	75-00-3	10	2	10	250	1200
Trichlorofluoromethane	75-69-4	10	2	10	250	1200
Acrolein	107-02-8	100	20	100	5000	12000
Acetone	67-64-1	20	10	20	1000	2500
Trichlorotrifluoroethane	76-13-1	5	1	5	250	620
Iodomethane	74-88-4	5	1	5	250	620
Carbon disulfide	75-15-0	5	1	5	250	620
Methylene chloride	75-09-2	5	1	5	250	620
tert-Butyl alcohol	75-65-0	200	50	200	10,000	25000
1,1-Dichloroethene	75-35-4	5	1	5	250	620
1,1-Dichloroethane	75-34-3	5	1	5	250	620
trans-1,2-Dichloroethene	156-60-5	5.0	1.0	5.0	250	310
Acrylonitrile	107-13-1	100	20	100	5000	12000
Methyl *tert*-butyl ether (MTBE)	1634-04-4	20	5	20	1000	2500
Hexane	110-54-3	5	1	5	250	620
cis-1,2-Dichloroethene	156-59-2	5.0	1.0	5.0	250	310
1,2-Dichloroethene (Total)	540-59-0	5	1	5	250	620
Tetrahydrofuran	109-99-9	20	5	20	1000	2500
Chloroform	67-66-3	5	1	5	250	620
1,2-Dichloroethane	107-06-2	5	1	5	250	620
Dibromomethane	74-95-3	5	1	5	250	620
2-Butanone	78-93-3	20	5	20	1000	2500
1,4-Dioxane	123-91-1	500	200	500	25000	62000
1,1,1-Trichloroethane	71-55-6	5	1	5	250	620
Carbon tetrachloride	56-23-5	5	1	5	250	620
Bromodichloromethane	75-27-4	5	1	5	250	620
1,2-Dichloropropane	78-87-5	5	1	5	250	620
cis-1,3-Dichloropropene	10061-01-5	5	1	5	250	620

Table 1

STL Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	25 mL[3] water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
Trichloroethene	79-01-6	5	1	5	250	620
Dibromochloromethane	124-48-1	5	1	5	250	620
1,2-Dibromoethane	106-93-4	5	1	5	250	620
1,2,3-Trichloropropane	96-18-4	5	1	5	250	620
1,1,2-Trichloroethane	79-00-5	5	1	5	250	620
Benzene	71-43-2	5	1	5	250	620
Ethylmethacrylate	97-63-2	5	1	5	250	620
trans-1,3-Dichloropropene	10061-02-6	5	1	5	250	620
Bromoform	75-25-2	5	1	5	250	620
4-Methyl-2-pentanone	108-10-1	20	5	20	1000	2500
2-Hexanone	591-78-6	20	5	20	1000	2500
Tetrachloroethene	127-18-4	5	1	5	250	620
Toluene	108-88-3	5	1	5	250	620
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	250	620
2-Chloroethyl vinyl ether	110-75-8	N/A[2]	N/A	50	1000	6200
Vinyl acetate	108-05-4	10	2	10	500	1200
Chlorobenzene	108-90-7	5	1	5	250	620
Ethylbenzene	100-41-4	5	1	5	250	620
Styrene	100-42-5	5	1	5	250	620
t-1,4-Dichloro-2-butene	110-57-6	5	1	5	250	620
m and p Xylenes		5.0	0.5	2.5	125	310
o-xylene	95-47-6	5.0	0.5	2.5	125	310
Total xylenes	1330-20-7	5	1	5	250	620
1,3-Dichlorobenzene	541-73-1	5	1	5	250	620
1,4-Dichlorobenzene	106-46-7	5	1	5	250	620
1,2-Dichlorobenzene	95-50-1	5	1	5	250	620
2,2-Dichloropropane	590-20-7	5	1	5	250	
Bromochloromethane	74-97-5	5	1	5	250	
1,1-Dichloropropene	563-58-6	5	1	5	250	
Bromodichloromethane	75-27-4	5	1	5	250	
1,2-Dichloropropane	78-87-5	5	1	5	250	
1,3-Dichloropropane	142-28-9	5	1	5	250	

Table 1

STL Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]				
		5 mL Water µg/L	25 mL[3] water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
Isopropylbenzene	98-82-8	5	1	5	250	
Bromobenzene	108-86-1	5	1	5	250	
n-Propylbenzene	103-65-1	5	1	5	250	
2-Chlorotoluene	95-49-8	5	1	5	250	
4-Chlorotoluene	106-43-4	5	1	5	250	
1,3,5-Trimethylbenzene	108-67-8	5	1	5	250	
tert-Butylbenzene	98-06-6	5	1	5	250	
1,2,4-Trimethylbenzene	95-63-6	5	1	5	250	
sec-butylbenzene	135-98-8	5	1	5	250	
4-Isopropyltoluene	99-87-6	5	1	5	250	
n-Butylbenzene	104-51-8	5	1	5	250	
1,2,4-Trichlorobenzene	120-82-1	5	1	5	250	
Napthalene	91-20-3	5	1	5	250	
Hexachlorobutadiene	87-68-3	5	1	5	250	
1,2,3-Trichlorobenzene	87-61-6	5	1	5	250	
Acetonitrile	75-05-8	100	20	100	5000	

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

[2] 2-Chloroethyl vinyl ether cannot be reliably recovered from acid preserved samples

[3] Optionally, 5 mL purge volume if adequate sensitivity is obtained.

Table 2

STL Primary Standard Calibration Levels, 5 mL purge[1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2-Dichloroethane-d4 (Surrogate)	5	20	50	100	200
Toluene-d8 (Surrogate)	5	20	50	100	200
4-Bromofluorobenzene (Surrogate)	5	20	50	100	200
Dichlorodifluoromethane	5	20	50	100	200
Chloromethane	5	20	50	100	200
Bromomethane	5	20	50	100	200
Vinyl chloride	5	20	50	100	200
Chloroethane	5	20	50	100	200
Trichlorofluoromethane	5	20	50	100	200
Acrolein	50	200	500	1000	2000
Acetone	5	20	50	100	200
Trichlorotrifluoroethane	5	20	50	100	200
Iodomethane	5	20	50	100	200
Carbon disulfide	5	20	50	100	200
Methylene chloride	5	20	50	100	200
tert-Butyl alcohol	100	400	1,000	2,000	4,000
1,1-Dichloroethene	5	20	50	100	200
1,1-Dichloroethane	5	20	50	100	200
trans-1,2-Dichloroethene	5	20	50	100	200
Acrylonitrile	50	200	500	1,000	2,000
Methyl *tert*-butyl ether (MTBE)	5	20	50	100	200
Hexane	5	20	50	100	200
cis-1,2-Dichloroethene	5	20	50	100	200
Tetrahydrofuran	5	20	50	100	200
Chloroform	5	20	50	100	200
1,2-Dichloroethane	5	20	50	100	200
Dibromomethane	5	20	50	100	200
2-Butanone	5	20	50	100	200
1,4-Dioxane	250	1000	2,500	5,000	10,000
1,1,1-Trichloroethane	5	20	50	100	200
Carbon tetrachloride	5	20	50	100	200
Bromodichloromethane	5	20	50	100	200
1,2-Dichloropropane	5	20	50	100	200
cis-1,3-Dichloropropene	5	20	50	100	200
Trichloroethene	5	20	50	100	200
Dibromochloromethane	5	20	50	100	200

Table 2

STL Primary Standard Calibration Levels, 5 mL purge[1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2-Dibromoethane	5	20	50	100	200
1,2,3-Trichloropropane	5	20	50	100	200
Acetonitrile	50	200	500	1000	2000
1,1,2-Trichloroethane	5	20	50	100	200
Benzene	5	20	50	100	200
Ethylmethacrylate	5	20	50	100	200
trans-1,3-Dichloropropene	5	20	50	100	200
Bromoform	5	20	50	100	200
4-Methyl-2-pentanone	5	20	50	100	200
2-Hexanone	5	20	50	100	200
Tetrachloroethene	5	20	50	100	200
Toluene	5	20	50	100	200
1,1,2,2-Tetrachloroethane	5	20	50	100	200
2-Chloroethyl vinyl ether	10	40	100	200	400
Vinyl acetate	5	20	50	100	200
Chlorobenzene	5	20	50	100	200
Ethylbenzene	5	20	50	100	200
Styrene	5	20	50	100	200
t-1,4-Dichloro-2-butene	5	20	50	100	200
m and p Xylenes	10	40	100	200	400
o-xylene	5	20	50	100	200
1,3-Dichlorobenzene	5	20	50	100	200
1,4-Dichlorobenzene	5	20	50	100	200
1,2-Dichlorobenzene	5	20	50	100	200
2,2-Dichloropropane	5	20	50	100	200
Bromochloromethane	5	20	50	100	200
1,1-Dichloropropene	5	20	50	100	200
Bromodichloromethane	5	20	50	100	200
1,2-Dichloropropane	5	20	50	100	200
1,3-Dichloropropane	5	20	50	100	200
Isopropylbenzene	5	20	50	100	200
Bromobenzene	5	20	50	100	200
n-Propylbenzene	5	20	50	100	200
2-Chlorotoluene	5	20	50	100	200
4-Chlorotoluene	5	20	50	100	200
1,3,5-Trimethylbenzene	5	20	50	100	200
tert-Butylbenzene	5	20	50	100	200

Table 2

STL Primary Standard Calibration Levels, 5 mL purge[1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2,4-Trimethylbenzene	5	20	50	100	200
sec-butylbenzene	5	20	50	100	200
4-Isopropyltoluene	5	20	50	100	200
n-Butylbenzene	5	20	50	100	200
1,2,4-Trichlorobenzene	5	20	50	100	200
Napthalene	5	20	50	100	200
Hexachlorobutadiene	5	20	50	100	200
1,2,3-Trichlorobenzene	5	20	50	100	200

[1] **Levels for 25 mL purge are 5 times lower in all cases**

Table 2A

STL Primary Standard Calibration Levels, Low Level[1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Dibromofluoromethane (Surrogate)	1	5	10	20	40
1,2-Dichloroethane-d4 (Surrogate)	1	5	10	20	40
Toluene-d8 (Surrogate)	1	5	10	20	40
Bromofluorobenzene (Surrogate)	1	5	10	20	40
Dichlorodifluoromethane	1	5	10	20	40
Chloromethane	1	5	10	20	40
Vinyl Chloride	1	5	10	20	40
Bromomethane	1	5	10	20	40
Chloroethane	1	5	10	20	40
Trichlorofluoromethane	1	5	10	20	40
Acrolein	10	50	100	200	400
Acetone	2	10	20	40	80
1,1-Dichloroethene	1	5	10	20	40
Trichlorotrifluoroethane	1	5	10	20	40
Iodomethane	1	5	10	20	40
Carbon Disulfide	1	5	10	20	40
Methylene Chloride	1	5	10	20	40
Acetonitrile	10	50	100	200	400
Acrylonitrile	10	50	100	200	400
Methyl tert-butyl ether	1	5	10	20	40
trans-1,2-Dichloroethene	1	5	10	20	40
Hexane	1	5	10	20	40
Vinyl acetate	1	5	10	20	40
1,1-Dichloroethane	1	5	10	20	40
tert-Butyl Alcohol	20	100	200	400	800
2-Butanone	2	10	20	40	80
cis-1,2-dichloroethene	1	5	10	20	40
2,2-Dichloropropane	1	5	10	20	40
Bromochloromethane	1	5	10	20	40
Chloroform	1	5	10	20	40
Tetrahydrofuran	1	5	10	20	40
1,1,1-Trichloroethane	1	5	10	20	40
1,1-Dichloropropene	1	5	10	20	40
Carbon Tetrachloride	1	5	10	20	40
1,2-Dichloroethane	1	5	10	20	40
Benzene	1	5	10	20	40
Trichloroethene	1	5	10	20	40
1,2-Dichloropropane	1	5	10	20	40
1,4-Dioxane	50	250	500	1000	2000
Dibromomethane	1	5	10	20	40

Table 2A

STL Primary Standard Calibration Levels, Low Level [1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Bromodichloromethane	1	5	10	20	40
2-Chloroethyl vinyl ether	2	10	20	40	80
cis-1,3-Dichloropropene	1	5	10	20	40
4-Methyl-2-pentanone	2	10	20	40	80
Toluene	1	5	10	20	40
trans-1,3-Dichloropropene	1	5	10	20	40
Ethyl Methacrylate	1	5	10	20	40
1,1,2-Trichloroethane	1	5	10	20	40
1,3-Dichloropropane	1	5	10	20	40
Tetrachloroethene	1	5	10	20	40
2-Hexanone	2	10	20	40	80
Dibromochloromethane	1	5	10	20	40
1,2-Dibromoethane	1	5	10	20	40
Chlorobenzene	1	5	10	20	40
1,1,1,2-Tetrachloroethane	1	5	10	20	40
Ethylbenzene	1	5	10	20	40
m + p-Xylene	2	10	20	40	80
Xylene-o	1	5	10	20	40
Styrene	1	5	10	20	40
Bromoform	1	5	10	20	40
Isopropylbenzene	1	5	10	20	40
1,1,2,2-Tetrachloroethane	1	5	10	20	40
1,4-Dichloro-2-butene	1	5	10	20	40
1,2,3-Trichloropropane	1	5	10	20	40
Bromobenzene	1	5	10	20	40
n-Propylbenzene	1	5	10	20	40
2-Chlorotoluene	1	5	10	20	40
1,3,5-Trimethylbenzene	1	5	10	20	40
4-Chlorotoluene	1	5	10	20	40
tert-Butylbenzene	1	5	10	20	40
1,2,4-Trimethylbenzene	1	5	10	20	40
sec-Butylbenzene	1	5	10	20	40
4-Isopropyltoluene	1	5	10	20	40
1,3-Dichlorobenzene	1	5	10	20	40
1,4-Dichlorobenzene	1	5	10	20	40
n-Butylbenzene	1	5	10	20	40
1,2-Dichlorobenzene	1	5	10	20	40
1,2-Dibromo-3-chloropropane	1	5	10	20	40

Table 2A

STL Primary Standard Calibration Levels, Low Level [1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2,4-Trichlorobenzene	1	5	10	20	40
Hexachlorobutadiene	1	5	10	20	40
Naphthalene	1	5	10	20	40
1,2,3-Trichlorobenzene	1	5	10	20	40
Cyclohexane	1	5	10	20	40
Methyl Acetate	2	10	20	40	80
Methylcyclohexane	1	5	10	20	40
1,3,5-Trichlorobenzene	1	5	10	20	40

[1] 25 mL purge samples analyzed at 5 mL purge on more sensitive equipment.

Table 3

STL Appendix IX Standard and Reporting Limits, 5 mL purge

Compound	CAS Number	Reporting Limits			
		5 mL Water µg/L	25mL[2] water µg/L	Low Soil µg/kg	Medium Soil µg/mL
Allyl Chloride	107-05-1	10	2	10	500
Acetonitrile	75-05-8	100	20	100	5000
Dichlorofluoromethane		10	2	10	500
Isopropyl ether	108-20-3	10	2	10	500
Chloroprene	126-99-8	5	2	5	250
n-Butanol	71-36-3	200	50	200	10,000
Propionitrile	107-12-0	20	4	20	1000
Methacrylonitrile	126-98-7	5	2	5	250
Isobutanol	78-83-1	200	50	200	10,000
Methyl methacrylate	80-62-6	5	2	5	250
1,1,1,2-Tetrachloroethane	630-20-6	5	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	10	2	10	500
Ethyl ether	60-29-7	10	2	10	500
Ethyl Acetate	141-78-6	20	4	20	1,000
2-Nitropropane	79-46-9	10	4	10	500
Cyclohexanone	108-94-1	N/A[1]	N/A[1]	N/A[1]	N/A[1]
Isopropylbenzene	98-82-8	5	1	5	250
2-Methylnaphthalene (Michigan only)	91-57-6	NA	5	NA	330

[1] Cyclohexanone decomposes to 1,1-dimethoxycyclohexane in methanolic solution. Reporting limits cannot be accurately determined.

[2] Optionally, 5 mL purge volume if adequate sensitivity is obtained.

Table 4

Recommended/STL Appendix IX Standard Calibration Levels, µg/L

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Allyl Chloride	5	20	50	100	200
Acetonitrile	50	200	500	1,000	2,000
Dichlorofluoromethane	5	20	50	100	200
Isopropyl ether	5	20	50	100	200
Chloroprene	5	20	50	100	200
n-Butanol	100	400	1,000	2,000	4,000
Propionitrile	10	40	100	200	400
Methacrylonitrile	5	20	50	100	200
Isobutanol	100	400	1,000	2,000	4,000
Methyl methacrylate	5	20	50	100	200
1,1,1,2-Tetrachloroethane	5	20	50	100	200
1,2-Dibromo-3-chloropropane	10	40	100	200	400
Ethyl ether	5	20	50	100	200
Ethyl Acetate	10	40	100	200	400
2-Nitropropane	10	40	100	200	400
Cyclohexanone	50	200	500	1,000	2,000
2-Methylnaphthalene (Michigan only)	2	10	20	40	80

Table 5

Reportable Analytes for STL Standard Tests, Primary Standard

Compound	CAS Number	STL Standard List	TCLP	TCL	Appendix IX	UTS
Dichlorodifluoromethane	75-71-8				X	X
Chloromethane	74-87-3	X		X	X	X
Bromomethane	74-83-9	X		X	X	X
Vinyl chloride	75-01-4	X	X	X	X	X
Chloroethane	75-00-3	X		X	X	X
Trichlorofluoromethane	75-69-4				X	X
Acrolein	107-02-8				X	X
Acetone	67-64-1	X		X	X	X
Trichlorotrifluoroethane	76-13-1					X
Ethanol	64-17-5					
Iodomethane	74-88-4				X	X
Carbon disulfide	75-15-0	X		X	X	X
Methylene chloride	75-09-2	X		X	X	X
tert-Butly alcohol	75-65-0					
1,1-Dichloroethene	75-35-4	X	X	X	X	X
1,1-Dichloroethane	75-34-3	X		X	X	X
trans-1,2-Dichloroethene	156-60-5	X		X	X	X
Total dichloroethene		X		X	X	X
Acrylonitrile	107-13-1				X	X
Methyl *tert*-butyl ether (MTBE)	1634-04-4					
Hexane	110-54-3					
cis-1,2-Dichloroethene	156-59-2	X		X		
Tetrahydrofuran	109-99-9					
Chloroform	67-66-3	X	X	X	X	X
1,2-Dichloroethane	107-06-2	X	X	X	X	X
Dibromomethane	74-95-3				X	X
2-Butanone	78-93-3	X	X	X	X	X
1,4-Dioxane	123-91-1				X	X
1,1,1-Trichloroethane	71-55-6	X		X	X	X
Carbon tetrachloride	56-23-5	X	X	X	X	X
Bromodichloromethane	75-27-4	X		X	X	X
1,2-Dichloropropane	78-87-5	X		X	X	X
cis-1,3-Dichloropropene	10061-01-5	X		X	X	X
Trichloroethene	79-01-6	X	X	X	X	X

Table 5

Reportable Analytes for STL Standard Tests, Primary Standard

Compound	CAS Number	STL Standard List	TCLP	TCL	Appendix IX	UTS
Dibromochloromethane	124-48-1	X		X	X	X
1,2-Dibromoethane	106-93-4				X	X
1,2,3-Trichloropropane	96-18-4				X	X
1,1,2-Trichloroethane	79-00-5	X		X	X	X
Benzene	71-43-2	X	X	X	X	X
Ethylmethacrylate	97-63-2				X	X
trans-1,3-Dichloropropene	10061-02-6	X		X	X	X
Bromoform	75-25-2	X		X	X	X
4-Methyl-2-pentanone	108-10-1	X		X	X	X
2-Hexanone	591-78-6	X		X	X	
Tetrachloroethene	127-18-4	X	X	X	X	X
Toluene	108-88-3	X		X	X	X
1,1,2,2-Tetrachloroethane	79-34-5	X		X	X	X
2-Chloroethyl vinyl ether	110-75-8					
Vinyl acetate	108-05-4				X	
Chlorobenzene	108-90-7	X	X	X	X	X
Ethylbenzene	100-41-4	X		X	X	X
Styrene	100-42-5	X		X	X	
t-1,4-Dichloro-2-butene	110-57-6				X	
m and p Xylenes		X		X	X	X
o-xylene	95-47-6	X		X	X	X
Total xylenes	1330-20-7	X		X	X	X
1,3-Dichlorobenzene	541-73-1					
1,4-Dichlorobenzene	106-46-7					
1,2-Dichlorobenzene	95-50-1					

Table 6

Reportable Analytes for STL Standard Tests, Appendix IX standard

Compound	Number	STL Standard List	TCLP	TCL	Appendix IX	UTS
Allyl Chloride	107-05-1				X	
Acetonitrile	75-05-8				X	X
Dichlorofluoromethane	75-43-4					
Isopropyl ether	108-20-3					
Chloroprene	126-99-8				X	
n-Butanol	71-36-3					
Propionitrile	107-12-0				X	
Methacrylonitrile	126-98-7				X	X
Isobutanol	78-83-1				X	X
Methyl methacrylate	80-62-6				X	X
1,1,1,2-Tetrachloroethane	630-20-6				X	X
1,2-Dibromo-3-chloropropane	96-12-8				X	X
Ethyl ether	60-29-7					X
Ethyl Acetate	141-78-6					X
2-Nitropropane	79-46-9					
Cyclohexanone	108-94-1					X
Isopropylbenzene	98-82-8					

Table 7

Internal Standards

	Standard Concentration µg/mL	Quantitation ion (5 mL purge)	Quantitation ion (25 mL purge)
Fluorobenzene	50	96	96
Chlorobenzene-d5	50	117	119
1,4-Dichlorobenzene-d4	50	152	152

Notes:

1) 5 µL of the internal standard is added to the sample. This results in a concentration of each internal in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 8

Surrogate Standards

Surrogate Compounds	Standard Concentration µg/mL
1,2-Dichloroethane-d_4	50
Dibromofluoromethane	50
Toluene-d_8	50
4-Bromofluorobenzene	50

Notes:

1) 5 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

3) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 9

Matrix Spike / LCS Control Compounds

Compound	Standard Concentration µg /mL
1,1-Dichloroethene	50
Trichloroethene	50
Toluene	50
Benzene	50
Chlorobenzene	50
n-Hexane (Ohio VAP only)	50

Notes:

1) 5 µL of the standard is added to the LCS or matrix spiked sample. This results in a concentration of each spike analyte in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Recovery and precision limits for LCS and MS/MSD are generated fromhistorical data and are maintained by the QA dedartment.

3) Full analyte spikes may also be used at the laboratories option or at client request.

Table 10

BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

Table 11

SPCC Compounds and Minimum Response Factors

Compound	8260B, 8260A Min. RF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	>0.100
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

Table 12

CCC compounds

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	<30.0	<20.0
1,1-Dichloroethene	<30.0	<20.0
Chloroform	<30.0	<20.0
1,2-Dichloropropane	<30.0	<20.0
Toluene	<30.0	<20.0
Ethylbenzene	<30.0	<20.0
n-Hexane (Ohio VAP only)	<30.0	<20.0

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d_4 (Surrogate)	65	102	
Dichlorodifluoromethane	85	87	50, 101,103
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58
Iodomethane	142	127	141
Carbon disulfide	76	78	
Trichlorotrifluoroethane	151	101	153
Ethanol	45	46	
Acetone	43	58	
Methylene chloride	84	49	51, 86
tert-Butyl alcohol	59	74	
trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl *tert* butyl ether	73		
Hexane	57	43	
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	
Tetrahydrofuran	42	71	
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	
Vinyl acetate	43	86	
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121
Benzene	78	52	77
Trichloroethene	130	95	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Toluene-d_8 (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65
4-Methyl-2-pentanone	43	58	57, 100

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77
Ethylbenzene	106	91	
Xylenes	106	91	
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133
Allyl Chloride	76	41	78
Acetonitrile	40	41	
Dichlorofluoromethane	67	69	
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74
Methyl methacrylate	41	69	100
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	

* The primary ion should be used for quantitation unless interferences are present, in which case a secondary ion may be used.

** m/z 43 may be used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

1. REQUIREMENTS FOR EPA 624

1.1. Method 624 is required for demonstration of compliance with NPDES wastewater discharge permits. This method can be applied only to aqueous matrices. The standard analyte list and reporting limits are listed in Table B-1.

1.2. The tune period for this method is defined as 24 hours.

1.3. The initial calibration curve for this method requires at least three points.

1.4. Sample concentrations are calculated using the average RRF from the initial calibration curve.

1.5. Each target analyte is assigned to the closest eluting internal standard.

1.6. Initial demonstration of Proficiency

1.6.1. The spiking level for the four replicate initial demonstration of proficiency is 20 µg/L. The acceptance criteria are listed in Table B-2

1.7. Initial calibration curve requirements:

1.7.1. Target compounds must have RSD ≤ 35%.

1.7.2. If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds. There is no correlation coefficient requirement for the regression curve.

1.8. Continuing calibration verification requirements:

1.8.1. The continuing calibration standard is from a different source than the initial calibration standard. The acceptance criteria are listed in Table B-2.

1.9. Matrix Spike and LCS requirements

1.9.1. The matrix spike and LCS are spiked at 20 µg/L. A matrix spike duplicate is not necessary for this method. The recovery limits for matrix spike and LCS recovery are listed in Table C-2.

1.10. Method clarifications, modifications and additions

1.10.1. Section 5.2.2 of the source method describes the trap packing materials as Tenax GC, Methyl silicone, silica gel and coconut charcoal. STL routinely employs the Supelco VOCARB 3000, which consists of Carbopack B and Carboxen 1000 and 1001.

1.10.2. Section 5.3.2 of the source method describes a packed analytical column. STL routinely employs capillary columns when performing this method.

1.10.3. The source method provides a suggested list of compounds for internal and surrogate standards. STL uses the following two compounds which are not on the table: Chlorobenzene-d_5 (internal standard) and 1,2-Difluorobenzene-d_4 (surrogate).

Table A-1.

Method 624 Analytes and Reporting Limits

Analytes	µg/L
Benzene	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	5
Carbon tetrachloride	5
Chlorobenzene	5
Chloroethane	5
2-Chloroethyl vinyl ether	5
Chloroform	5
Chloromethane	5
Dibromochloromethane	5
1,2-Dichlorobenzene	5
1,3-Dichlorobenzene	5
1,4-Dichlorobenzene	5
1,1-Dichloroethane	5
1,2-Dichloroethane	5
1,1-Dichloroethene	5
trans-1,2-Dichloroethene	5
1,2-Dichloropropane	5
cis-1,3-Dichloropropene	5
trans-1,3-Dichloropropene	5
Ethylbenzene	5
Methylene chloride	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	5
Vinyl chloride	5

Table A-2.

Method 624 QC Acceptance Criteria

Analytes	Daily QC check acceptance criteria (20µg/L spike)	Mean recovery, 4 replicate initial demonstration acceptance criteria (20µg/L spike)	Standard deviation, 4 replicate initial demonstration acceptance criteria (20µg/L spike)	Matrix spike and LCS acceptance criteria (% recovery)
Benzene	12.8-27.2	15.2-26.0	6.9	37-151
Bromodichloromethane	13.1-26.9	10.1-28.0	6.4	35-155
Bromoform	14.2-25.8	11.4-31.1	5.4	45-169
Bromomethane	2.8-37.2	D-41.2	17.9	D-242
Carbon tetrachloride	14.6-25.4	17.2-23.5	5.2	70-140
Chlorobenzene	13.2-26.8	16.4-27.4	6.3	37-160
Chloroethane	7.6-32.4	8.4-40.4	11.4	14-230
2-Chloroethyl vinyl ether	D-44.8	D-50.4	25.9	D-305
Chloroform	13.5-26.5	13.7-24.2	6.1	51-138
Chloromethane	D-40.8	D-45.9	19.8	D-273
Dibromochloromethane	13.5-26.5	13.8-26.6	6.1	53-149
1,2-Dichlorobenzene	12.6-27.4	11.8-34.7	7.1	18-190
1,3-Dichlorobenzene	14.6-25.4	17.0-28.8	5.5	59-156
1,4-Dichlorobenzene	12.6-27.4	11.8-34.7	7.1	18-190
1,1-Dichloroethane	14.5-25.5	14.2-28.5	5.1	59-155
1,2-Dichloroethane	13.6-26.4	14.3-27.4	6.0	49-155
1,1-Dichloroethene	10.1-29.9	3.7-42.3	9.1	D-234
trans-1,2-Dichloroethene	13.9-26.1	13.6-28.5	5.7	54-156
1,2-Dichloropropane	6.8-33.2	3.8-36.2	13.8	D-210
cis-1,3-Dichloropropene	4.8-35.2	1.0-39.0	15.8	D-227
trans-1,3-Dichloropropene	10.0-30.0	7.6-32.4	10.4	17-183
Ethylbenzene	11.8-28.2	17.4-26.7	7.5	37-162
Methylene chloride	12.1-27.9	D-41.0	7.4	D-221
1,1,2,2-Tetrachloroethane	12.1-27.9	13.5-27.2	7.4	46-157
Tetrachloroethene	14.7-25.3	17.0-26.6	5.0	64-148
Toluene	14.9-25.1	16.6-26.7	4.8	47-150
1,1,1-Trichloroethane	15.0-25.0	13.7-30.1	4.6	52-162
1,1,2-Trichloroethane	14.2-25.8	14.3-27.1	5.5	52-150
Trichloroethene	13.3-26.7	18.6-27.6	6.6	71-157
Trichlorofluoromethane	9.6-30.4	8.9-31.5	10.0	17-181
Vinyl chloride	0.8-39.2	D-43.5	20.0	D-251

STL NORTH CANTON STANDARD OPERATING PROCEDURE

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(SUPERSEDES: Revision 3.7 (Dated 03/20/01))

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1. SCOPE AND APPLICATION

This SOP describes procedures for preparation (extraction and cleanup) of semivolatile organic analytes in aqueous, TCLP leachate, and soil matrices for analysis by Gas Chromatography (GC) and Gas Chromatography / Mass Spectrometry (GC/MS). The procedures are based on SW-846 and 600 series methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA) and for wastewater testing.

1.1. Extraction procedures for the following determinative methods are covered:

8081A, 8082, 8141A, 8151B, 8270C, 8310, 8015B, 8100, 608, 610, and 625

1.2. The extraction procedures here may be appropriate for other determinative methods when appropriate spiking mixtures are used.

1.3. Other extraction procedures (PFE, SPE, accelerated Soxhlet, etc.) may be used but are not currently covered in this SOP.

1.4. The applicable LIMS method codes are: P2 (8141A), QJ (8081A), QS (8151A), QL (8270C), SG (8310), DM (608), VT (610), DP (625), A1 (8100), HS (8015B)

2. SUMMARY OF METHOD

2.1. Separatory Funnel Extraction

A measured volume of sample, typically 1 liter, is adjusted, if necessary, to a specified pH and serially extracted with methylene chloride using a separatory funnel.

2.2. Continuous Liquid/Liquid Extraction

A measured volume of sample, typically 1 liter, is placed into a continuous liquid/liquid extractor, adjusted, if necessary, to a specific pH and extracted with methylene chloride for 18-24 hours.

2.3. Sonication Extraction

A measured weight of sample, typically 30 g, is mixed with anhydrous sodium sulfate to form a free flowing powder. This is solvent extracted three times using an ultrasonic horn.

2.4. Soxhlet Extraction

A 30 g sample is mixed with anhydrous sodium sulfate to form a free flowing powder. This is extracted with refluxing solvent.

2.5. Cleanup and Concentration

Procedures are presented for removing interferents from sample extracts, and for drying and concentration of the extract to final volume for analysis.

2.6. Phenoxy acid herbicide extractions

Procedures for the extraction and cleanup of phenoxy acid herbicides are presented in Appendix A.

3. DEFINITIONS

Definitions of terms used in this SOP may be found in the glossary of the STL North Canton Laboratory Quality Manual (LQM), current version .

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL North Canton associates. The following requirements must be met:

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded, other gloves will be cleaned immediately. Viton gloves may be worn when halogenated solvents are used for extractions or sample preparation. Nitrile gloves may be used when other solvents are handled. [Note: VITON is readily degraded by acetone]. When good manual dexterity is needed, for example, when handling small quantities/containers, disposable gloves (such as latex or N-DEX®) shall be used. While these gloves protect against splashes, they give little or no protection against contact with large quantities of solvent, and no protection against spills or immersion.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

Chemicals that have been classified as **carcinogens** or **potential carcinogens** under OSHA include methylene chloride, PCBs, certain pesticides.

The following materials are known to be **corrosive**: Concentrated sulfuric acid, sodium hydroxide, 1N nitric acid.

Sulfuric acid cleanup must not be performed on any matrix that may have water present as a violent reaction between the acid and water may result in acid exploding out of the vessel.

Chemicals known to be **flammable** are: Diethyl ether, acetone, hexane, 2-propanol.
Mercury is a highly toxic compound that must be handled with care. The analyst must be aware of the appropriate handling and clean-up techniques before handling this material.

5.4. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents and glassware cleaning procedures that involve solvents such as methylene chloride will be conducted in a fume hood with the sash closed as far as the operations will permit. Use of methylene chloride for glassware cleaning should be avoided as far as possible. If more than 500 mL of Methylene chloride is spilled, evacuate the area until the area has been cleaned by a Hazardous Material Team.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Glassware should be cleaned with soap and water, rinsed with water and dried in an oven at 400°C for at least 2 hours. Alternatively the glassware can be solvent rinsed with acetone or methanol followed by methylene chloride after the water rinse.

6.2. Equipment and supplies for extraction procedures

EQUIPMENT AND SUPPLIES	Sep fun.	CLLE	Soni	Sox	Conc
Separatory Funnel: 2 L	√				
Separatory Funnel Rack	√				
Balance: >1400 g capacity, accurate ±1 g	√	√			
pH indicator paper, wide-range: covers extraction pH	√	√			
Graduated cylinder: 1 liter. (other sizes may be used)	√	√			
Erlenmeyer Flask or Fleaker: 125 & 300 mL (other sizes optional)	√		√		
Centrifuge	√				
Auto-Shaker	√				
Methylene Chloride Collection Tank	√	√			

EQUIPMENT AND SUPPLIES	Sep fun.	CLLE	Soni	Sox	Conc
Solvent Dispenser Pump or 100 mL Graduated Cylinder	√		√		
Continuous Liquid/Liquid Extractor		√			
Round or flat Bottom: 250, 500 mL or 1 L		√			
Boiling Chips: Contaminant free, approximately 10/40 mesh (Teflon® PTFE, carbide or equivalent).		√		√	√
Cooling Condensers		√		√	
Heating Mantle: Rheostat controlled		√		√	
Auto-timer for heating mantle		√		√	
Beakers: 250 & 400 mL, graduated			√		
Balance: >100 g capacity, accurate ±0.1 g			√	√	
Soxhlet Extractor				√	
Sonicator (at least 300 watts)			√		
Sonicator horn, 3/4 inch			√		
Kuderna-Danish (K-D) Apparatus: 500 mL					√
Concentrator Tube: 10 mL, attached to K-D with clips					√
Supply Manifold: Heated, capable of temperature control (± 5°C) up to 95°C.					√
MegaCT: 150 ml					√
Snyder Column: Three-ball macro					√
Water Bath: Heated, with concentric ring cover, capable of temperature control (± 5°C) up to 95°C. The bath must be used in a hood or with a solvent recovery system.					√
Vials: Glass, 2 mL, 4 mL, and 10 mL capacity with Teflon®-lined screw-cap					√
Nitrogen Blowdown Apparatus					√
Nitrogen: reagent grade.					√
Culture tubes: 10 mL, 16 mmx100 mm					√
Syringe: 1 mL	√	√	√	√	
Phase Separation Paper	√	√	√	√	
Glass Wool	√	√	√	√	
Glass Funnel: 75 X 75 mm	√	√	√	√	√
Disposable Pipets	√	√	√	√	√
Aluminum foil	√	√	√	√	√
Paper Towels	√	√	√	√	√
Wrist Shaker					

6.3. Equipment and Supplies for Cleanup Procedures

EQUIPMENT AND SUPPLIES	GPC	Florisil	Sulfur	Acid	A/B	FC
Gel permeation chromatography system (GPC Autoprep Model 1002A or 1002B Analytical Biochemical Laboratories, Inc. or Zymark	√					

EQUIPMENT AND SUPPLIES	GPC	Florisil	Sulfur	Acid	A/B	FC
Benchmate or equivalent).						
Bio Beads: (S-X3) -200-400 mesh, 70 gm (Bio-Rad Laboratories, Richmond, CA, Catalog 152-2750 or equivalent).	√					
Chromatographic column: 700 mm x 25 mm ID glass column. Flow is upward.	√					
Ultraviolet detector: Fixed wavelength (254 nm) and a semi-prep flow-through cell.	√					
Strip chart recorder, recording integrator, or laboratory data system.	√					
Syringe: 10 mL with Luerlok fitting.	√					
Syringe filter assembly, with disposable 5 um filter discs, Millipore No. LSWP 01300 or equivalent.	√					
Chromatographic column: 250 mm long x 10 mm ID; with Pyrex glass wool at the bottom and a Teflon stopcock (for silica gel cleanup).	√					
Vacuum system for eluting multiple cleanup cartridges. Vac Elute Manifold - Analytichem International, J.T. Baker, or Supelco (or equivalent). The manifold design must ensure that there is no contact between plastics containing phthalates and sample extracts.		√				
Vacuum trap made from a 500 mL sidearm flask fitted with a one-hole stopper and glass tubing.		√				
Vacuum pressure gauge.		√				
Rack for holding 10 mL volumetric flasks in the manifold.		√				
Mechanical shaker or mixer: Vortex Genie or equivalent.			√	√		
Separatory Funnels with Ground-Glass Stoppers: 250 mL					√	
Erlenmeyer Flasks: 125 mL					√	
Disposable Pipets		√	√	√	√	√
Culture tubes: 10 mL, 16 mmx100 mm	√	√	√	√	√	√

7. REAGENTS AND STANDARDS

7.1. Reagents for Extraction Procedures

All reagents must be ACS reagent grade or better unless otherwise specified.

REAGENTS	Sep fun.	CLLE	Soni	Sox	Conc
Sodium hydroxide (NaOH), Pellets: Reagent Grade	√	√			
Sodium hydroxide solution, 10 N: Dissolve 40 g of NaOH in reagent water and dilute to 100 mL.	√	√			
Sulfuric acid (H_2SO_4), Concentrated: Reagent Grade	√	√			
Sulfuric acid (1:1): Carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.	√	√			

REAGENTS	Sep fun.	CLLE	Soni	Sox	Conc
Hydrochloric Acid (HCl)					
Organic free reagent water.	√	√			
Sodium sulfate (Na_2SO_4), Granular, Anhydrous: Purify by heating at 400°C a minimum of two hours.	√	√	√	√	√
Extraction/Exchange Solvents: Methylene chloride, hexane, acetonitrile, acetone, pesticide quality or equivalent	√	√	√	√	√
Acetone: Used for cleaning	√	√	√	√	√

7.2. Reagents for Cleanup Procedures

REAGENTS	GPC	Florisil	Sulfur	Acid	A/B	*FC*
Florisil: 500 mg or 1 g cartridges with stainless steel or Teflon frits (calalog 694-313, Analytichem, 24201 Frampton Ave., Harbor City, CA, or equivalent.)		√				
Mercury: triple distilled			√			
Tetrabutylammonium hydrogen sulfate			√			
Sodium sulfite			√			
Tetrabutylammonium (TBA) sulfite reagent: Prepare reagent by dissolving 3.39 g of Tetrabutylammonium hydrogen sulfate in 100 mL organic-free reagent water. Extract this solution 3 times with 20 mL portions of hexane. Discard the hexane extracts. Add 25 g sodium sulfite to the water solution.			√			
2-Propanol			√			
Nitric acid: 1N			√			
Copper powder: remove oxides (if powder is dark) by treating with 1N nitric acid, rinse with organic-free reagent water to remove all traces of acid, rinse with acetone, and dry under a stream of nitrogen.			√			
Sulfuric acid, Concentrated				√		
Sodium hydroxide, Pellets					√	
Sodium hydroxide, 10N: Dissolve 40 g of NaOH in 100 mL of reagent water					√	
Sulfuric acid (H_2SO_4), Concentrated: Reagent Grade					√	
Sulfuric acid (1:1): Carefully add 500 mL of H_2SO_4 to 500 mL of reagent water. Mix well.					√	
Fluorocarbon: PF-5080, 3M						√

7.3. Standards

7.3.1. Stock Standards

Stock standards are purchased as certified solutions or prepared from neats. Semivolatile stock standards are stored at ≤ 6°C. All stock standards must be protected from light. Stock standard solutions must be replaced after one year (from the time of

preparation, if prepared in house, or from the time the ampoule is opened if purchased.) Standards must be allowed to come to room temperature before use.

7.3.2. Surrogate Spiking Standards

Prepare or purchase surrogate spiking standards at the concentrations listed in Table 5. Surrogate spiking standards are purchased or prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.3.3. Matrix Spiking and Laboratory Control Spiking Standards.

The same spiking solution is used for the matrix spike and the Laboratory Control Sample. Prepare MS/LCS spiking standards at the concentrations listed in Table 6. Spiking standards are purchased or prepared as dilutions of the stock standards. Surrogate spiking solutions must be refrigerated and protected from light. The standards must be replaced at least every six months or sooner if there is reason to believe that the standard has degraded or concentrated.

7.3.4. GPC calibration solution - prepare or purchase a solution in methylene chloride that contains the following analytes in the concentrations listed below:

Analyte	mg/mL
Corn Oil	25.0
Bis (2-ethylhexyl) phthalate	1.0
Methoxychlor	0.2
Perylene	0.02
Sulfur	0.08

NOTE: Sulfur is not very soluble in methylene chloride, however, it is soluble in warm corn oil. Therefore, one approach is to weigh out the corn oil, warm it, and transfer the weighed amount of sulfur into the warm corn oil. Mix it and then transfer into a volumetric flask with methylene chloride, along with the other calibration compounds. This standard has a lifetime of 6 months.

8. SAMPLE COLLECTION PRESERVATION AND STORAGE

8.1. Samples are not chemically preserved.

8.2. Samples are stored at 4°C ± 2°C in glass containers with Teflon®-lined caps.

8.3. Holding Times

8.3.1 Extraction is initiated within 7 days of the sampling date for aqueous samples, 14 days for solid and waste samples.

8.3.2 For TCLP leachates, extraction is initiated within seven days from when the TCLP Leach tumbling has been completed, excluding the filtration step. If the filtration step requires extended times, this time counts as part of the seven day holding time.

8.3.3. Analysis of the extracts is completed within forty days of extraction.

9. QUALITY CONTROL

9.1. Quality Control Batch

The batch is a set of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The batch must contain a method blank, an LCS and a matrix spike / matrix spike duplicate. (In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS / MSD). If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD. See policy QA-003 for further definition of the batch.

9.2. Definition of matrix

The possible matrix types are aqueous, soil, waste and TCLP leachate.

9.3. Insufficient Sample

If insufficient sample is available to process a MS/MSD, then a second LCS must be processed. The LCS pair is then evaluated according to the MS/MSD criteria. Use of a LCS pair in place of a MS/MSD must be documented.

9.4. Sample count

Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count. Field samples are included.

9.5. Method Blank

A method blank consisting of all reagents added to the samples must be prepared and analyzed with each batch of samples. Surrogates are spiked into the method blank at the same level as the samples. The method blank is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

9.5.1. Aqueous Method Blanks use 1000 mL of reagent water spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.5.2. Solid method blanks use 30 g of sodium sulfate spiked with the surrogates. The method blank goes through the entire analytical procedure, including any cleanup steps.

9.5.3. TCLP method blanks use 250 mL of leachate fluid (100 mL for herbicides) spiked with the surrogates. The leachate may optionally be diluted to 1000 mL with reagent water.

The method blank goes through the entire analytical procedure, including any cleanup steps.

9.6. Laboratory Control Sample (LCS)

Laboratory Control Samples are well-characterized, laboratory generated samples used to monitor the laboratory's day to day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision. The LCS goes through the entire analytical procedure, including any cleanup steps.

9.6.1. The LCS is made up in the same way as the method blank (See sections 9.5.1 - 9.5.3) but spiked with the LCS standard and the surrogates.

9.7. Surrogates

9.7.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples.

9.7.2. Each applicable sample, blank, LCS and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

9.8. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second spiked aliquot of the same sample which is prepared and analyzed along with the sample and matrix spike.

9.9. Initial Demonstration of Capability

The initial demonstration and method detection limit studies described in section 13 must be acceptable before analysis of samples may begin.

9.10. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

9.11. STL North Canton QC Program

Further details of QC and corrective action guidelines are presented in the STL North Canton QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION AND STANDARDIZATION

10.1. On a weekly basis, measure 1.0 mL of solvent into an autovial using a gastight syringe that is manufactured to a certified volume delivery tolerance of ±0.01 mL (1mL total volume). The "standard" autovial is sealed and the top and bottom of the meniscus are marked. The autovials containing the sample extracts are then compared against the "standard" vial to ensure that the final volume is consistently 1.0 ± 0.01mL. If a new box of autovials are used, then the steps are repeated to further ensure that variations due to vial size and shape are minimized. A log is kept of the lot number of the vials and the day the vials were prepared.

10.2. Refer to section 11.8.1.2. for calibration of the GPC. Otherwise this section is not applicable.

11. PROCEDURE

Procedures for separatory funnel liquid/liquid extraction (11.2), continuous liquid/liquid extraction (11.3), sonication extraction (11.4), soxhlet extraction (11.5), waste dilution (11.6), extract concentration (11.7), and extract cleanup (11.8) are presented in this section.

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file. Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Separatory Funnel Liquid/Liquid Extraction of Water Samples.

A flow chart for this procedure is included in Section 17.

11.2.1. Measure the initial sample pH with wide-range pH paper and record on the extraction benchsheet. If sample is a leachate (e.g. TCLP), compare the current pH against leachate log, note on the benchsheet if there is any discrepancy.

11.2.2. The normal sample volume is 1 liter. Other sample volumes may be used to obtain specific reporting limits, and reduced sample volumes, diluted to 1 liter with reagent water, may be used for very dirty samples.

11.2.3. Weigh the sample container on a balance (± 1 g), taring the sample and container. Transfer the sample to the separatory funnel. . Add the appropriate volume of surrogate spiking solution (see Table 3). Also add appropriate volume of matrix spiking solution to any matrix spike / matrix spike duplicate samples (see.Table 4) Mix well.

If the sample is a 600 series analysis rinse the sample bottle with 60 mL methylene chloride and transfer to the separatory funnel. Reweigh the container. Assume a density of 1 g/mL and record the difference as the sample volume on the benchsheet to the nearest milliliter.

Note: Aqueous samples must be determined volumetrically for Ohio VAP samples.

Warning: Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the sample container has been sealed and inverted. Vent into hood away from analysts and other samples.

11.2.3.1. If the entire sample bottle will not be used, transfer the aliquot to the separatory funnel, then add the spiking solutions to the sample in the separatory funnel.

Note: Alternative methods of measurement of sample volume include a) transferring the sample to a measuring cylinder and b) marking a meniscus on the sample bottle and then measuring the volume of water required to fill the bottle to the meniscus after the sample is transferred. The former method is not recommended because of the risk of cross contamination while the latter is not recommended because of poor accuracy. However, either method may be necessary for specific client programs.

11.2.4. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP. Use 1 L of reagent water for method blanks and LCS. The LCS is spiked with the surrogate and matrix spike solutions, the method blank only with the surrogates.

11.2.5. Use 250 mL of leachate for TCLP pesticides and TCLP semivolatiles, measured in a graduated cylinder. An alternative method would be to tare 400ml beaker on the balance and then add 250g to the beaker. Assume a density of 1g/mL and record the volume on the benchsheet. The leachate may be made up to 1 L in volume with reagent water.

11.2.6. For a TCLP method blank,LCS and LCS Dup measure 250 mL of the buffer solution used in the leaching procedure and transfer to the separatory funnel. Add 60 mL of methylene chloride to the separatory funnel. The TCLP leachate may be diluted to approximately 1 liter before extraction if desired.

11.2.7. Adjust sample pH as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH necessary. Recheck the sample with pH paper by dipping a disposable pipette into the sample and wetting the pH paper. Record adjusted pH, spiking volumes and standard numbers on the benchsheet. Return spiking solutions to the refrigerator as soon as possible.

11.2.8. Seal and shake or rotate the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. An autoshaker may be used to shake and rotate the separatory funnel.

Warning: Dichloromethane creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.

11.2.9. Allow the organic layer to separate from the water phase until complete visible separation has been achieved (approximately 10 minutes). If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. If the emulsion cannot be broken (recovery of <80% of the methylene chloride*), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in continuous liquid-liquid extraction (Section 11.2.). If this is done, the sample must be extracted as part of a valid CLLE batch.

*Note: 15 - 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 35 mL from the first shake and still be acceptable. Subsequent shakes should recover at least 50 mL of solvent.

11.2.10. Fill a funnel with 10-20 g of anhydrous sodium sulfate. The funnel can be plugged with glass wool or filter paper may be used to hold the sodium sulfate. Drain the solvent extract from the separatory funnel through the prepared filtration funnel into a clean glass container. The extract may be drained directly into the KD flask. Close the stopcock just before the water level begins draining out of the separatory funnel. If the sodium sulfate becomes saturated with water add more to the funnel or replace the existing sodium sulfate with fresh drying agent.

11.2.11. Repeat the extraction process two more times using fresh 60 mL portions of solvent, combining the three solvent extracts in the collection container.

11.2.12. If extraction at a secondary pH is required, adjust the pH of the sample in the separatory funnel to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure with pH paper and record the adjusted pH on the benchsheet. Serially extract with three 60 mL portions of methylene chloride, as outlined in Steps 11.1.7 to 11.1.9. Collect these three extracts in the same container used for the previous fraction.

11.2.13. Rinse the extract residue from the sodium sulfate by pouring 20-30 mL of clean methylene chloride through the funnel and into the collection container.

11.2.14. Dispose of solvent and water remaining in the extractor into the appropriate waste container.

11.2.15. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.7 for concentration and Section 11.8 for cleanup.

11.3. Continuous Liquid/Liquid Extraction from Water Samples.

A flow chart for this procedure is included in Section 17.

11.3.1. Assemble the apparatus. Add 250-400mL of methylene chloride to the extractor body. Add 3 to 5 boiling chips to the round-bottom distilling flask.

11.3.2. Measure the initial sample pH with wide-range pH paper and record on the extraction benchsheet. If sample is a leachate (e.g. TCLP), compare the current pH against leachate log, note on the benchsheet if there is any discrepancy.

11.3.3. Weigh the sample container on a balance (± 1 g), taring the sample and container. Transfer the sample to the continuous liquid/liquid extractor . Add the appropriate volume of surrogate spiking solution (see Table 3). Also add appropriate volume of matrix spiking solution to any matrix spike / matrix spike duplicate samples (see.Table 4) Mix well. If the sample is a 600 series analysis rinse the sample bottle with 60 mL methylene chloride and transfer to the continuous liquid/liquid extractor. Reweigh the container. Assume a density of 1 g/mL and record the difference as the sample volume on the benchsheet to the nearest milliliter.

Note: Aqueous samples must be determined volumetrically for Ohio VAP samples.

Warning: Dichloromethane creates excessive pressure very rapidly! Therefore, during the rinse for 600 series methodology venting should be done immediately after the sample container has been sealed and inverted. Vent into hood away from analysts and other samples.

11.3.3.1. If the entire sample bottle will not be used, transfer the aliquot to the extractor, then add the spiking solutions to the sample in the extractor.

Note: Alternative methods of measurement of sample volume include: a.) transferring the sample to a measuring cylinder and b.) marking a meniscus on the sample bottle and then measuring the volume of water required to fill the bottle to the meniscus after the sample is transferred. The former method is not recommended because of the risk of cross contamination while the latter is not recommended because of poor accuracy. However, either method may be necessary for specific client programs.

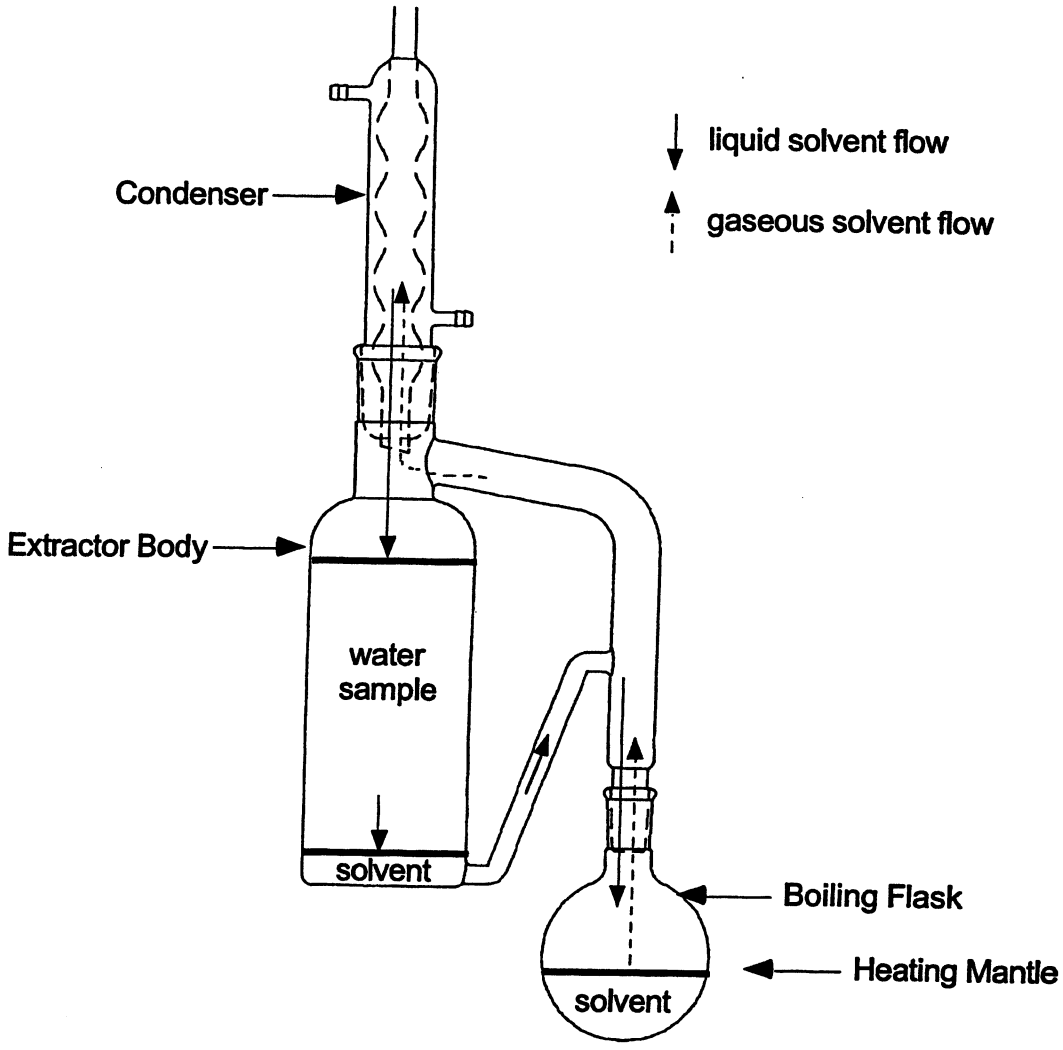
11.3.3.2. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP. Use 1 L of reagent water for method blanks and LCS. The method blank is spiked with the surrogates, the LCS and matrix spikes with the surrogates and matrix spiking solutions..

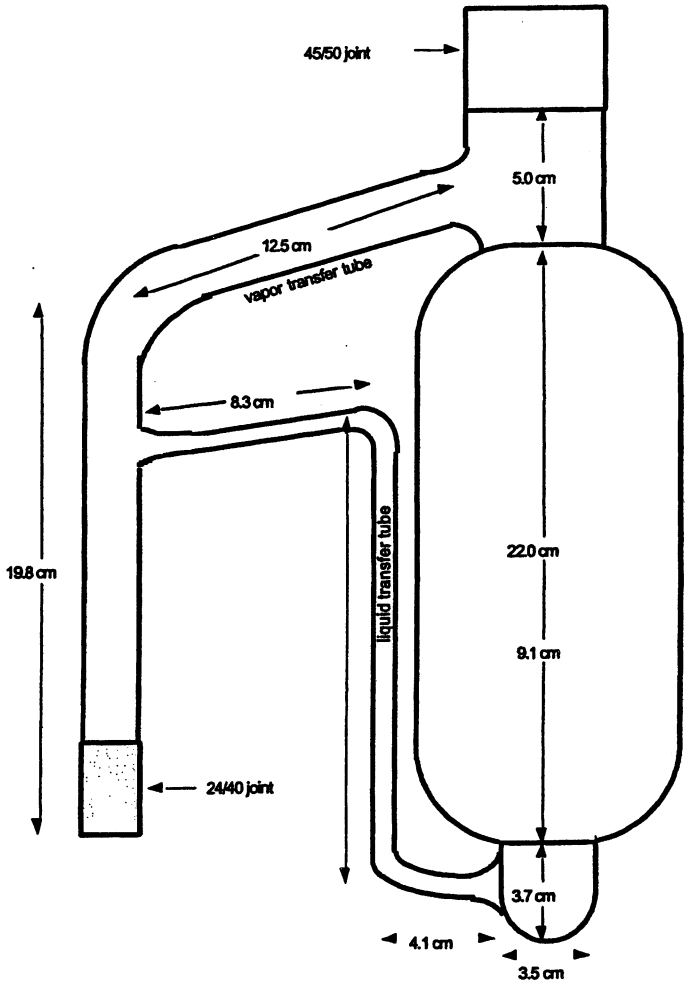
11.3.3.3. Use 250mL of leachate for TCLP for TCLP semivolatiles and TCLP pesticides measured in a graduated cylinder. An alternative method would be to tare 400ml beaker on the balance

and then add 250g to the beaker. Assume a density of 1g/mL and record the volume on the benchsheet. Dilute to about 1 liter with reagent water.

11.3.3.4. For a TCLP method blank, LCS and LCS Dup measure 250 mL of the buffer solution used in the leaching procedure and transfer to the separatory funnel. Dilute to about 1 liter with reagent water.

11.3.3.5. Less than one liter of sample may be used, for highly contaminated samples, or if the reporting limit can be achieved with less than one liter of sample. In this event dilute the sample to about 1 liter with reagent water.




45/50 joint
5.0 cm
12.5 cm
vapor transfer tube
8.3 cm
19.8 cm
liquid transfer tube
22.0 cm
9.1 cm
24/40 joint
3.7 cm
4.1 cm
3.5 cm

11.3.4. Adjust sample pH as indicated in Table 1 for the initial extraction. Use the minimum amount of 1:1 H_2SO_4 or 10 N NaOH necessary. Recheck the sample with pH paper. Record adjusted pH, spiking volumes and standard numbers on the benchsheet. Return spiking solutions to the refrigerator as soon as possible.

11.3.5. Add reagent water to the extractor body until approximately 250 mL of methylene chloride is pushed over into the round-bottomed flask to ensure proper operation and solvent cycling. Attach cold condenser (about 10°C). Turn on heating mantle. Inspect joints for leaks once solvent has begun cycling. Extract for 18-24 hours. (24 hours required for 600 series)

11.3.6. If extraction at a secondary pH is required, (see Table 1) turn off the heating mantle and allow the extractor to cool. Detach the condenser and adjust the pH of the sample in the extractor body to the pH indicated in Table 1 with a minimum amount of 10 N NaOH or 1:1 H_2SO_4. Measure with pH paper and record the adjusted pH on the benchsheet. Reattach the condenser and turn on heating mantle. Extract for 18-24 hours.

11.3.7. Turn off the heating mantle and allow the extractor to cool.

11.3.8. Place a funnel containing 10-20 g of anhydrous sodium sulfate on the Kuderna-Danish (K-D) apparatus or other glass container. The funnel can be plugged with glass wool enabling it to hold the granular anhydrous sodium sulfate or phase separation filter paper may be used.

11.3.9. Dry the extract in the round bottom flask by filtering it through the sodium sulfate filled funnel. Note that it is not necessary or advisable to attempt to add the solvent remaining in the continuous extractor body to the extract.

11.3.10. Collect the dried extract in a K-D or other glass container. Rinse the flask which contained the solvent extract with 20-30 mL of methylene chloride and add it to the funnel to complete the quantitative transfer. Dispose of solvent and water remaining in the extractor in the appropriate waste container.

Note: Some types of CLLE apparatus have built in drying columns. If this type of apparatus is used then a drying step subsequent to the extraction may not be necessary.

11.3.11. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.7 for concentration and Section 11.8 for cleanup.

11.4. Sonication

A flow chart for this procedure is included in Section 17.

11.4.1. Determination of percent moisture (Optional - if a different group performs this test, refer to the facility SOP.)

In some cases, sample results are needed on a dry weight basis. If this is the case, weigh 5-10 g of sample into a suitable tared container (typically an aluminum weigh pan. Determine the % moisture by drying overnight (at least 12 hours) at 105°C. Allow to cool in a desiccator before weighing.

$$\% \text{ Moisture} = \frac{(\text{weight of wet sample} - \text{weight of dry sample})}{\text{weight of wet sample}} \times 100$$

11.4.2. Determination of pH (Optional - if a different group performs this test, refer to the facility SOP.)

If pH determination is required, transfer 10 g of soil to a beaker. Add 10 mL of water. Stir for five minutes then let stand for 1 hour. Determine the pH of the sample with a glass electrode and pH meter.

11.4.3. Decant and discard any water layer on a sediment/soil sample. Record and document if a water layer was discarded on the benchsheet. Homogenize the sample by mixing it thoroughly in the container. If this is not possible place the sample in clean beaker and homogenize. Upon completion of homogenization in beaker return sample to original container. Discard foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by client. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator).

11.4.4. 11.4.4. Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

11.4.5. Weigh 30 g of sample ± 0.2g into a 250 or 400 mL beaker. Record the weight to the nearest 0.01 g in the appropriate column on the benchsheet. Use 30 g of sodium sulfate for the method blank and 30 g of sodium sulfate with 30 g of reagent sand for the LCS.

11.4.6. Mix weighed sample with a spatula adding enough anhydrous sodium sulfate (approximately 30 g) to be free flowing. (If the sample is not free flowing extraction efficiency may be reduced)

11.4.7. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP.

11.4.8. Add the appropriate volume of surrogate spiking solution (see Table 3). Also add appropriate volume of matrix spiking solution to any matrix spike / matrix spike

duplicate samples (see.Table 4) Add 1 mL of the surrogate spiking solution to each sample, method blank, Laboratory Control Sample (LCS), and matrix spikes. Refer to Table 6 for details of the surrogate spiking solutions. Add 1 mL of the appropriate matrix spiking solution to each Matrix Spike/Matrix Spike Duplicate (MS/MSD) and LCS. Refer to Tables 3 and 5 for details of the spiking solutions. Record spiking volumes and standard numbers on the benchsheet. Return spiking solutions promptly to refrigerator.

Note: The same volume of surrogate and matrix spiking solution is used if GPC is indicated since the final volume would be reduced to compensate for loss of extract during the GPC procedure.

11.4.9. Immediately add a minimum of 100 mL of solvent to the beaker.

Solvents:

Semivolatile GC/MS Organochlorine pesticides and PCBs	1:1 Methylene Chloride / Acetone
Hydrocarbons (DRO, PAH, etc.) and OPPs	Methylene Chloride

Note: Steps 11.4.5 - 11.4.9 should be performed rapidly to avoid loss of the more volatile extractables.

11.4.10. Place the bottom surface of the appropriate disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

11.4.11. Sonicate for 3 minutes, making sure the entire sample is agitated. If the W-380 or W-385 sonicator is used the output should be set at 6 for the 3/4 inch high gain (Q) horn or 10 for the 3/4 inch standard horn. with mode switch on pulse, and percent-duty cycle knob set at 50%.

Note: Do ***not*** use ***Microtip*** probe.

11.4.12. Loosely plug the stem of a 75 mm x 75 mm glass funnel with glass wool and/or line the funnel with filter paper. Add 10-20 g of anhydrous sodium sulfate to the funnel cup.

11.4.13. Place the prepared funnel on a collection apparatus (beaker or K-D Apparatus.

11.4.14. Decant and filter extracts through the prepared funnel into a clean beaker or K-D Apparatus.

11.4.15. Repeat the extraction two more times with additional 100 mL minimum portions of solvent each time. Decant off extraction solvent after each sonication. On the final sonication pour the entire sample (sediment and solvent) into the funnel and rinse with an additional 10 mL-20 mL of the methylene chloride/acetone appropriate solvent (Refer to Table in 11.4.9).

Note: Alternatively, the three extracts may be collected together and then filtered through the sodium sulfate.

11.4.16. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.7 for concentration and Section 11.8 for cleanup.

11.4.17. Sonicator Tuning.

11.4.17.1. Tune the sonicator according to manufacturer's instructions. The sonicator must be tuned at least every time a new horn is installed.

11.5. Soxhlet

11.5.1. Determination of % moisture

In some cases, sample results are needed on a dry weight basis. If this is the case, weigh 5-10 g of sample into a suitable tared container (typically an aluminum weigh pan. Determine the % moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing.

$$\% \text{ Moisture} = \frac{(\text{weight of wet sample} - \text{weight of dry sample})}{\text{weight of wet sample}} \times 100$$

11.5.2. Determination of pH

If pH determination is required, transfer 10 g of soil to a beaker. Add 10 mL of water. Stir for 1 hour. Determine the pH of the sample with a glass electrode and pH meter.

11.5.3. Decant and discard any water layer on a sediment/soil sample. Record and document if a water layer was discarded on the benchsheet. Homogenize the sample by mixing it thoroughly in the container. If this is not possible place the sample in clean beaker and homogenize. Upon completion of homogenization in beaker return sample to original container. Discard foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by client. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator).

11.5.4. Remove surrogate and matrix spiking solutions from refrigerator and allow to warm to room temperature.

11.5.5. Weigh 30 g of sample ± 0.2g into a beaker, recording the weight to the nearest 0.01 g on the benchsheet.. Use 30 g of sodium sulfate for the method blank and LCS. Add 30 g of anhydrous sodium sulfate and mix well. The mixture should have a free flowing texture. If not, add more sodium sulfate. Add the sample/sodium sulfate mixture to a soxhlet thimble, but do not pack the thimble tightly. The extraction thimble must drain freely for the duration of the extraction

period. A glass wool plug above and below the sample in the soxhlet extractor is an acceptable alternative for the thimble.

11.5.6. Add the appropriate amount of surrogate and matrix spiking solution as indicated in Tables 3, 4, 5, and 6.

Sample weights less than 30 g but over 5 g may be used if the appropriate reporting limits can be met.

11.5.7. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP, using sodium sulfate as the matrix. The weight of sodium sulfate used should be approximately the weight of soil used in each sample.

11.5.8. Place approximately 200mL of solvent into a 250 mL flat bottom flask containing one or two clean boiling chips. Attach the flask to the extractor and extract the sample for 16-24 hours at 4-6 cycles per hour. Check the system for leaks at the ground glass joints after it has warmed up.

NOTE: If a reduced quantity of sample is extracted, it is usually necessary to increase the amount of sodium sulfate added or increase the solvent boiling rate to properly set the cycling rate.

Solvents:

Semivolatile GC/MS Organochlorine pesticides and PCBs	1:1 Methylene Chloride / Acetone
Hydrocarbons (DRO, PAH, etc.) and OPPs	Methylene Chloride

11.5.9. Allow the extract to cool after the extraction is complete, then disassemble by gently twisting the soxhlet from the flask. Dry the extract in the flask by filtering it through a sodium sulfate filled funnel.

11.5.10. Collect the dried extract in a K-D or other glass container. Rinse the flask which contained the solvent extract with 20-30 mL of methylene chloride and add it to the funnel to complete the quantitative transfer.

11.5.11. Cover with aluminum foil if the extract is not concentrated immediately. Refer to Section 11.7 for concentration and Section 11.8 for cleanup.

11.6. Waste Dilution

11.6.1. This method is used for materials that are soluble in an organic solvent.

11.6.2. Transfer 10 mL of the solvent to be used for dilution into a Teflon capped vial. Mark the meniscus on the vial, then discard the solvent.

11.6.3. Tare the vial, then transfer approximately 1g of sample to the vial. Record the weight to the nearest 0.01 g.

11.6.4. Add 1 mL appropriate volume of surrogate solution (Table 3). 11.6.5. Dilute to 10 mL with the appropriate solvent. (Methylene Chloride for GC/MS and Hexane for pesticides pesticide and PCB analysis.)

11.6.6. Add 2 g + 0.1 g sodium sulfate to the sample. Cap and shake for 2 minutes.

11.6.7. Add 4-5 g sodium sulfate to a small funnel. The funnel can be plugged with glass wool or phase separation filter paper may be used to hold the sodium sulfate.

11.6.8. Pour the sample through the funnel, collecting as much as possible in a clean vial. Do NOT rinse the funnel with additional solvent, and do NOT concentrate the sample. The final volume is defined as 10 mL.

11.6.9. Label the sample, which is now ready for cleanup or analysis.

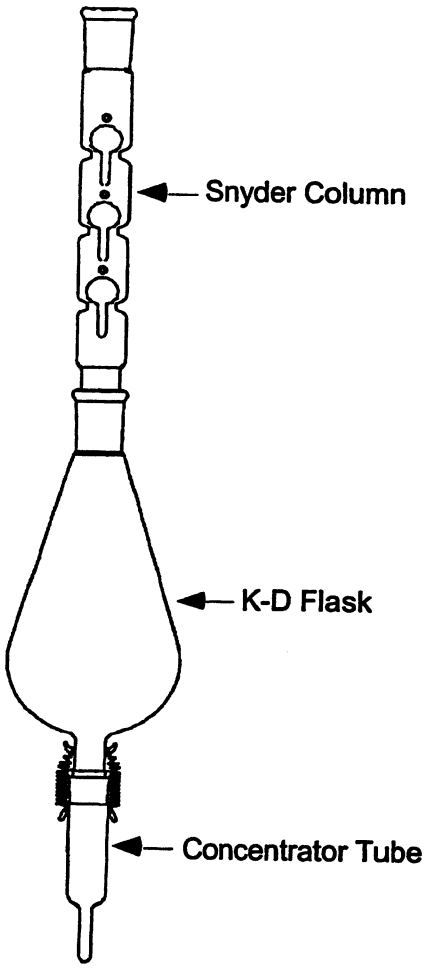
11.7. Concentration

According to the type of sample and any cleanup procedures needed, different final solvents and volumes will be required. Refer to Table 2 for the appropriate final volumes and concentrations.

11.7.1. Kuderna-Danish (KD) Method:

11.7.1.1. Assemble a Kuderna-Danish concentrator by attaching a 10 mL concentrator tube to the 500 mL KD flask. Transfer the sample to the K-D flask.

11.7.1.2. Add one or two clean boiling chips and the extract to be concentrated to the KD flask and attach a three ball Snyder Column. Add approximately 1 mL of clean methylene chloride to the top of the Snyder column (this is important to ensure that the balls are not stuck and that the column will work properly).



11.7.1.3. Place the KD apparatus on a water bath (90-98°C) so that the tip of the concentrator tube is submerged. The water level should not reach the joint between the concentrator and the KD flask. At the proper rate of distillation, the balls will actively chatter but the chambers should not flood.

11.7.1.4. Concentrate to 5-15 mL. If the determinative method requires a solvent exchange add the appropriate exchange solvent (50 mL hexane, 20 ml hexane/acetone, or 4 mL acetonitrile,4mL toluene (Herbicide TCLP), or 10 mL toluene) to the top of the Snyder Column, and then continue the water bath concentration back down to 1-4 mL. Refer to

Table 2 for details of exchange solvents and final volumes. The Snyder column may be insulated if necessary to maintain the correct rate of distillation.

Note: Add an additional boiling chip with the addition of exchange solvent. An alternative technique for solvent exchange is to replace the macro Snyder column and KD flask with a micro Snyder column, concentrate to approximately 1 mL, add the appropriate solvent, and concentrate back down to 1 mL. The extract must be cool before the macro Snyder assembly is removed.
Note: It is very important not to concentrate to dryness as analytes will be lost.

11.7.1.5. Remove the KD apparatus from the water bath and allow to cool for a minimum of 10 minutes. If the level of the extract is above the level of the concentrator tube joint, continue to distill the solvent as necessary. Again, allow the KD flask to cool for a minimum of 10 minutes.

11.7.1.6. If the final volume is 5 or 10 mL the extract may be made up to volume in the graduated KD tube or transferred to a 12 mL vial previously marked at the appropriate volume level. Document the final volume. Otherwise proceed to section 11.7.2

11.7.2. Nitrogen Evaporation to Final Concentration

11.7.2.1. Transfer the entire extract to a calibrated evaporation tube. Rinse the concentrator tube with 1-2 mL of the appropriate solvent and transfer the solvent rinsate to the evaporation tube.

11.7.2.2. Place the tube in a warm water bath that is at least 5°C below the boiling temperature of the solvent being evaporated and evaporate the solvent using a gentle stream of nitrogen. The nitrogen flow will form a slight depression on the surface of the solvent, but should not create splattering of the extract.

Boiling points of commonly used solvents are:

Methylene chloride	40°C
Acetone	56°C
Hexane	69°C
Acetonitrile	82°C
Toluene	111°C

11.7.2.3. During the course of the evaporation rinse the sides of the evaporation tube twice with approximately 1 mL of clean solvent. The first rinse should be about half way through the process, with the second rinse when the solvent volume gets close to 1 mL. Concentrate the solvent accurately to the calibrated volume line and transfer the extract to the appropriate storage vial.

Note: It is very important not to concentrate to dryness as analytes will be lost.

11.7.2.4. An alternative technique is to follow the previous steps concentrating the solvent to slightly below the required final volume and then drawing the extract into a syringe. Rinse the evaporation tube with a small amount of solvent and draw additional solvent into the syringe to make up the accurate final volume.

Note: It is very important not to concentrate to dryness as analytes will be lost.

Note: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

Alternative QES Concentration Method:

This concentration method uses a hot water jacketed concentrator tube (CT) instead of the hot water bath and concentrator tube used in Section 11.7.1.1. The construction of the jacketed concentrator tube reduces the tendency of the extract to evaporate to dryness. Thus, low boiling analytes are retained in the extract better with less analyst monitoring of the concentration process.

Assemble the jacketed concentrator tube, KD body and hot water hoses. Add 1 mL of exchange solvent (if needed) and one large, clean boiling chip.

NOTE: The boiling chips used in the jacketed concentrator tube must be large enough to prevent them falling down into the tip of the CT. If the boiling chip is not in the proper position, the extract may superheat and bump vigorously if the extract solvent warms up slowly.

Pour the extract to be concentrated into the KD flask, and attach a three-ball Snyder column.

Turn on the hot (95°C) water flow to the jacketed concentrator tube.

Concentrate to 1 mL. If the determinative method requires a solvent exchange, add the appropriate exchange solvent (5-10 mL hexane, 1 mL acetonitrile, 4 mL acetone/hexane) to the top of the Snyder column. Continue to concentrate until the Snyder column balls stop chattering.

Cool the jacketed concentrator tube until it is cold to the touch.

Quantitatively transfer the extract and dilute to final volume, or continue concentration with nitrogen evaporation (section 11.7.2).

11.7.3. Turbovap Method

11.7.3.1. Turn on the Turbovap and adjust the water temperature to 5-10°C less than the boiling point of the solvent to be evaporated.

11.7.3.2. Switch all endpoint sensors to the "NO" position.

11.7.3.3. Adjust the water bath level

11.7.3.4. Adjust the nitrogen gas pressure to approximately 12 psi.

11.7.3.5. Transfer the extract into the Turbovap tube and load into the Turbovap. Do not fill the Turbovap tubes over approximately 3/4 full.

11.7.3.6. Reset the sensor and close the lid.

Note: If the extract splashes when the nitrogen flow starts, reduce the nitrogen flow or transfer a portion of the extract back into the original extract container.

11.7.3.7. As the extract concentrates, transfer the remainder of the extract into the appropriate Turbovap tube. After all of the extract has been transferred, rinse the flask with a few mL of methylene chloride and add to the Turbovap tube.

11.7.3.8. During the concentration rinse the Turbovap tube walls with a few mL of solvent 1 or 2 times.

11.7.3.9. Concentrate the extract to slightly less than the required final volume.

11.7.3.10. If solvent exchange is required, concentrate to 1-4 mL and add 50 mL of the exchange solvent. Concentrate back down to the appropriate volume. Refer to table 2 for details of exchange solvents and final volumes.

11.7.3.11. Transfer the concentrated extract to volumetric glassware for adjustment of final volume, using a small amount of solvent to rinse the tube and complete the transfer.

Note: Water contamination from condensation during concentration is not acceptable. If water is present, remove the Turbovap tube and filter the extract through sodium sulfate. Transfer to a clean Turbovap tube and continue the concentration.

Note 2: Dark, opaque or turbid samples may not concentrate. If this occurs, set the concentrator sensor to dryness and supervise the entire concentration procedure.

11.8. Cleanup Techniques

The following techniques may be used to remove interfering peaks, and /or to remove materials that may cause column deterioration and/ or loss of detector sensitivity.

Gel Permeation Chromatography (Section 11.8.1) is a generally applicable technique which can be used to prepare extracts for Semivolatiles (8270), and pesticides (8080) analysis. It is capable of separating high molecular weight material from the sample analytes, and so is particularly useful if tissue or vegetable matter is part of the sample, and for many soil samples.
Note: GPC used only for CLP Projects

Florisil column cleanup (Section 11.8.2) is particularly useful for cleanup of pesticides for analysis by method 8080 and should normally be applied to these samples unless the matrix is clean. It separates compounds with a different polarity from the target analytes.
Note: GPC used only for CLP Projects
Gel Permeation Chromatography and Florisil column cleanup may both be applied to samples for analysis by method 8080. In this case the GPC should be performed first.

Sulfur cleanup (Section 11.8.3) is generally applied to samples for analysis by method 8080, since the Electron Capture Detector responds strongly to sulfur. It is performed after GPC and Florisil cleanup.

Sulfuric acid cleanup (Section 11.8.4) is applied to samples requiring analysis for Polychlorinated Biphenyls (PCBs) only. Most organic matter is destroyed by the sulfuric acid.
WARNING: Sulfuric acid cleanup must not be performed on any matrix that may have water present as a violent reaction between the acid and water may result in acid exploding out of the vessel.

Acid Base Partition Cleanup (Section 11.8.5) is useful for separating organic acids and phenols from basic and neutral organics.

Fluorocarbon cleanup (Section 11.8.7) is used to remove hydrocarbons from water samples to be analyzed for water soluble alcohols.

11.8.1. Gel Permeation Chromatography (GPC)

Note: GPC systems include the GPC Autoprep Model 1002A or 1002B Analytical Biochemical Laboratories, Inc., Zymark Benchmate, or equivalent.

11.8.1.1. GPC Column Preparation

11.8.1.1.1. Weigh out 70 g of Bio Beads (SX-3) into a 400-mL beaker.

11.8.1.1.2. Add approximately 300 mL of methylene chloride and stir gently.

11.8.1.1.3. Cover with aluminum foil and allow the beads to swell for a minimum of two hours. Maintain enough solvent to sufficiently cover the beads at all times.

11.8.1.1.4. Position and tighten the outlet bed support (top) plunger assembly in the tube by inserting the plunger and turning it clockwise until snug. Install the plunger near the column end but no closer than 5 cm (measured from the gel packing to the collar).

11.8.1.1.5. Turn the column upside down from its normal position with the open end up. Place the tubing from the top plunger assembly into a waste beaker below the column.

11.8.1.1.6. Swirl the bead/solvent slurry to get a homogeneous mixture and pour the mixture into the open end of the column. Transfer as much as possible with one continuous pour trying to minimize bubble formation. Pour enough to fill the column. Wait for the excess solvent to drain out before pouring in the rest. Add additional methylene chloride to transfer the remaining beads and to rinse the beaker and the sides of the column. If the top of the gel begins to look dry, add more methylene chloride to rewet the beads.

11.8.1.1.7. Wipe any remaining beads and solvent from the inner walls of the column with a laboratory tissue. Loosen the seal slightly on the other plunger assembly (long plunger) and insert it into the column. Make the seal just tight enough so that any beads on the glass surface will be pushed forward, but loose enough so that the plunger can be pushed forward.

CAUTION: Do not tighten the seal if beads are between the seal and the glass surface because this can damage the seal and cause leakage.

11.8.1.1.8. Push the plunger until it meets the gel, then compress the column bed about 4 cm.

11.8.1.1.9. Connect the column inlet to the solvent reservoir and place the column outlet tube in a waste container. Pump methylene chloride through the column at a rate of 5 mL/min. for one hour.

11.8.1.1.10. After washing the column for at least one hour, connect the column outlet tube to the inlet side of the UV detector. Connect the system outlet to the outlet side of the UV detector. Placing a restrictor (made from a piece of capillary tubing of 1/16"OD x 10/1000"ID x 2") in the outlet tube from the UV detector will prevent bubble formation which causes a noisy UV baseline. The restrictor will not effect the flow rate. After pumping methylene chloride through the column for an additional 1-2 hours, adjust the inlet bed support plunger until approximately 6-10 psi back-pressure is achieved. Push the plunger in to increase pressure or slowly pull outward to reduce pressure.

11.8.1.1.11. When the GPC column is not to be used for several days, connect the column inlet and outlet lines to prevent column drying and/or

channeling. If channeling occurs, the gel must be removed from the column, re-swelled, and re-poured as described above. If drying occurs, pump methylene chloride through the column until the observed column pressure is constant and the column appears wet. Always recalibrate after column drying has occurred to verify that retention volumes have not changed.

11.8.1.2. Initial Calibration of the GPC Column

11.8.1.2.1. Before use, the GPC must be calibrated based on monitoring the elution of standards with a UV detector connected to the GPC column.

11.8.1.2.2. Pump solvent through the GPC column for 2 hours. Verify that the flow rate is 4.5-5.5 mL/min. Corrective action must be taken if the flow rate is outside this range. Record the column pressure (should be 6-10 psi) and room temperature (22°C is ideal).

Note: Changes in pressure, solvent flow rate, and temperature conditions can affect analyte retention times and must be monitored. If the flow rate and/or column pressure do not fall within the above ranges, a new column should be prepared.

11.8.1.2.3. Inject the calibration solution and retain a UV trace that meets the following requirements (See resolution calculation in section 11.8.1.6):

- Peaks must be observed and should be symmetrical for all compounds in the calibration solution.
- Corn oil and phthalate peaks must exhibit >85% resolution.
- Phthalate and methoxychlor peaks must exhibit >85% resolution.
- Methoxychlor and perylene peaks must exhibit >85% resolution.
- Perylene and sulfur peaks must not be saturated and must exhibit >90% baseline resolution.

11.8.1.2.4. A UV trace that does not meet the criteria in paragraph 11.8.1.2.3 indicates the need for system maintenance and/or the need for a new column.

11.8.1.2.5. Determine appropriate collect and dump cycles.

11.8.1.2.6. The calibrated GPC program for pesticides/PCB should dump >85% of the phthalate and should collect >95% of the methoxychlor and perylene. Use a wash time of 10 minutes.

11.8.1.2.7. For semivolatile extracts, initiate a column eluate collection just before the elution of bis (2-ethylhexyl) phthalate and after the elution of the corn oil. Stop eluate collection shortly after the elution of perylene. Stop collection

before sulfur elutes. Use a wash time of 10 minutes after the elution of sulfur.

11.8.1.2.8. Reinject the calibration solution after appropriate dump and collect cycles have been set.

11.8.1.2.9. Measure and record the volume of collected GPC eluate in a graduated cylinder.

11.8.1.2.10. The retention times for both bis(2-ethylhexyl) phthalate and perylene must not vary more than +/- 5% between calibrations.

11.8.1.3. GPC calibration check

Check the calibration of the GPC immediately after the initial calibration and at least every 7 days thereafter, while the column is in use.

11.8.1.3.1. Inject the calibration solution, and obtain a UV trace. If the retention times of bis(2-ethylhexyl)phthalate or perylene have changed by more than ± 5% use this run as the start of a new initial calibration. Otherwise, proceed with the recovery check. Excessive retention time shifts may be caused by poor laboratory temperature control or system leaks, an unstabilized column, or high laboratory temperature causing outgassing of methylene chloride. Pump methylene chloride through the system and check the retention times each day until stabilized.

11.8.1.4. GPC Recovery Check for Pesticides/ PCBs

11.8.1.4.1. The recovery from the GPC must be verified immediately after the initial calibration and at least every 7 days, when the instrument is in use. Two recovery check solutions are used. The first mixture is prepared by diluting 1.0 2.0 mL of the pesticide matrix spiking solution (Table 6) to 10 mL in methylene chloride. The second mixture is prepared by diluting 12.0 mL of the PCB only matrix spiking solution (Table 6) to 10 mL with methylene chloride.

11.8.1.4.2. Load the pesticide matrix spike mixture, the PCB mixture, and a methylene chloride blank onto the GPC using the GC dump and collect values.

Note: If the analysis is for PCBs only, then the pesticide recovery check is not necessary.

11.8.1.4.3. After collecting the GPC calibration check fraction, concentrate, solvent exchanging to hexane. Adjust the final volume to 5.0 mL, and analyze by GC/EC. Refer to concentration, section 11.7.

11.8.1.4.4. The methylene chloride blank may not exceed more than one half the reporting limit of any analyte. And if the recovery of each of the single component analytes is 80-10120% and if the Aroclor pattern is the same as previously run standards, then the analyst may use the column. If the above criteria are not met, there may be a need for system maintenance.

11.8.1.5. GPC Recovery Check for Semivolatiles

11.8.1.5.1. The recovery from the GPC must be verified immediately after the initial calibration and at least every 7 days, when the instrument is in use. Dilute 1.0 mL of the semivolatiles matrix spiking solution (Table 6) to 10 mL in methylene chloride.

11.8.1.5.2. Load the matrix spike mixture and a methylene chloride blank onto the GPC using the semivolatiles dump and collect values.

11.8.1.5.3. After collecting the GPC recovery check fraction, concentrate to 0.5 mL, and analyze by GC/MS. Refer to the concentration section 11.7.

Recovery of the matrix spike analytes should be at least 60%. The blank should not contain any analytes at or above the reporting limit. If these conditions are met the column may be used for sample analysis. Otherwise correct the contamination problem, or extend the collect time to improve recovery of target analytes. THIS IS NOT SPECIFIED OR REQUIRED BY THE SOW.

11.8.1.6. Sample Extract Cleanup

11.8.1.6.1. Reduce the sample extract volume to 1-2 mL, then adjust to 10 mL with methylene chloride prior to cleanup. This reduces the amount of acetone in the extract. Refer to section 11.7.

11.8.1.6.2. Start the pump and let the flow stabilize for 2 hours. The solvent flow rate should be 4.5-5.5 mL/min. The ideal laboratory temperature to prevent outgassing of the methylene chloride is 22°C. The normal backpressure is 6-10 psi.

11.8.1.6.3. In order to prevent overloading of the GPC column, highly viscous sample extracts must be diluted prior to cleanup. Any sample extract with a viscosity greater than that of a 1:1 glycerol:water solution (by visual comparison) must be diluted and loaded into several loops.

11.8.1.6.4. Samples being loaded onto the GPC should be filtered with a 5 micron (or less) filter disk. Attach a filter to a 10 mL Luerlok syringe and filter the 10 mL sample extract into the sample tube.

Note: The Zymark Benchmate automatically filters the sample extracts, but manual prefiltration of very dirty extracts may be necessary.

11.8.1.6.5. Load the filtered (or unfiltered if using the Benchmate) samples into the proper sample tubes and place on the GPC.

Note: For the GPC Autoprep Model 1002A, wash the loading port with methylene chloride after loading each sample loop in order to minimize cross contamination. This step is automated on the GPC Autoprep 1002B and on the Benchmate.

11.8.1.6.6. Set the collect, dump, and wash times determined by the calibration procedure.

11.8.1.6.7. Switch to the run mode and start the automated sequence. Process each sample using the collect and dump cycle times established by the calibration procedure.

11.8.1.6.8. Collect each sample in a suitable glass container. Monitor sample volumes collected.

11.8.1.6.9. Any samples that were loaded into 2 or more positions must be recombined.

11.8.1.6.10. Concentrate semivolatile sample extracts to 0.5 mL in methylene chloride. Refer to the concentration section 11.7.

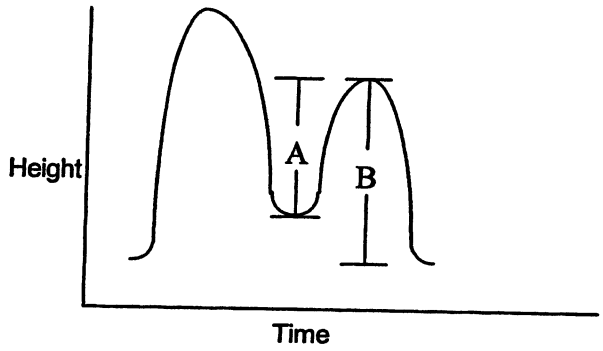
11.8.1.6.11. Solvent exchange pesticide/PCB sample extracts into hexane and concentrate to 5.0 mL. Refer to the concentration section 11.7.

11.8.1.7. Calculations

11.8.1.7.1. Resolution

To calculate the resolution between two peaks on a chromatograph, divide the depth of the valley between the peaks by the peak height of the smaller peak being resolved and multiply by 100.

Resolution Calculation



$$\% \text{ Resolution} = \frac{A}{B} \times 100$$

Where: A = depth of valley to height of smaller peak
B = peak height of smaller peak

11.8.1.7.2. Dump Time

Mark on the chromatograph the point where collection is to begin. Measure the distance from the injection point. Divide the distance by the chart speed. Alternatively the collect and dump times may be measured by means of an integrator or data system.

$$\text{Dump time (min)} = \frac{\text{Distance (cm) from injection to collection start}}{\text{Chart speed (cm / min)}}$$

11.8.1.7.3. Collection Time

$$\text{Collection time (min)} = \frac{\text{Distance (cm) between collection start and stop}}{\text{Chart speed (cm / min)}}$$

11.8.2. Florisil Cartridge Cleanup

Florisil cleanup is generally used for organochlorine pesticides, although it may be applied to other analytes. Sections 11.8.2.1 through 11.8.2.8 outline the procedure for organochlorine pesticides, while section 11.8.2.9 outlines modifications required for other analytes.

Note 1: Systems for eluting multiple cleanup cartridges include the Supelco, Inc. Solid Phase Extraction (SPE) assemblyor equivalent.

Note 2: Follow the lab specific procedure when using ABC Model 1002B.

11.8.2.1. Before Florisil cleanup sample volume must be reduced to 10 mL (5 mL if GPC cleanup was used) and the solvent must be hexane. Refer to Section 11.7 for details of concentration.

11.8.2.2. Attach a vacuum manifold to a vacuum pump or water aspirator with a trap installed between the manifold and the vacuum. Adjust the vacuum in the manifold to 5-10 psi.

11.8.2.3. Place one Florisil cartridge into the vacuum manifold for each sample extract. Prior to cleanup of samples, pre-elute each cartridge with 5 mL of hexane/acetone (9:1). Adjust the vacuum applied to each cartridge so that the flow through each cartridge is approximately 2 mL/min. Do not allow the cartridges to go dry.

11.8.2.4. Just before the cartridges go dry, release the vacuum to the manifold and remove the manifold top.

11.8.2.5. Place a rack of clean labeled 12 mL concentrator tubes into the manifold and replace the manifold top. Make sure that the solvent line from each cartridge is placed inside the appropriate tube.

11.8.2.6. After the clean tubes are in place, vacuum to the manifold is restored and 2.0 mL of the extract is added to the appropriate Florisil cartridge.

11.8.2.7. The pesticides/aroclors in the extract concentrates are then eluted through the column with 8 mL of hexane/acetone (90:10) and are collected into the 10 mL culture tube or concentrator tube held in the rack inside the vacuum manifold.

11.8.2.8. Transfer the extract to a graduated concentrator tube and concentrate the extract to 2 mL. Refer to the concentration section. (11.7)

Note 1: A cartridge performance standard must be run with each lot of Florisil cartridges.

Note 2: Florisil cartridge performance check--every lot number of Florisil must be tested before use. Add 0.5 ug/mL of 2,4,5-trichlorophenol solution and 0.5 mL of GC Standard Mix A (midpoint concentration) to 4 mL hexane. Reduce volume to 0.5 mL. Add the concentrate to a pre-washed Florisil cartridge and elute with 9 mL hexane/acetone [(90:10)(v/v)]. Rinse cartridge with 1.0 mL hexane two additional times. Concentrate eluate to 1.0 mL final volume and transfer to vial. Analyze the solution by GC/EC. The test sample must show 80 to 120% recovery

of all pesticide analytes with <5% trichlorophenol recovery, and no peaks interfering with target compounds can be detected. This standard has a lifetime of six months. Alternatively, this standard may be purchased as a stock solution.

11.8.2.9. Modifications for other analyte classes

11.8.2.9.1. PCBs

Pre-elute the cartridge with 4 mL hexane. Add 2 mL of the sample extract and elute with 3 mL hexane. The eluant will contain the PCBs together with any heptachlor, aldrin, 4,4'DDE and part of any 4,4'DDT. Any BHC isomers, heptachlor epoxide, chlordane, endosulfan I and II, endrin aldehyde and endrin sulfate and methoxychlor will be retained on the column and can be eluted in a separate fraction with 8 mL 90:10 hexane:acetone if required.

11.8.3. Sulfur Removal

11.8.3.1. Sulfur can be removed by one of three methods: mercury, copper, or tetrabutylammonium sulfite (TBA) according to laboratory preference. If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract to settle the crystals, and carefully draw off the sample extract with a disposable pipet, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean concentrator tube before proceeding with further sulfur cleanup.

11.8.3.2. Sulfur Removal with Elemental Mercury

Note: Use Mercury sparingly in order to minimize exposure and disposal costs.

11.8.3.2.1. Transfer 2 mL of sample extract into a clean concentrator tube or Teflon sealed vial.

11.8.3.2.2. Add one to three drops of mercury to the extract vial and seal.

11.8.3.2.3. Shake well for 15-30 seconds. If prolonged shaking is required, use a mechanical shaker.

11.8.3.2.4. Remove the extract from the mercury using a disposable pipette and transfer to a clean vial.

11.8.3.2.5. If black precipitate forms, sulfur was present. Shake again, then centrifuge. After centrifugation, transfer the supernate to a clean test tube and repeat. Do this until relatively little precipitate remains, or the screens indicate that cleanup is complete.

11.8.3.2.6. Properly dispose of the mercury waste.

11.8.3.3. Sulfur Removal with Copper

11.8.3.3.1. Transfer 1.0 mL of sample extract into a centrifuge or concentrator tube.

11.8.3.3.2. Add approximately 2 g of cleaned copper powder (see 7.2 for copper cleaning procedure) to the sample extract tube.

11.8.3.3.3. Mix for one minute on a mechanical shaker.

11.8.3.3.4. If the copper changes color, sulfur was present. Repeat the sulfur removal procedure until the copper remains shiny.

11.8.3.3.5. Transfer the supernate to a clean vial.

11.8.3.4. Sulfur Removal with Tetrabutylammonium (TBA) Sulfite Reagent

11.8.3.4.1. Transfer 1.0 mL of sample extract into a culture tube.

11.8.3.4.2. Add 1.0 mL TBA sulfite reagent and 2 mL 2-propanol to the sample extract. Cap and shake for 1 minute. If clear crystals (precipitated sodium sulfite) form, sufficient sodium sulfite is present.

11.8.3.4.3. If a precipitate does not form, add sodium sulfite in approximately 0.1 g portions until a solid residue remains after repeated shaking.

11.8.3.4.4. Add 5 mL organic free reagent water and shake for 1 minute. Allow sample to stand for 5-10 minutes. (Centrifuge if necessary to separate the layers). Transfer the sample extract (top layer) to a vial. The final volume is defined as 1.0 mL in section 11.8.3.4.1.

11.8.4. Sulfuric Acid Cleanup

11.8.4.1. Add approximately 2-5 mL of concentrated sulfuric acid to 2 mL of sample extract in a Teflon capped vial.

Caution: There must be no water present in the extract or the reaction may shatter the sample container.

11.8.4.2. Shake or vortex for about thirty seconds and allow to settle. (Centrifuge if necessary)

11.8.4.3. Remove the sample extract (top layer) from the acid using a Pasteur pipet and transfer to a clean vial. ***CAUTION:*** It is not necessary to remove all the extract since the final volume is already determined. Transfer of small amounts of sulfuric acid along with the extract will result in extremely rapid degradation of the chromatographic column.

11.8.4.4. If the sulfuric acid layer becomes highly colored after shaking with the sample extract, transfer the hexane extract to a clean vial and repeat the cleanup procedure until color is no longer being removed by the acid, or a maximum of 5 acid cleanups.

11.8.4.5. Properly dispose of the acid waste.

11.8.5. Acid/Base Partition Cleanup

11.8.5.1. Place 10 mL of the solvent extract from a prior extraction procedure into a 125 mL separatory funnel.

11.8.5.2. Add 20 mL of methylene chloride to the separatory funnel.

11.8.5.3. Slowly add 20 mL of DI water which has been previously adjusted to a pH of 12 to 13 with 10 N sodium hydroxide.

11.8.5.4. Seal and shake the separatory funnel for at least two minutes with periodic venting to release excess pressure.

CAUTION: Methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the separatory funnel has been sealed.

11.8.5.5. Allow the organic layer to separate from the aqueous phase for a minimum of ten minutes.

11.8.5.6. If an emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods.

11.8.5.7. Separate the aqueous phase and transfer it to a 125 mL Erlenmeyer flask. Repeat the extraction two more times using fresh 20 mL aliquots of dilute sodium hydroxide (pH 12-13). Combine the aqueous extracts.

11.8.5.8. Water-soluble organic acids and phenols will be primarily in the aqueous phase. Base/neutral analytes will be in the methylene chloride. If the analytes of interest are only in the aqueous phase discard the methylene chloride and proceed to Section 11.8.5.9. If the analytes of interest are only in the methylene chloride, discard the aqueous phase and proceed to Section 11.8.5.11.

11.8.5.9. Externally cool the flask with ice while adjusting the aqueous phase to a pH of 1-2 with sulfuric acid (1:1). Transfer the cool aqueous phase to a clean 125 mL separatory funnel.

11.8.5.10. Add 20 mL of methylene chloride to the separatory funnel and shake for at least two minutes. Allow the methylene chloride to separate from the aqueous phase and collect the

methylene chloride in an Erlenmeyer flask. Repeat the extraction two more times using fresh methylene chloride and extracting at pH 1-2. Combine the three extracts.

11.8.5.11. Dry the extract by passing through a funnel containing 10-20 g anhydrous sodium sulfate. Rinse the funnel with an additional 20-30 mL of clean methylene chloride

11.8.5.12. Cover with aluminum foil if the extract is not concentrated immediately. Refer to section 11.7 for concentration.

11.8.5.13. Dispose of solvent and water remaining in the separatory funnel into the appropriate waste container.

11.8.7. Fluorocarbon Cleanup

This procedure is appropriate for the removal hydrocarbons from water samples prior to analysis for water soluble alcohols

11.8.7.1. Transfer 1-2 mL of water sample to a 10 mL culture tube.

11.8.7.2. Add 1-2 mL of fluorocarbon solvent (PF-5080) to the culture and cap.

11.8.7.3. Shake for 30-60 seconds.

11.8.7.4. After the two phases have separated, pipette about 1 mL of water sample (top layer) into an autosampler vial for analysis. If necessary, centrifuge to enhance the phase separation.

12. DATA ANALYSIS AND CALCULATIONS

Not applicable

13. METHOD PERFORMANCE

13.1. Method detection limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The procedure for the determination of the method detection limit is given in STL North Canton QA Policy #: QA-005

13.2. Initial demonstration

Each laboratory must make an initial demonstration of capability for each individual method. This requires analysis of four QC Check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which should contain all the analytes of interest. The spiking level should be equivalent to a mid-level calibration. (For certain tests more than one set of QC check samples may be necessary in order to demonstrate capability for the full analyte list.)

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.2.2. Calculations and acceptance criteria for the QC check samples are given in the determinative SOPs. (CORP-GC-0001, CORP-MS-0001, 0002)

13.3. Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

Within the constraints of following the methodology in this SOP, use of organic solvents should be minimized.

15. WASTE MANAGEMENT

Waste generated in this procedure will be segregated, and disposed according to the facility hazardous waste procedures. The Laboratory Environmental, Health and Safety Coordinator should be contacted if additional information is required.

15.1. Solvent waste must be disposed of in the clearly labeled waste cans.

15.2. Acid waste must be collected in the clearly labeled acid waste containers.

15.3. Solid materials (soil, gloves, soiled paper products) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.4. Methylene chloride saturated water and remaining sample after the extraction is dumped into Methylene chloride waste cans.

16. REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III (December 1996). Sections 3500B, 3510C, 3520C, 3540C, 3550B, 3600C, 3610B, 3620B, 3640A, 3650B, 3660B, AND 3665A.

17. MISCELLANEOUS

17.1. Modifications from Reference method

17.1.1. Some surrogate spiking concentrations are modified from those recommended in SW-846, in order to make the concentrations more consistent with the calibration levels in the determinative methods.

17.1.2. Aqueous sample volumes may be determined by weight.

17.1.3. Spiking levels for method 608 have been reduced by a factor of ten to bring the levels within the normal calibration range of the instrument.

17.1.4. 10 g of soil is used for pH determination, rather than the 50 g suggested in the reference method. The volume of water is also adjusted to maintain the sample / water ratio specified in the method.

17.2. Modifications from previous revision

17.2.1. SOP change forms are on record in the Quality Assurance Department.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4. Tables

Table 1
Liquid /Liquid Extraction Conditions

Determinative Method	Initial Ext. pH	Secondary Ext. pH
BNA: 8270C[1]	1-2	11-12
625	11-12	1-2
Pest/PCB: 8081A, 8082 & 608	5-9	None
OPP: 8141A	as received	None
Hydrocarbons: 8015B	as received	None
PAH: 8310, & 610	as received	None

[1] If the laboratory has validated acid only 8270 extraction for the target compound list required then the base extraction step may be omitted. The required validation consists of a 4 replicate

initial demonstration of capability and a method detection limit study. (See section 13). Additionally, either of the base or acid fractions of Method 8270 can be run first.

Table 2
Exchange Solvents and Final Volumes

Final Volumes and Exchange Solvents if no cleanup is used		
Type	Exchange Solvent for Analysis	Final Volume for Analysis in mL
Semivolatiles	N/A	1.0 or 5.0* 2.0 for H2O**
PCB	Hexane	10.0 for solids 5.0 for H2O 2.0 for H2O**
Pesticides	Hexane	10.0 for solids 5.0 for H2O
PAH by HPLC	Acetonitrile	1.0 mL
TPH	N/A	1.0
OPP	Hexane/Acetone	2.0

*Requires high sensitivity mass spec tune (refer to NC-MS-0015 PAH by SIM)
** Michigan work requires a final volume of 2 mL.

Final Volumes and exchange solvents if GPC cleanup is used CLP ONLY			
Type	Exchange Solvent for GPC	Final Volume for GPC	Final Volume and solvent for Analysis
Semivolatiles Semivolatiles	CH_2Cl_2 CH_2Cl_2	10 mL[1] 10 mL[1]	0.5 mL CH_2Cl_2 -OLM03.1 2.5 mL CH_2Cl_2 -OLM04.2
Pesticides	CH_2Cl_2	10 mL[1]	5 mL, hexane

[1] Final volume for GPC may be 4 mL if a 2 mL sample loop is used

Final volumes and exchange solvents if Florisil cleanup is used CLP ONLY			
Type	Exchange Solvent for Florisil	Final Volume for Florisil	Final Volume and solvent for Analysis
Pesticides	Hexane	10 mL (2 mL aliquot used)	2 mL, hexane

Final volumes and exchange solvents if both GPC and Florisil cleanup are used CLP ONLY					
Type	Exchange Solvent for GPC	Final Volume for GPC	Exchange Solvent for Florisil	Final Volume for Florisil	Final volume for analysis
Pesticides	Methylene Chloride	10 mL	Hexane	5 mL (2 mL aliquot used)	2 mL, hexane

Note: Different final volumes may be necessary to meet special client reporting limit requirements.

Table 3		
Surrogate Spiking Solutions		
Analyte Group	Surrogate Spike Solution ID	Volume (mL)
BNA	100/150 ppm BNA	0.5
BNA-SIM	100/150 ppm BNA	0.05
BNA-Michigan H2O	100/150 ppm BNA	0.2
Pest	0.2 ppm DCB/TCX	1.0
TPH	40ng C9	1.0
PCB	0.2 ppm DCB/TCX	1.0
PAH	1.0 ug/mL p-Terphenyl-d14 5.0 ug/mL Benzo(e)pyrene	1.0
OPP	10 ug/mL Triphenyl Phosphate	1.0

Table 4		
Matrix Spike and LCS Solutions		
Analyte Group	Matrix Spike Solution ID	Volume (mL)
BNA, BNA TCLP, NPDES	100 ppm BNA All-Analyte Spike	0.5
BNA- Michigan H2O	100 ppm BNA All-Analyte Spike	0.2
BNA NPDES	100 ppm BNA All-Analyte Spike	1.0
Pest	Pest NPDES Spike	1.0
Pest TCLP	Pest TCLP Spike	1.0
Pest NPDES	Pest NPDES Spike	1.0
PCB	10 ppm PCB Spike	1.0

Table 5
Surrogate Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
BNA	2-Fluorobiphenyl	Methanol	100
	Nitrobenzene-d5		100
	p-Terphenyl-d14		100
	2-Fluorophenol		150
	Phenol-d6		150
	2,4,6-Tribromophenol		150
	1,2-Dichlorobenzene-d4		100
	2-Chlorophenol-d4		150
Pest/PCB	Decachlorobiphenyl	MethanolAcetone	0.2
	Tetrachloro-m-xylene		0.2
TPH	Nonane (C9)	Methanol	40.0
PAH	p-Terphenyl-d-14	CH3CN	1.0
	Benzo(e)pyrene		5.0
OPP	Triphenylphosphate	Acetone	10.0

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
TCL BNA	Acenaphthene	Methanol	100
	4-Chloro-3-Methylphenol		150
	2-Chlorophenol		150
	1,4-Dichlorobenzene		100
	2,4-Dinitrotoluene		100
	4-Nitrophenol		150
	N-Nitroso-Di-n-Propylamine		100
	Pentachlorophenol		150
	Phenol		150
	Pyrene		100
	1,2,4-Trichlorobenzene		100
BNA TCLP	1,4-Dichlorobenzene	Methanol	100
	2,4-Dinitrotoluene		100
	Hexachlorobenzene		100
	Hexachlorobutadiene		100
	Hexachloroethane		100
	2-Methylphenol		100
	3-Methylphenol		100
	4-Methylphenol		100
	Nitrobenzene		100
	Pentachlorophenol		100
	Pyridine		100
	2,4,5-Trichlorophenol		100
	2,4,6-Trichlorophenol		100

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
BNA NPDES		Methanol	
	Acenaphthene		100
	Acenaphthylene		100
	Anthracene		100
	Benzo(a)anthracene		100
	Benzo(b)fluoranthene	Methanol	100
	Benzo(k)fluoranthene		100
	Benzo(a)pyrene		100
	Benzo(ghi)perylene		100
	Benzyl butyl phthalate		100
	Bis(2-chloroethyl)ether		100
	Bis(2-chloroethoxy)methane		100
	Bis(2-ethylhexyl)phthalate		100
	Bis(2-chloroisopropyl)ether		100
	4-Bromophenyl phenyl ether		100
	2-Chloronaphthalene		100
	4-Chlorophenyl phenyl ether		100
	Chrysene		100
	Dibenzo(a,h)anthracene		100
	Di-n-butylphthalate		100
	1,3-Dichlorobenzene		100
	1,2-Dichlorobenzene		100
	1,4-Dichlorobenzene		100
	3,3'-Dichlorobenzidine		100

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	Diethyl phthalate		100
	Dimethyl phthalate		100
	2,4-Dinitrotoluene		100
	2,6-Dinitrotoluene		100
	Di-n-octylphthalate		100
	Fluoranthene		100
	Fluorene		100
	Hexachlorobenzene		100
	Hexachlorobutadiene		100
	Hexachloroethane		100
	Indeno(1,2,3-cd)pyrene		100
	Isophorone		100
	Naphthalene		100
	Nitrobenzene		100
	N-Nitrosodi-n-propylamine		100
	Phenanthrene		100
	Pyrene		100
	1,2,4-Trichlorobenzene		100
	4-Chloro-3-methylphenol		100
	2-Chlorophenol		100
	2,4-Dichlorophenol		100
	2,4-Dimethylphenol		100
	2,4-Dinitrophenol		100
	2-Methyl-4,6-dinitrophenol		100

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	2-Nitrophenol		100
	4-Nitrophenol		100
	Pentachlorophenol		100
	Phenol		100
	2,4,6-Trichlorophenol		100
Pest TCLP	Heptachlor	MethanolAcetone	0.5
	Heptachlor epoxide		0.5
	Lindane		0.5
	Endrin		0.5
	Methoxychlor		1.0
Pest NPDES /Pest	Aldrin	MethanolAcetone	1.0
	alpha-BHC		1.0
	beta-BHC		1.0
	delta-BHC		1.0
	gamma-BHC (Lindane)		1.0
	4,4'-DDD		1.0
	4,4'-DDE		1.0
	4,4'-DDT		1.0
	Dieldrin		1.0
	alpha-Endosulfan		1.0
	beta-Endosulfan		1.0

Table 6			
Matrix Spike Components			
Type	Compounds	Solvent	Conc. (µg/mL)
	Endosulfan Sulfate		1.0
	Endrin		1.0
	Heptachlor		1.0
	Heptachlor Epoxide		1.0

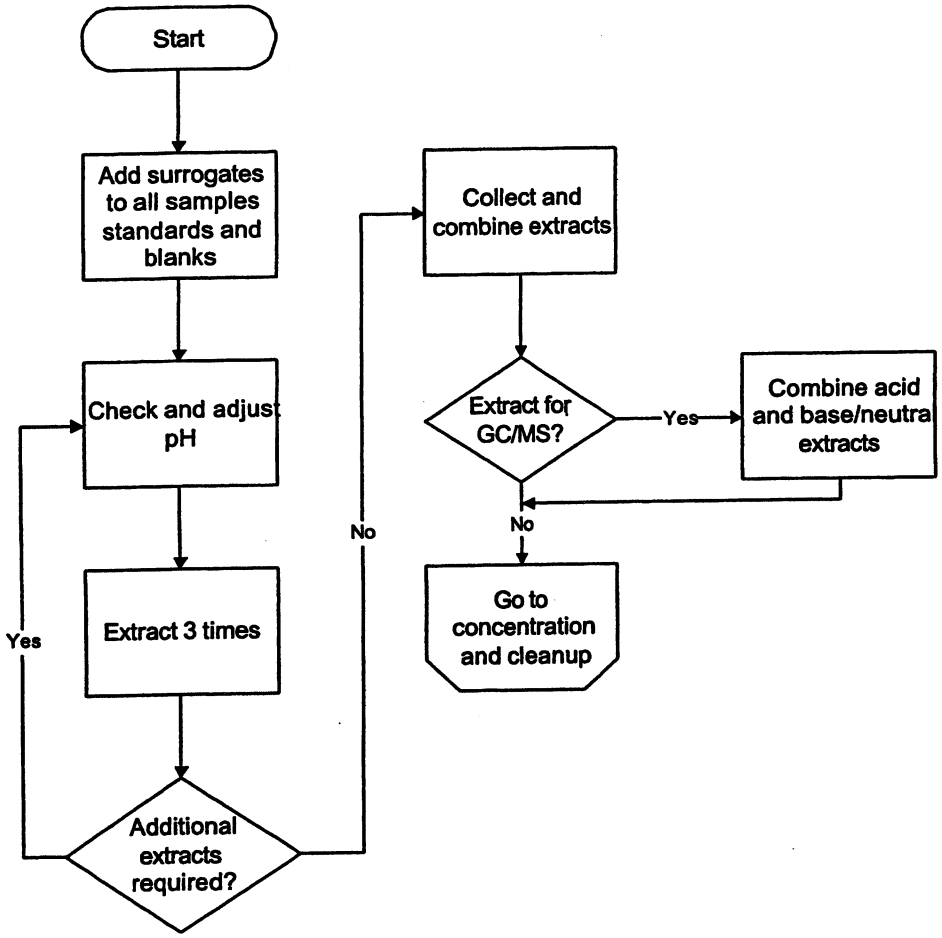
Diesel Range Organics (8015B) Spike	
Compound	**Final Concentration**
n-decane	50 µg/ml
n-dodecane	50 µg/ml
n-tetradecane	50 µg/ml
n-hexadecane	50 µg/ml
n-octadecane	50 µg/ml
n-eicosane	50 µg/ml
n-docosane	50 µg/ml
n-tetracosane	50 µg/ml
n-hexacosane	50 µg/ml
n-octacosane	50 µg/ml

Organophorphorous Pesticides (8141A)	
Compound	**Final Concentration**
dimethoate	20 µg/mL
disulfoton	20 µg/mL
famphur	20 µg/mL
methyl parathion	20 µg/mL
parathion (ethyl)	20 µg/mL
phorate	20 µg/mL
sulfotepp	20 µg/mL
thionazin	20 µg/mL
o,o,o-triethyl phosphorothioate	20 µg/mL
triphenylphosphate (surrogate)	20 µg/mL

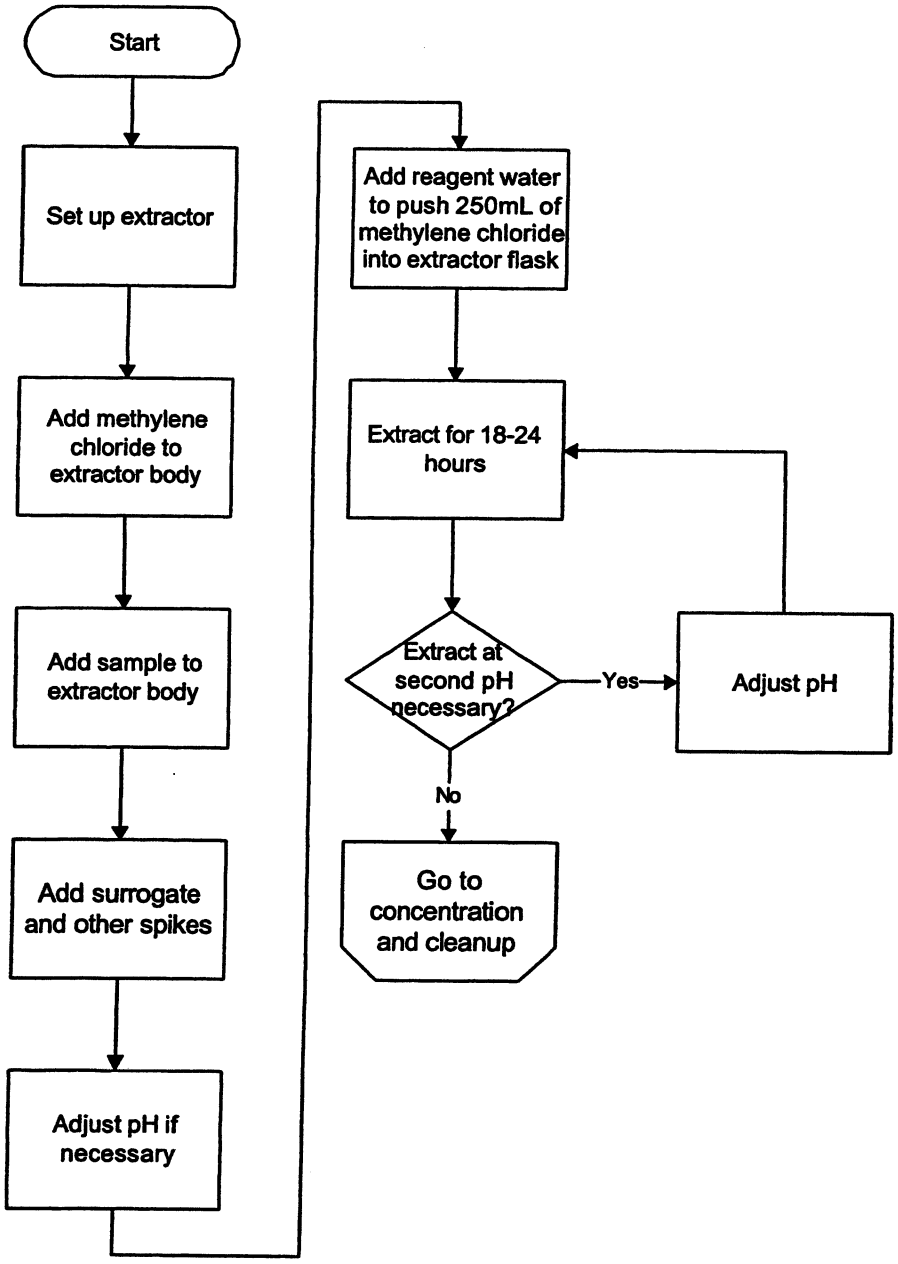
Polynuclear Aromatic Hydrocarbons (8310)	
Compound	**Final Concentration**
Acenaphthylene	10 µg/mL
Carbazole	10 µg/mL
Naphthalene	10 µg/mL
1-Methylnaphthalene	10 µg/mL
2-Methylnaphthalene	10 µg/mL
Acenaphthene	10 µg/mL
Fluorene	2 µg/mL
Phenanthrene	2 µg/mL
Anthracene	2 µg/mL
Fluoranthene	2 µg/mL
Pyrene	2 µg/mL
Benzo(a)anthracene	2 µg/mL
Chrysene	2 µg/mL
Benzo(a)pyrene	2 µg/mL
Benzo(k)fluoranthene	2 µg/mL
Benzo(a)pyrenek)fluoranthene	2 µg/mL
Dibenzo(a,h)anthracene	2 µg/mL
Benzo(g,h,I)perylene	2 µg/mL
Indeno(1,2,3-cd)pyrene	2 µg/mL

17.5. Flow diagrams

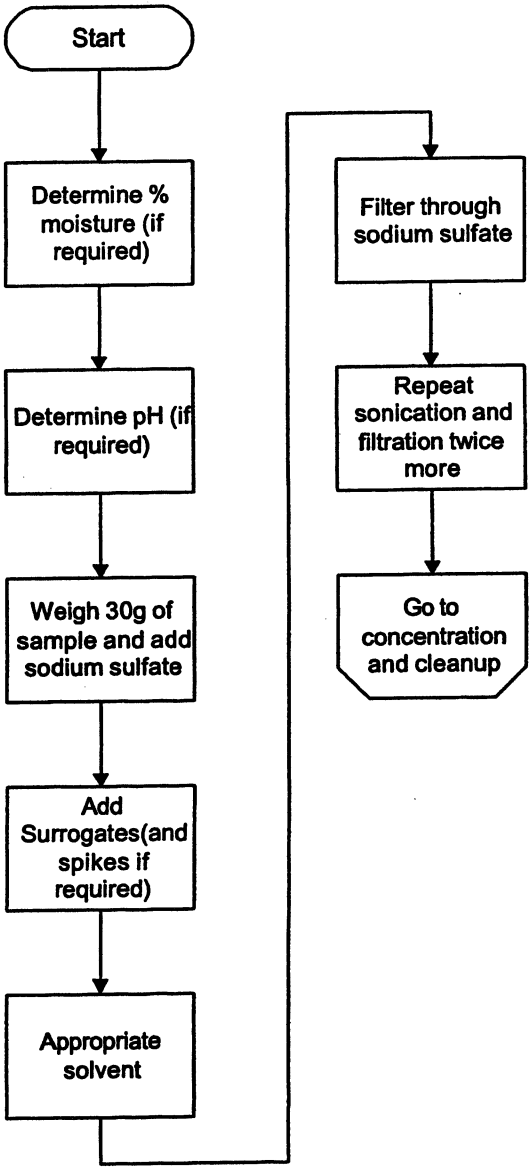
17.5.1. Separatory funnel extraction



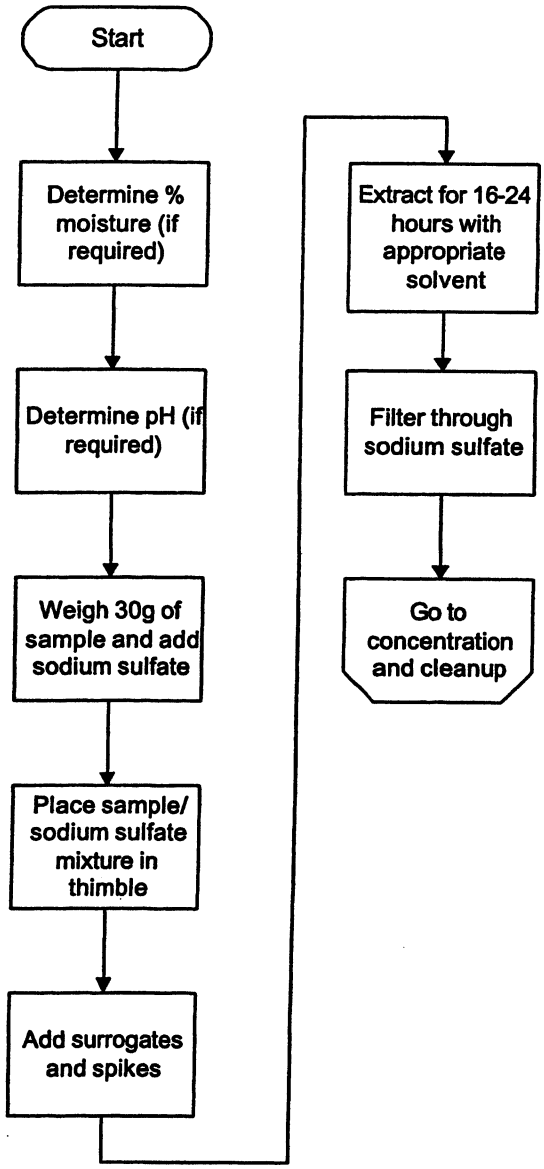
17.5.2. Continuous liquid/liquid extraction


Start
Set up extractor
Add methylene chloride to extractor body
Add sample to extractor body
Add surrogate and other spikes
Adjust pH if necessary
Add reagent water to push 250mL of methylene chloride into extractor flask
Extract for 18-24 hours
Extract at second pH necessary?
Yes
Adjust pH
No
Go to concentration and cleanup

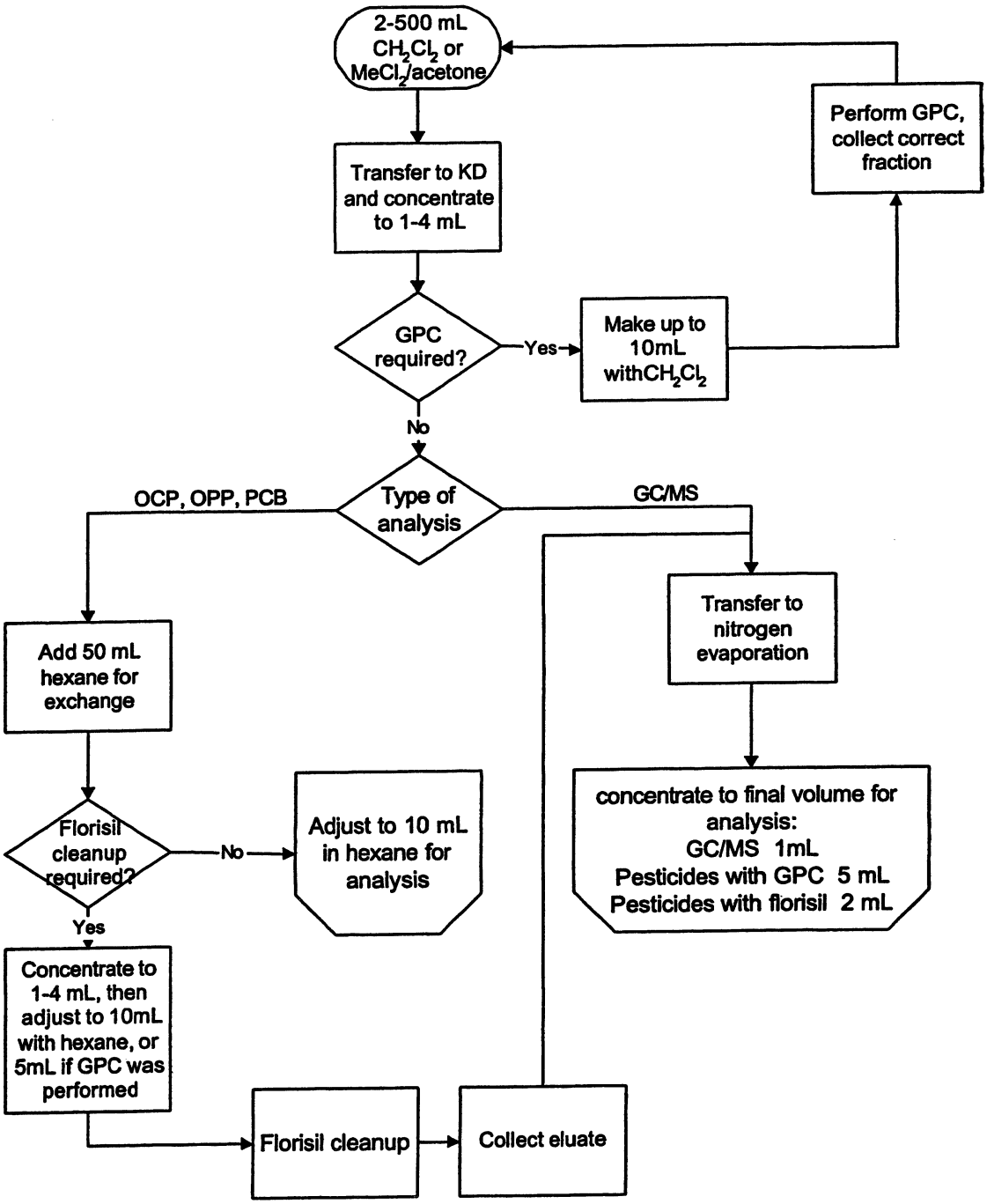
17.5.3. Sonication Extraction



17.5.4. Soxhlet extraction



17.5.5. Concentration and cleanup



18. SCOPE AND APPLICATION

This method is applicable to the extraction of chlorinated herbicides in waters, solids, oils, and TCLP extracts. Appropriate compounds for extraction by this method are listed in CORP-GC-0001, Appendix D, Gas Chromatography of Phenoxy Acid Herbicides based on Method 8151A.

19. SUMMARY OF METHOD

This method is based on SW846 method 8151A. Aqueous samples are hydrolyzed if esters and acids are to be determined, then washed with methylene chloride by a separatory funnel extraction. After acidifying the sample the free acids are extracted into diethyl ether. Solids are extracted into methylene chloride/ acetone by sonication. If esters and acids are to be determined, the extract is hydrolyzed and extracted into diethyl ether. For both soils and aqueous samples, the free acid herbicides in the ether extract are esterified. The final volume is adjusted to prepare the extract for gas chromatography.

20. DEFINITIONS

Refer to section 3 of the main body of this SOP.

21. INTERFERENCES

Refer to section 4 of the main body of this SOP.

22. SAFETY

22.1. Refer to section 5 of the main body this SOP for basic safety information.

22.2. DIAZOMETHANE is an extremely toxic gas with an explosion potential. Since the explosion potential is catalyzed by imperfections in glass, generation of diazomethane must be carried out in glassware free of scratches, cracks, chips and which does not have ground glass joints. Solutions of diazomethane will be kept at temperatures below 90°C. Diazomethane must be generated and handled in a fume hood.

22.3. Diethyl ether is extremely flammable. It also tends to form peroxides when exposed to air. The peroxides can present an explosion hazard, especially when the ether is concentrated.

22.4. Diethyl ether must be free of peroxides as demonstrated by EM (or equivalent) Quant test strips. This test can be done every time the ether is used or once per week if the bottle is marked with the test date(s).

22.5. Concentrated potassium hydroxide solution is highly caustic.

23. EQUIPMENT AND SUPPLIES

23.1. Refer to Section 6 of the main body of this SOP for basic extraction equipment and supplies. Additional equipment and supplies needed for this procedure are listed below.

23.2. Diazomethane generation apparatus

23.3. EM Peroxide test strips

24. REAGENTS AND STANDARDS

24.1. Reagents are listed in Section 7 of the main body of this SOP. Additional Reagents and standards needed for this procedure are listed below.

24.2. Reagents

24.2.1. Potassium hydroxide solution, 37% aqueous solution, (w/v): Dissolve 37 g of potassium hydroxide pellets in reagent water and dilute to 100 mL. **Caution:** Considerable heat will be generated. Other volumes of solution may be made up as convenient.

24.2.2. Sodium hydroxide solution, 10N. Dissolve 400 g NaOH in reagent water and dilute to 1.0L. **Caution:** Considerable heat will be generated. Other volumes of solution may be made up as convenient.

24.2.4. Sulfuric acid, 1:1 Slowly add 500 mL concentrated sulfuric acid to 500 mL water. **Caution:** Considerable heat will be generated. The acid must be added to the water. Wear protective clothing and safety glasses. Other volumes of solution may be made up as convenient.

24.2.5. Diazald, 99% purity

24.2.7. Sodium sulfate, Na_2SO_4, Anhydrous, granular, acidified: Heat sodium sulfate in a shallow tray at 400°C for a minimum of 4 hours to remove phthalates and other interfering organic substances. In a large beaker, acidify by slurrying 500 g sodium sulfate with just enough diethyl ether to cover. Add 20 mL of concentrated sulfuric acid and mix thoroughly. Place the mixture on a steam bath in a hood to evaporate the ether, or allow the ether to evaporate overnight. Larger or smaller batches of acidified sodium sulfate may be prepared using the reagents in the same proportions.

24.2.8. Sodium Chloride, NaCl

24.2.9. Acidified 5% sodium sulfate solution

Add 50 g of sodium sulfate to one liter of reagent water. Add 10 mL of concentrated H_2SO_4. (This reagent may be prepared in different quantities if the proportions are kept the same).

24.2.10. Diethyl ether, reagent grade.

24.2.11. Methanol, reagent grade.

24.2.12. Silicic gel

24.2.13. 2% methanolic KOH,semi-conductor grade

24.3. Standards

24.3.1. Surrogate Standard
See Table A3.

24.3.2. Matrix Spike and LCS standard
See Table A4.

25. SAMPLE COLLECTION PRESERVATION AND STORAGE

25.1. Sample collection and storage is described in Section 8 of the main body of this SOP.

26. QUALITY CONTROL

26.1. Refer to Section 9 of the main body of this SOP for Quality control procedures.

27. CALIBRATION AND STANDARDIZATION

Not applicable

28. PROCEDURE

28.1. Preparation of Aqueous Samples

28.1.1. Weigh the sample bottle and pour approximately 500 ml(100 mL for TCLP leachates) into a 2 liter separatory funnel. Reweigh the bottle and record the sample volume on the benchsheet, assuming a density of 1.0. Alternatively, measure 500 ml in a graduated cylinder. If less than 1 liter was used, reagent water may be added to make the volume up to 1 liter.

Note: Aqueous samples must be determined volumetrically for Ohio VAP samples.

28.1.2. Spike each sample blank, LCS and MS with 1.0 mL of DCAA surrogate solution. Spike matrix spikes and LCS with 1 mL of herbicide matrix spiking solution. (Refer to tables A1 and A2)

28.1.3. Add 60-80 g of NaCl to the sample and shake to dissolve the salt.

28.1.4. Hydrolysis

Use this step only if herbicide esters in addition to herbicide acids are to be determined. This is normally the case. If the herbicide esters are not to be determined, omit this step and go to 11.1.10.
Add 3 mL of 10N NaOH to the sample, seal and shake. Check the pH of the sample with pH paper. If the pH of the sample is not $\geq$12 adjust to $\geq$12 by adding more NaOH. Let the sample sit at room temperature for 2 hours to complete the hydrolysis.

28.1.6. If the whole contents of the sample bottle were not used, add 60 mL of methylene chloride to the separatory funnel.

28.1.7. Extract the sample by shaking or rotating vigorously for 2 minutes, venting as necessary. (An automatic shaker may be used. Place the samples in tumbler and allow them to tumble for one (1) hour. Allow the organic layer to separate from the aqueous layer. If an emulsion layer greater than one third of the solvent layer forms, use mechanical techniques to complete the phase separation. Suggested techniques are stirring, filtration through glass wool and centrifugation.

28.1.8. Discard the **methylene chloride** phase.

28.1.9. Add a second 60 mL of methylene chloride and repeat the extraction a second time, discarding the methylene chloride. Repeat the extraction a third time.

28.1.10. Add 6 mL of cold (4°C) 1:1 sulfuric acid to the sample. Seal, and shake to mix. Check the pH of the sample with pH paper. If the pH is not $\leq$ 2, and more acid to adjust the pH to $\leq$ 2.

Caution: Addition of acid may cause heat and / or pressure build up.

28.1.11. Add 100 mL diethyl ether to the sample and extract by shaking or rotating vigorously for 2 minutes, venting as necessary. (An automatic shaker may be used. Place samples in tumbler and tumble for twenty (20) minutes for each extraction). Allow the organic layer to separate from the aqueous layer. If a emulsion layer greater than one third of the solvent layer forms, use mechanical

techniques to complete the phase separation. Suggested techniques are stirring, filtration through glass wool and centrifugation.

28.1.12. Drain the aqueous layer into a clean flask or beaker. Collect the ether phase in a clean flask or bottle containing approximately 10g of acidified anhydrous sodium sulfate.

28.1.13. Return the aqueous phase to the separatory funnel, add 100 mL diethyl ether and repeat the extraction procedure a second time., combining the ether extracts. Repeat the extraction a third time with 100 mL diethyl ether. Discard the aqueous phase after the third extraction.

28.1.14. Allow the extract to remain in contact with the sodium sulfate for at least 2 hours, shaking periodically. (May be left overnight). The drying step is critical: if the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate. The amount of sodium sulfate is sufficient if some free flowing crystals are visible when the flask or bottle is swirled or shaken.

28.1.15. Proceed to section 11.5, concentration.

28.2. Extraction of soil and sediment samples

28.2.1. Decant and discard any water layer on a sediment/soil sample. Record and document if a water layer was discarded on the benchsheet. Homogenize the sample by mixing it thoroughly in the container. If this is not possible place the sample in clean beaker and homogenize. Upon completion of homogenization in beaker return sample to original container. Discard foreign objects such as sticks, leaves and rocks, unless extraction of this material is required by client. If the sample consists primarily of foreign materials consult with the client (via the Project Manager or Administrator). Decant and discard any water layer on a sediment/soil sample.

28.2.2. Weigh 50.0 g of moist solid sample into an clean glass jar. Use 50 g of sodium sulfate for the Method Blank and 50g Ottawa sand for the LCS. Acidify the sample with 5 mL of concentrated HCl.

28.2.3. There should be a small amount of liquid phase. If not, add reagent water until there is. Stir well with a spatula. (Note: This is not necessary for the method blank or LCS)

28.2.4. After 15 minutes, stir with a spatula and check the pH of the liquid phase. Add more acid if necessary to bring the pH to <2, repeating the stirring and standing time after each acid addition. (Note: The pH of the method blank and LCS are not determined.)

28.2.5. Add 60 g of acidified sodium sulfate and mix well. The sample should be free flowing. If not, add more sodium sulfate.

28.2.6. Spike each sample blank, LCS and MS with 1.0 mL of DCAA surrogate solution. Spike matrix spikes and LCS with 1 mL of herbicide matrix spiking solution. (Refer to tables A1 and A2)

28.2.7. Add a minimum of 100 mL of 1:1 methylene chloride:acetone to the beaker or 100 mL of methylene chloride for long list (dinoseb).

28.2.8. Place the bottom surface of the appropriate disrupter horn tip approximately ½ inch below the surface of the solvent, but above the sediment layer.

28.2.9. Sonicate for 3 minutes, making sure the entire sample is agitated. If the W-380 or W-385 sonicator is used the output should be set at 6 for the 3/4 inch high gain (Q) horn or 10 for the 3/4 inch standard horn with mode switch on pulse, and percent-duty cycle knob set at 50%.

28.2.10. Loosely plug the stem of a 75 mm x 75 mm glass funnel with glass wool and/or line the funnel with filter paper. Add 10-20 g of anhydrous sodium sulfate to the funnel cup.

28.2.11. Place the prepared funnel on a collection apparatus. If the herbicide esters are *not* to be determined, the collection apparatus is a bottle or flask containing approximately 10g of anhydrous acidified sodium sulfate. If the herbicide esters *are* to be determined, (normally the case) the collection apparatus is glassware suitable for the hydrolysis step, typically a KD flask or Turbovap tube.

28.2.12. Decant and filter extracts through the prepared funnel into the collection apparatus.

28.2.13. Repeat the extraction two more times with additional 100 mL minimum portions of the appropriate solvent each time. Decant off extraction solvent after each sonication. On the final sonication pour the entire sample (sediment and solvent) into the funnel and rinse with an additional 10 mL-20 mL of the methylene chloride/acetone.

Note: Alternatively, the three extracts may be collected together and then filtered through the sodium sulfate.

28.2.14. If the herbicide esters are not to be determined, dry the extract as described in 11.4.2 or go to cleanup, section 11.3. If the herbicide esters are to be determined (normally the case) proceed to section 11.2.15

28.2.15. Add 5 mL of 37% aqueous potassium hydroxide and 30 mL of water to the extract. Check the pH with pH paper. If the pH is not $\geq$12, adjust with additional KOH.

28.2.16. Heat on a water bath at 60-65°C for 2 hours. Allow to cool. Higher temperatures, up to 90°C, may be used if needed to remove the ether layer within 2 hours.

28.2.17. Transfer the solution to a separatory funnel and extract three times with 100 mL portions of methylene chloride. **Discard the methylene chloride phase.** The aqueous solution contains the herbicides.

28.2.18. Adjust the pH of the solution to $\leq$2 with 1:1 sulfuric acid.

28.2.19. Extract three times with 40 mL diethyl ether.

28.2.20. Proceed to section 11.3, Cleanup, if required, or Section 11.4, Extract drying.

28.3. Cleanup

This cleanup step may be necessary if the procedure for determining the herbicide acids only is being followed. (See section 11.2.14) It is not normally required if the acids and esters are being determined. (The usual case.) If cleanup is not required, proceed to section 11.4, Extract drying.

28.3.1. Prepare 45 mL of basic extraction fluid by mixing 30 mL of reagent water with 15 mL of 37% KOH. Use three 15 mL portions of this fluid to partition the extract from section 11.2.12 or 11.2.20, using a small separatory funnel. **Discard the organic phase.**

28.3.2. Adjust the pH of the solution to $\leq$2 with cold (4°C) sulfuric acid. (1:1). Extract once with 40 mL diethyl ether and twice with 20 mL diethyl ether.

Caution: Addition of acid may cause heat and / or pressure build up.

28.4. Extract drying

28.4.1. Pour the extracts through a funnel containing acidified sodium sulfate into a flask or bottle containing approximately 10 g acidified sodium sulfate. Rinse the funnel with a little extra diethyl ether.

28.4.2. Allow the extract to remain in contact with the sodium sulfate for at least 2 hours, shaking periodically. (May be left overnight). The drying step is critical: if the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate. The amount of sodium sulfate is sufficient if some free flowing

crystals are visible when the flask or bottle is swirled or shaken. Proceed to section 11.5, concentration.

28.5. Concentration

28.5.1. Transfer the ether extract into a Turbovap concentrator tube or a 500 mL K-D flask equipped with a 10 mL concentrator tube. Use a stirring rod to crush the caked sodium sulfate during transfer. Rinse the flask or bottle with 20-30 mL ether to complete transfer.

28.5.2. Attach a three ball Snyder column to the K-D apparatus, prewet the column with a few mL of ether from the top, and place the apparatus on a water bath at approximately 60°C. At the proper rate of distillation, the balls of the column will chatter, but the chambers will not flood. When the apparent volume reaches 2 mL, remove from the water bath and allow to completely cool.

28.5.3. Carefully disassemble the concentrator tube and rinse the lower glass joint with a small amount of diethyl ether.

28.5.5. The extract is now ready for esterification by either the diazomethane solution method (11.6) or the bubbler method (11.7)

28.7. Esterification (Bubbler Method)

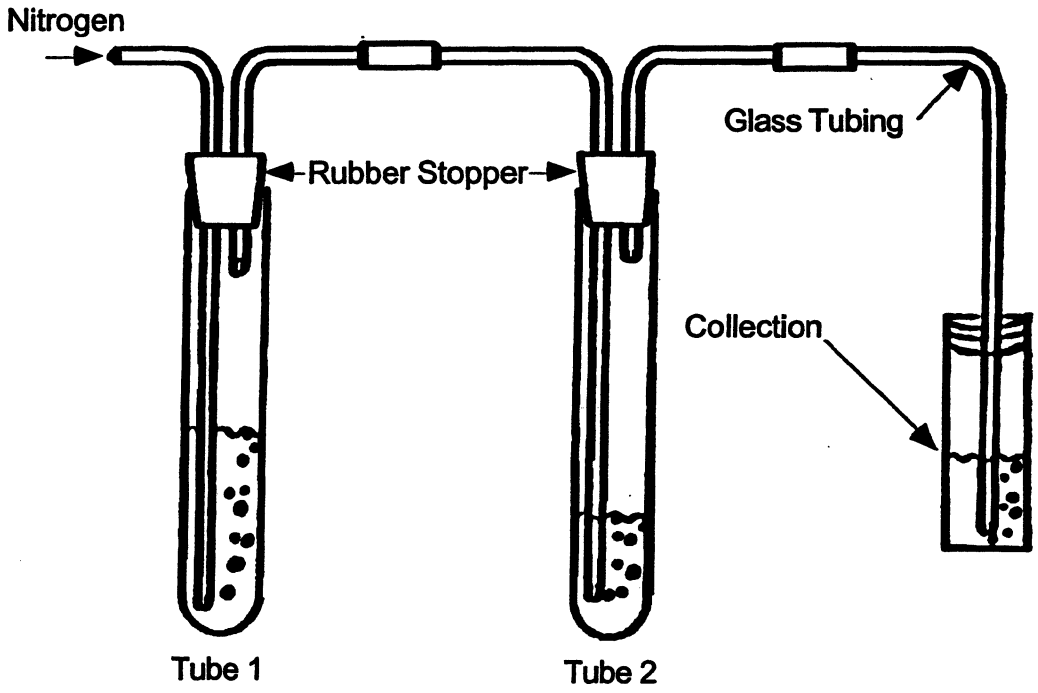
28.7.1. Assemble the diazomethane apparatus (see figure below) in a hood. Add 10 mL of diethyl ether to tube 1. Add 5 mL of 2% methanolic KOH, 3 mL of ether and 0.5-1 g of diazald to tube 2.

28.7.2. Place the tip of the disposable pipette into the vial containing the first sample extract. Apply nitrogen flow (approx. 10 mL/min) to bubble diazomethane through the sample extract for about 1 minute, or until the yellow color persists. Replace the disposable pipette and place the tip into the vial containing the second extract. Continue until the diazald is consumed. (An additional 0.1-0.5 g diazald may be added to extend the generation of diazomethane).

28.7.3. Allow the extracts to stand for 20 minutes, then add approximately 0.2 g of silicic gel to each extract. Allow to stand for an additional 20 minutes.

28.7.4. Adjust the volume to 10 mL with hexane. The sample is now ready for gas chromatography.

28.7.5 A routine 10X dilution occurs on final extracts for all samples. Due to a QuantIMS limitation, the dilution factor field in QuantIMS cannot be used when a dilution is routine, because the dilution factor is automatically applied to all reference values creating reporting problems. For the herbicide analysis, the extract volume will be 10mL and an aliquot at 10X dilution will be analyzed. The final extract volume recorded on the laboratory bench sheet will be recorded as 100mL to avoid using the dilution factor field in QuantIMS.



29. DATA ANALYSIS AND CALCULATIONS

Not applicable

30. METHOD PERFORMANCE

Refer to CORP-GC-0001 for details of method performance.

31. POLLUTION PREVENTION

Refer to section 14 of the main body of this SOP.

32. WASTE MANAGEMENT

Refer to section 15 of the main body of this SOP.

33. REFERENCES

33.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Chlorinated Herbicides, Method 8151A.

34. MISCELLANEOUS

34.1. Modifications from Reference Method

34.1.1. Directions to add sufficient reagent water to the soil sample so that the pH can be measured have been added (Section 11.1.2)

34.1.2. The bubbler esterification method uses methanolic KOH in place of the aqueous KOH / carbitol mixture recommended in method 8150B. This has been found to provide a more effective and reliable esterification.

34.2. Modifications from previous revisions

References have been updated

34.3. Tables

Table A1

Herbicide Surrogate Spiking Solutions

Analyte Group	Surrogate Spike Solution ID	Volume (mL)
Herbicides	Herbicides Water	1.0
Herbicides	Herbicides Soil	1.0

Table A2

Herbicide Matrix Spike and LCS Solutions

Analyte Group	Matrix Spike Solution ID	Volume (mL)
Herbicides	Herbicides MS-Soil	1.0
Herbicides	Herbicides MS-Water	1.0

Table A3
Herbicide Surrogate Spike Components

Type	Compounds[1]	Solvent	Conc. (ug/mL)
Herbicides WS	2,4-DCAA	Acetone	2
Herbicides SS	2,4-DCAA	Acetone Methanol	20

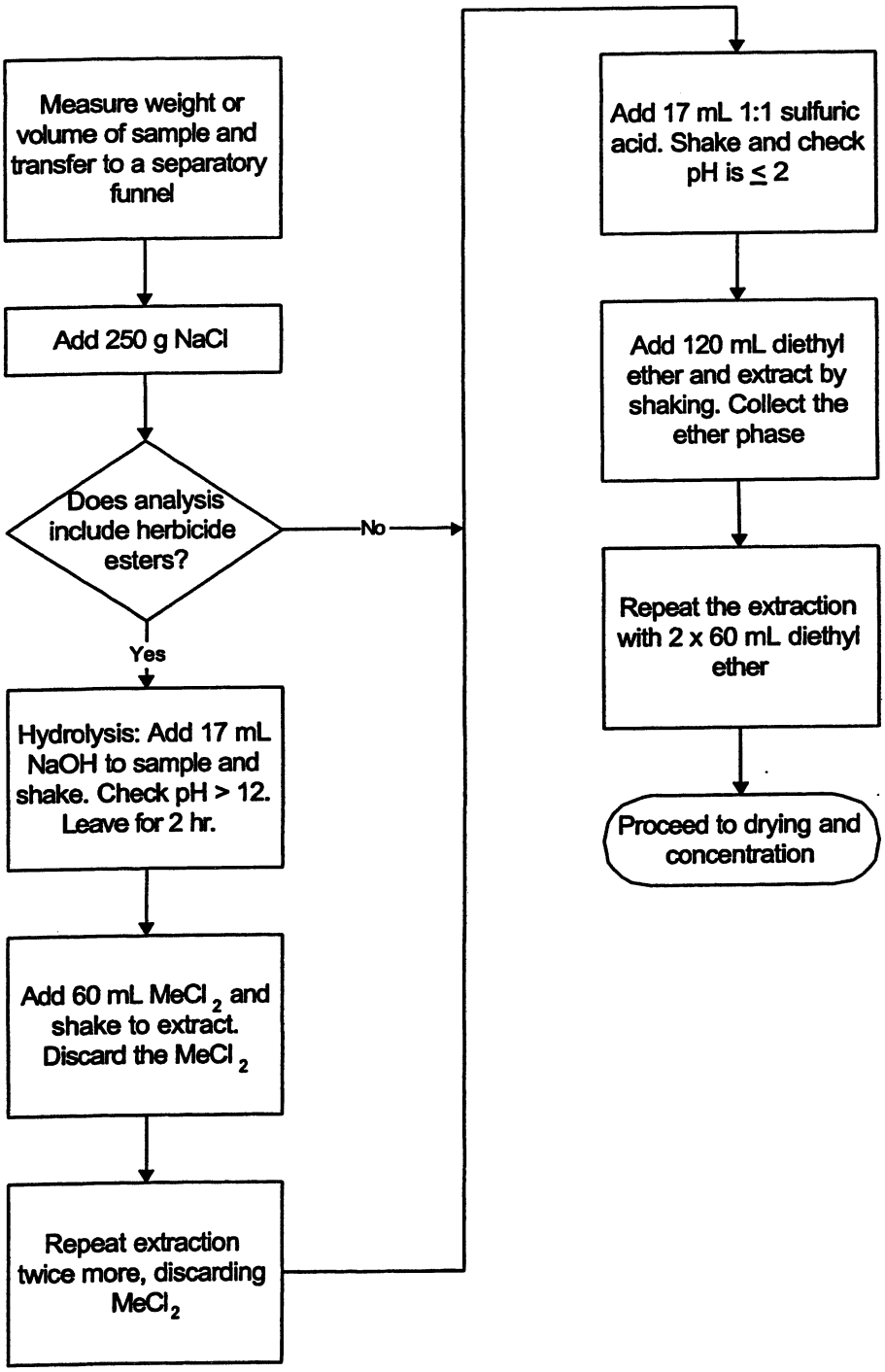
[1]The surrogate is spiked as the free acid

Table A4
Herbicide Matrix Spike Components

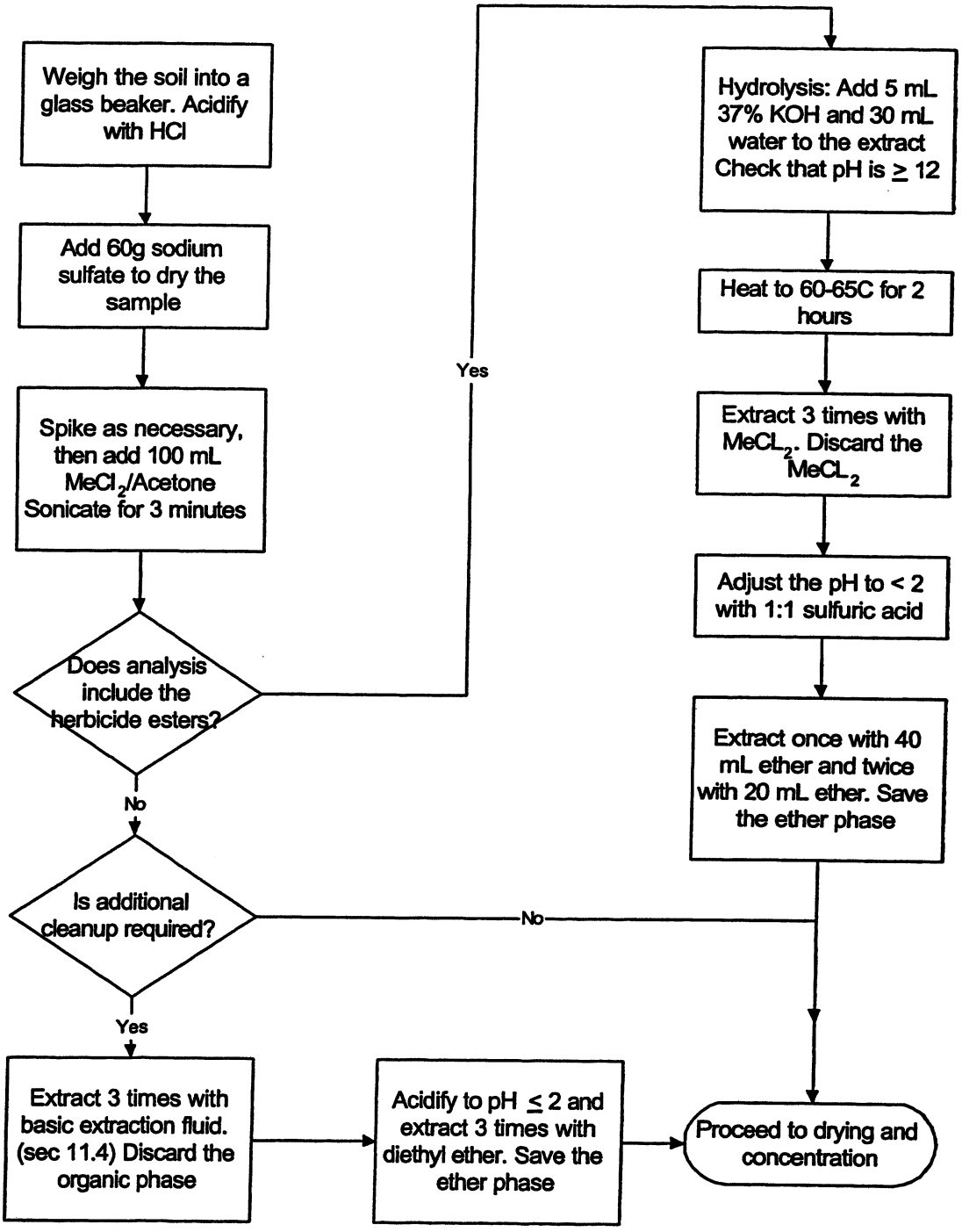
Type	Compounds[1]	Compounds[1] Solvent	Soil Conc. (ug/mL) Solvent	Water Conc. (ug/mL)
Herbicides MS	2,4-D	Acetone	20 Methanol	2
	2,4-DB		20	2
	2,4,5-TP (Silvex)		5	0.5
	Dalapon		10	1
	Dicamba		10	1
	Dichloroprop		10	1
	Dinoseb		3	.3
	2,4,5-T		5	0.5

[1]The herbicide spiking solution contains the herbicides as the free acids.

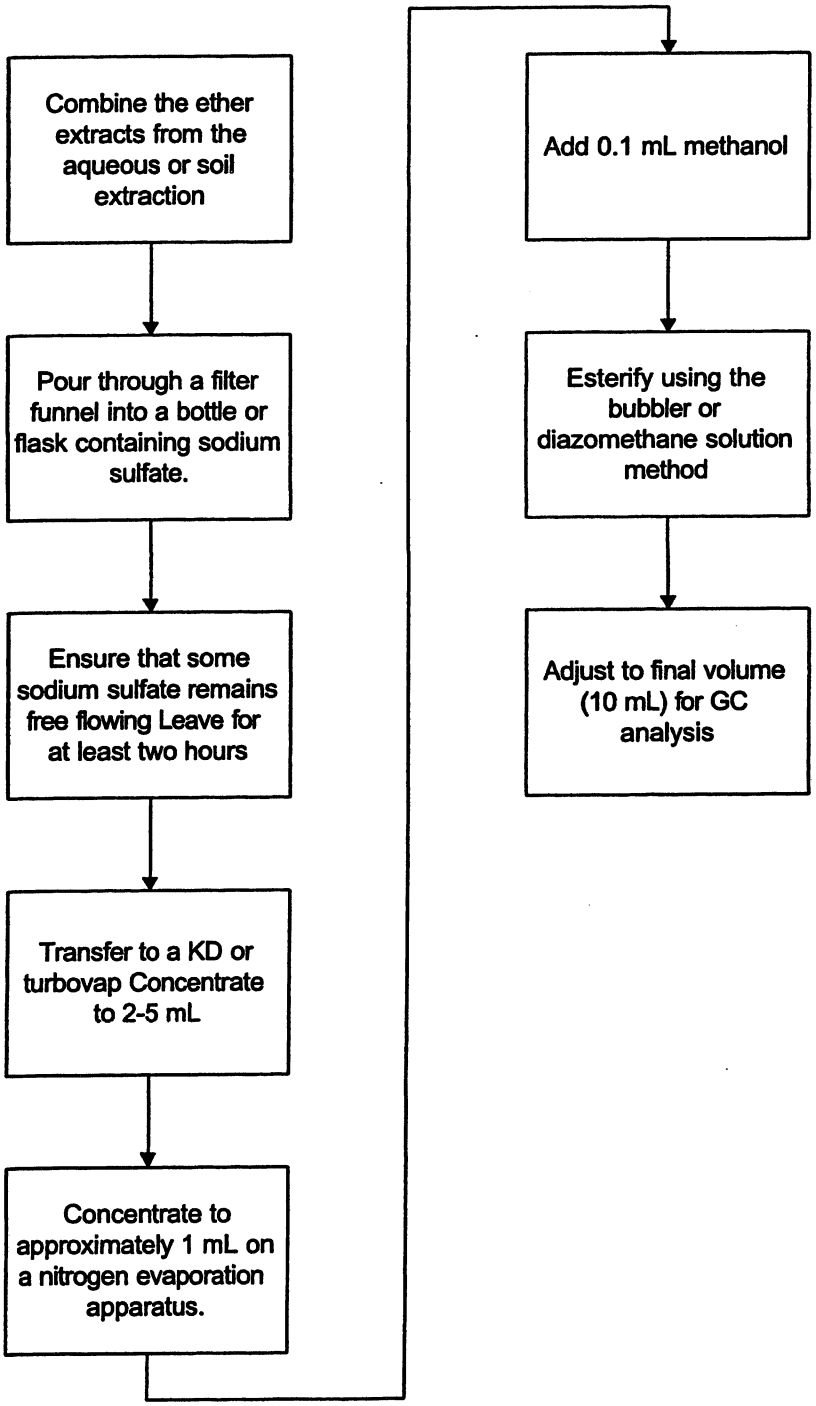
Extraction of Aqueous Samples



Extraction of Soils and Sediments



Drying, Concentration and Esterification



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SOP No: CORP-MS-0001NC
Revision No: 2.4
Revision Date: 05/29/01
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STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: GC/MS ANALYSIS BASED ON METHODS 8270C AND 625

(SUPERSEDES: Revision 2.3, 02/15/01)

Prepared by: _____ 5/30/01
Date

Reviewed by: Mark Ulman 5-30-01
Technology Specialist Date

Approved by: _____ 5/29/01
Quality Assurance Manager Date

Approved by: _____ 5-30-01
Environmental Health and Safety Date

Approved by: _____ 5-30-01
Laboratory Director Date

Approved by: _____
Coroporate Technology Date

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STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: GC/MS ANALYSIS BASED ON METHODS 8270C AND 625

(SUPERSEDES: Revision 2.3, 02/15/01)

Prepared by: _____
Date

Reviewed by: _____
Technology Specialist — Date

Approved by: _____
Quality Assurance Manager — Date

Approved by: _____
Environmental Health and Safety — Date

Approved by: _____
Laboratory Director — Date

Approved by: _____ 6/4/01
Coroporate Technology — Date

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1. SCOPE AND APPLICATION

1.1 This method is based upon SW846 8270C, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices. The modifications presented in Attachment A may be followed for analysis of wastewater following method 625. Direct injection of a sample may be used in limited applications. Refer to Tables 1, 2, 3 and 4 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. Additional compounds may be amenable to this method. If non-standard analytes are required, they must be validated by the procedures described in section 13 before sample analysis.

1.2 The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- Hexachlorophene is not amenable to analysis by this method.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method.

1.3 The standard reporting limit (SRL) of this method for determining an individual compound is approximately 0.33 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to Tables 1 and 2 for specific SRLs. Reporting limits will be proportionately higher for sample extracts that require dilution.

1.4 The associated LIMS codes are QL (8270C) and DP (625).

2 SUMMARY OF METHOD

2.1 Aqueous samples are extracted with methylene chloride using a separatory funnel, a continuous extractor or Accelerated One-Step™. Solid samples are extracted with methylene chloride / acetone using sonication, soxhlet, accelerated soxhlet or pressurized fluid extraction. Waste dilution is used for samples that are miscible with the solvent. The extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS. Extraction procedures are detailed in SOP# CORP-OP-0001. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

3 DEFINITIONS

3.1 CCC (Calibration Check Compounds) - A subset of target compounds used to evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCC's is specified for calibration acceptance.

3.2 SPCC (System Performance Check Compounds) - Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance.

3.3 Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the STL North Canton QC Program document (QA-003) for further details of the batch definition.

3.4 Method Blank - An analytical control consisting of all reagents, internal standards and surrogate standards, that is carried through the entire analytical procedure. The method

blank is used to define the level of laboratory background and reagent contamination.

3.5 LCS (Laboratory Control Sample) - A blank spiked with the parameters of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked materials demonstrates that the laboratory techniques for this method are acceptable.

3.6 MS (Matrix Spike)- aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

3.7 MSD (Matrix Spike Duplicate)- a second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method.

4 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.2 The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.3 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample.

4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.5 Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5 SAFETY PRECAUTIONS

5.1 Procedures shall be carried out in a manner that protects the health and safety of all STL

North Canton associates. The following requirements must be met:

5.1.1 Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.1.2 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

5.1.3 Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include: Benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, and n-nitrosodimethylamine. Primary standards should be purchased in solution. If neat materials must be obtained, they shall be handled in a hood.

5.1.4 Exposure to chemicals must be maintained as low as reasonably achievable; therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers should be kept closed unless transfers are being made.

5.1.5 All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported immediately to a laboratory supervisor.

6 EQUIPMENT AND SUPPLIES

6.1 Gas Chromatograph/Mass Spectrometer System: An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

6.2 Column: 30 m x 0.32 mm I.D. (or 0.25 mm I.D.) 0.5-µm film thickness silicon-coated fused-silica capillary column (J & W Scientific DB-5.625 or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.3 Mass Spectrometer: Capable of scanning from 35 to 500 AMU every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 6 when 50 ng of the GC/MS

tuning standard is injected through the GC.

6.4 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.5 Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.

6.6 Syringe: 10 µL Hamilton Laboratory grade syringes or equivalent.

6.7 Carrier gas: Ultra high purity helium.

7 REAGENTS AND STANDARDS

7.1 A minimum five point calibration curve is prepared. The low point should be at or below the reporting limit. Refer to Tables 12 and 13 for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration.

7.2 An Internal Standard solution is prepared. Compounds in the I.S. Mix are: acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.

7.2.1 Internal Standards are added to all standards and extracts to result in 8 ng or 40ng injected onto the column. For example, if the volume of an extract used was 200 µL, 20 µL of a 400 µg/mL internal standard solution would be added for a 1 µL injection.

7.3 Surrogate Standard Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. Surrogate compounds and levels are listed in Table 11.

7.4 GC/MS Tuning Standard: A methylene chloride solution containing 10 µg/mL or 50 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT, should also be included in the Tuning Standard at 10 µg/mL or 50 µg/mL.

7.5 Laboratory Control Spiking Solution: Prepare as indicated in the preparative methods. See appropriate preparation SOP. LCS compounds and levels are listed in Tables 9 and 10.

7.6 Matrix Spike Solution: Prepare as indicated in the preparative methods. See preparation SOP. The matrix spike compounds and levels are the same as the LCS compounds.

7.7 The standards listed in 7.1 to 7.6 should be refrigerated at $\leq$ 6°C when not in use. Refrigeration at -10°C to -20°C may be used if it can be demonstrated that analytes do not fall out of solution at this temperature. The standards must be replaced at least once a year. The continuing calibration standard must be replaced every week and is stored at $\leq$ 6°C.

8 SAMPLE PRESERVATION AND STORAGE

8.1 Reference appropriate facility SOP for sample bottle preservation and storage.

8.2 Samples are stored at 4 $\pm$ 2°C. Samples and extracts should be stored in suitable glass containers with Teflon lined caps. (Extracts will normally be stored for 30 days after invoicing.)

8.3 Water samples are extracted within seven days of sampling and the extracts are analyzed within forty days of extraction. Solids, sludges, and organic liquids are extracted within fourteen days of sampling and the extracts are analyzed within forty days of extraction.

9 QUALITY CONTROL

9.1 Initial Demonstration of Capability

9.1.1 For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in section 13 must be acceptable before analysis of samples may begin. Refer to the flow chart in section 17.4.1.

9.1.2 For non-standard analytes an MDL study should be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2 Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery +/- 3 standard deviations for surrogates, MS and LCS Precision limits for matrix spikes / matrix spike duplicates are mean relative percent difference +/- 3 standard deviations.

9.2.1 These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported unless the dilution is more

than 5X.

9.2.2 All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.3 Refer to the QC program document (QA-003) for further details of control limits.

9.3 Method Blank

A method blank is prepared and analyzed with each batch of samples. The method blank consists of reagent water for aqueous samples, and sodium sulfate for soil samples (Refer to SOP No. CORP-OP-0001 for details). Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

- If the analyte is a common laboratory contaminant (phthalate esters), the data may be reported with qualifiers if the concentration of the analyte is less than five times the RL. Such action must be taken in consultation with the client.

- Reanalysis of any samples with reportable concentrations of analytes found in the method blank is required unless other actions are agreed with the client.

- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.3.1 The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.3.2 If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments may be made in a narrative to provide further documentation.

9.3.3 Refer to the STL North Canton QC Program document (QA-003) for further details of the corrective actions.

9.3.4 Sample results are NOT blank subtracted unless specific requests and arrangements have been made with a client or agency.

9.4 Instrument Blank

9.4.1 Instruments must be evaluated for contamination during each 12 hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of methylene chloride with the internal standards added. It is evaluated in the same way as the method blank.

9.5 Laboratory Control Sample (LCS)

9.5.1 A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. All analytes must be within established control limits. The LCS is spiked with the compounds listed in Tables 9 and 10 unless specified by a client or agency.

9.5.2 If any analyte in the LCS is outside the laboratory established historical control limits, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS).

- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5.3 Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike/matrix spike duplicate (MS/MSD) is prepared and analyzed with every batch of samples. The MS/MSD is spiked with the same subset of analytes as the LCS

(See Tables 9 and 10). Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.

- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.

- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7 Surrogates

9.7.1 Every sample, blank, and QC sample is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 11.

9.7.2 If any surrogates are outside limits the following corrective actions must take place (except for dilutions):

- Check all calculations for error.

- Ensure that instrument performance is acceptable.

- Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.

- Re-extract and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client.

It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

Note: If all associated QC meets criteria (blank, LCS/LCSD), up to one surrogate per fraction may be outside of acceptance criteria , as long as the recovery is greater than 10%.

9.7.3 If the sample with surrogate recoveries outside the recovery limits was a sample used for an MS/MSD and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample, the MS, and the MSD do not require reanalysis as this phenomenon would indicate a possible matrix problem.

9.7.4 If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only the reanalyzed data should be reported. (Unless the reanalysis was outside holding times, in which case reporting both sets of results may be appropriate.)

9.7.5 If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effect.

9.8 Nonconformance and Corrective Action

9.8.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.9 Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.10 STL North Canton QC Program

Further details of QC and corrective action guidelines are presented in the STL North Canton QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10 CALIBRATION AND STANDARDIZATION

10.1 Summary

10.1.1 The instrument is tuned for DFTPP, calibrated initially with a five-point calibration

curve, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 5.

10.2 All standards and extracts are allowed to warm to room temperature before injecting.

10.3 Instrument Tuning

At the beginning of every twelve hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table 6) is achieved for DFTPP (decafluorotriphenylphosphine).

10.3.1 Inject 10 ng or 50 ng of the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 6 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

10.3.2 The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. Benzidine and pentachlorophenol should not exhibit excessive tailing. If DDT is an analyte of interest, it must be included in the tuning standard, and its breakdown must be < 20%. Refer to section 12 for the appropriate calculations.

10.4 Initial Calibration

10.4.1 Internal Standard Calibration Procedure: Internal standards are listed in Table 7. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

10.4.2 Compounds should be assigned to the IS with the closest retention time.

10.4.3 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest. Six standards must be used for a quadratic least squares calibration. Quadratic fit may NOT be used for samples analyzed under South Carolina Certification. It may also be useful to analyze six calibration levels and use the lower five for most analytes and the upper five for analytes that have poor response. Add the internal standard mixture to result in 8 ng or 40 ng on column. The concentrations of all analytes are listed in tables 12 and 13.

10.4.4 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against concentration for each compound and internal standard. Calculate response factors (RF), average response factors, and the percent RSD of the response factors

for each compound using the equations in section 12 and verify that the CCC and SPCC criteria in section 10.4.5 and 10.4.6 are met. **No sample analysis may be performed unless these criteria are met.**

10.4.5 System Performance Check Compounds (SPCCs): The minimum average RF for semivolatile SPCCs is 0.050. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

SPCC Compounds:

N-nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

10.4.6 Calibration Check Compounds (CCCs): The %RSD of the response factors for each CCC in the initial calibration must be less than 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this criterion.

10.4.6.1 If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.4.6.2 CCC Compounds:

Phenol
Acenaphthene
1,4-Dichlorobenzene
N-nitrosodiphenylamine
2-Nitrophenol
Pentachlorophenol
2,4-Dichlorophenol
Fluoranthene
Hexachlorobutadiene
Di-n-octylphthalate
4-Chloro-3-methylphenol
Benzo(a)pyrene
2,4,6-Trichlorophenol

10.4.7 If the average of all %RSDs in the initial calibration is ≤ 15%, then all analytes may use average response factor for calibration.

10.4.7.1 If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve then the appropriate curve should be used for quantitation.

10.4.7.2 If the average of all the %RSDs in the initial calibration is > 15%, then calibration on a curve must be used for those analytes with %RSD > 15%. Linear or quadratic curve fits may be used. Linear curve fits only may be used for South Carolina Certification. The analyst should consider instrument maintenance to improve the linearity of response. Use of 1/Concentration^2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. If Relative Standard Error (RSE) is used to evaluate the curve it must be better than 15%. If the % RSD is >15%, the analyst may drop the low or high points in the ICAL, as long as a minimum of 5 points are maintained and the quantitation range is adjusted accordingly. If the % RSD is still >15%, a quadratic or linear curve may be used. The correlation coefficient (r) must be ≥ 0.990. If the correlation coefficient is < 0.990, then any hits for these compounds must be flagged as estimated.

10.4.8 Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. For South Carolina Certification, only a linear calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called 1/X^2 weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4.9 If time remains in the 12 hour period initiated by the DFTPP injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.10 Quantitation is performed using the calibration curve or average response factor form the initial curve, not the continuing calibration.

10.5 Continuing Calibration

10.5.1 At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. A 10 ng or 50 ng injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 6.

10.5.2 Following a successful DFTPP analysis the continuing calibration standard(s) are analyzed. The standards must contain all semivolatile analytes, including all required surrogates. A mid level calibration standard is used for the continuing calibration.

10.5.3 The following criteria must be met for the continuing calibration to be acceptable:

- The SPCC compounds must have a response factor of ≥ 0.05.
- The percent difference or drift of the CCC compounds from the initial calibration must be $\leq 20\%$. (see section 12 for calculations) In addition, the percent difference or drift of all analytes must be $\leq 50\%$, with allowance being made for up to six target compounds to have percent drift greater than 50%.
- The internal standard response must be within 50-200% of the response in the mid level of the initial calibration.
- The internal standard retention times must be within 30 seconds of the retention times in the mid-level of the initial calibration.
- NOTE: There is no internal standard criteria for samples. Criteria is only for continuing and initial calibrations.
- NOTE: Ohio VAP rules require that any sample with internal standard outliers be reanalyzed.

10.5.3.1 If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client.

10.5.4 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the DFTPP have passed. (A sample *injected* less than 12 hours after the DFTPP is acceptable.)

NOTE: Additional compound analysis outside the TCL (Target Compound List) have no calibration criteria.

11 PROCEDURE

11.1 Sample Preparation

Samples are prepared following SOP CORP-OP-0001.

11.2 Sample Analysis Procedure

11.2.1 Calibrate the instrument as described in section 10. Depending on the target compounds required by the client, it may be necessary to use more than one calibration standard.

11.2.2 All samples must be analyzed using the same instrument conditions as the preceeding continuing calibration standard.

11.2.3 Add internal standard to the extract to result in 40 ng injected on column (for example, 8 µL or 50 µL internal standard solution in 0.5 mL of extract for a 1 µL injection). Mix thoroughly before injection into the instrument.

11.2.4 Inject the sample extract into the GC/MS system using the same injection technique as used for the standards.

11.2.5 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in section 12. Quantitation is based on the initial calibration, not the continuing calibration.

11.2.6 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst or automatically by the data system.

11.2.7 Target compounds identified by the data system are evaluated using the criteria listed in section 12.1.

11.2.8 Library searches of peaks present in the chromatogram that are not target compounds (Tentatively Identified Compounds, TIC) may be performed if required by the client. They are evaluated using the criteria in section 12.3.

11.3 Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.3.1 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgement. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

11.3.2 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

11.4 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them at 4 ± 2°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

11.5 Retention time criteria for samples

If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.5.1 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

11.6 Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to the facility specific SOP for determination of percent moisture.

11.7 Procedural Variations

11.7.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. Any unauthorized deviations from this procedure must also be documented as a non-conformance, with a cause and corrective action described.

11.8 Troubleshooting Guide

11.8.1 Daily Instrument Maintenance

In addition to the checks listed in the instrument maintenance schedule in the STL North Canton Quality Assurance Manual (LQM), current version, the following daily maintenance should be performed.

- Clip Column as necessary.
- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Perform mass calibration as necessary.

11.8.2 Major Maintenance

A new initial calibration is necessary following major maintenance. Major maintenance includes changing the column, cleaning the ion volume or repeller, cleaning the source, and replacing the multiplier. Refer to the manufacturer's manual for specific guidance.

12 DATA ANALYSIS AND CALCULATIONS

12.1 Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NBS library. Two criteria must be satisfied

to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- The characteristic ions of a compound must maximize in the same scan or within one scan of each other.
- The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

12.1.1 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation.

12.2 Mass chromatogram searches.

Certain compounds are unstable in the calibration standard and cannot be calibrated in the normal way. In particular, the compound hexachlorophene (CAS 70-30-4) falls into this category, and is required for Appendix IX analysis. For this analyte a mass chromatogram search is made.

12.2.1 Hexachlorophene

Display the mass chromatograms for mass 196 and mass 198 for the region of the chromatogram from at least 2 minutes before chrysene-d12 to at least 4 minutes after chrysene-d12. If peaks for both ions coincide then the analyst evaluates the spectrum for the presence of hexachlorophene. No quantitation is possible.

12.3 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines should not use normalization routines that would

misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches shall the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

- Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30%and 70%.)
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or presence of coeluting compounds.
- Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.
- Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

12.4 Anyone evaluating data is trained to know how to handle isomers with identical mass spectra and close elution times. These include:

Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

Extra precautions concerning these compounds are to more closely scrutinize retention time vs. the calibration standard and also to check that all isomers have distinct retention times.

A second category of problem compounds would be the poor responders or compounds that chromatograph poorly. Included in this category would be:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol

Manually checking the integrations would be appropriate for these compounds.

12.5 Calculations

12.5.1 Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{RF} \times 100$$

RF = Mean of RFs from intial caibration for a compound

SD = Standard deviation of RFs from initial calibration for a compound,

$$= \sqrt{\sum_{i=1}^{N} \frac{\left(RFi - \overline{RF}\right)^2}{N-1}}$$

RFi = RF for each of the calibration levels

N = Number of RF values

12.5.2 Continuing calibration percent drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

C_{actual} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.5.3 Concentration in the extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration. For South Carolina Certification the concentration

of each identified analyte and surrogate in the extract is calculated from the linear curve only fitted to the initial calibration points, or from the average RF of the initial calibration.

12.5.3.1 Average response factor

If the average of all the %RSDs of the response factors in the initial calibration is ≤ 15%, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

12.5.3.2 Linear fit (Use only Linear fit for South Carolina Certification)

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

C_{ex}= Concentration in extract, µg/mL

R_x= Response for analyte

R_{is}= Response for internal standard

C_{is}= Concentration of internal standard

A= Intercept

B= Slope

12.5.3.3 Quadratic fit

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)$$

C= Curvature

12.5.4 The concentration in the sample is then calculated.

12.5.4.1 Aqueous Calculation

$$\text{Concentration, } \mu g / L = \frac{C_{ex} V_t}{V_o}$$

Where:

V_t = Volume of total extract, µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1 mL extract will mean V_t = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, V_t = 2,000.)

V_o = Volume of water extracted (mL)

12.5.5 Sediment/Soil, Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis:

$$\text{Concentration, } \mu g / kg = \frac{C_{ex} V_t}{W_s D}$$

W_s = Weight of sample extracted or diluted in grams

D = (100 - % moisture in sample)/100, for a dry weight basis or 1 for a wet weight basis

12.6 MS/MSD percent recovery calculation.

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

S_{SR} = Spike sample result

S_R = Sample result

S_A = Spike added

12.7 Relative % Difference calculation for the MS/MSD

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

RPD = Relative percent difference

MS_R = Matrix spike result

MSD_R = Matrix spike duplicate result

12.8 Relative response factor calculation.

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

A_x=Area of the characteristic ion for the compound being measured

A_{is}=Area of the characteristic ion for the specific internal standard

C_x=Concentration of the compound being measured (µg/L)

C_{is} =Concentration of the specific internal standard (µg/L)

12.9 Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x = Area of the total ion chromatogram for the compound being measured

A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

12.10 Percent DDT breakdown

$$\% \text{ DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDarea}}$$

The total ion current areas are used for this calculation

13 METHOD PERFORMANCE

13.1 Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: QA-005.

13.2 Initial Demonstration

Each laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to the level 4 calibration standard.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in table 14.

13.2.3 If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3 Non-standard analytes

For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

13.4 Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.5 Data Quality Objectives (DQO). Refer to project-specific Quality Assurance plans for DQO information.

14 POLLUTION PREVENTION

14.1 This section is not applicable to this procedure.

15 WASTE MANAGEMENT

15.1 Waste generated during aliquotting and from used vials must be disposed of in accordance with the facility hazardous waste procedures. The Health and Safety Director should be contacted if additional information is required.

16 REFERENCES

16.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update II, October 1994, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270C.

16.2 J. W. Eichelberger, L. E. Harris, and W. L. Budde, "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography/Mass Spectrometry," Analytical Chemistry, 47, 995 (1975)

17 MISCELLANEOUS

17.1 Modifications from Reference Method

17.1.1 A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.2 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.2 Modifications from Previous Revision

17.2.1 This SOP has been substantially revised to meet the requirements of method 8270C.

17.2.2 Directions for analysis be method 625 have been added as an attachment.

17.2.3 SOP has been revised to include a 5 mL final volume.

17.3 Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4 Tables

Table 1

STL North Canton Primary Standard[1] and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Pyridine	110-86-1	20	660
N-nitrosodimethylamine	62-75-9	10	330
Aniline	62-53-3	10	330
Phenol	108-95-2	10	330
Bis(2-chloroethyl)ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	10	330
1,4-Dichlorobenzene	106-46-7	10	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	10	330
2-Methylphenol	95-48-7	10	330
2,2'-oxybis(1-chloropropane)[2]	108-60-1	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic acid	65-85-0	50	1600
Bis(2-chloroethoxy)methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene	77-47-4	50	1600
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2-Chloronaphthalene	91-58-7	10	330
2-Nitroaniline	88-74-4	50	1600
Dimethyl phthalate	131-11-3	10	330
Acenaphthylene	208-96-8	10	330
3-Nitroaniline	99-09-2	50	1600
Acenaphthene	83-32-9	10	330
2,4-Dinitrophenol	51-28-5	50	1600
4-Nitrophenol	100-02-7	50	1600
Dibenzofuran	132-64-9	10	330

Table 1

STL North Canton Primary Standard[1] and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
2,4-Dinitrotoluene	121-14-2	10	330
2,6-Dinitrotoluene	606-20-2	10	330
Diethylphthalate	84-66-2	10	330
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	10	330
4-Nitroaniline	100-01-6	50	1600
4,6-Dinitro-2-methylphenol	534-52-1	50	1600
N-Nitrosodiphenylamine	86-30-6	10	330
Azobenzene	103-33-3	10	330
4-Bromophenyl phenyl ether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Pentachlorophenol	87-86-5	50	1600
Phenanthrene	85-01-8	10	330
Anthracene	120-12-7	10	330
Carbazole	86-74-8	10	330
Di-n-butyl phthalate	84-74-2	10	330
Fluoranthene	206-44-0	10	330
Benzidine	92-87-5	100	3300
Pyrene	129-00-0	10	330
Butyl benzyl phthalate	85-68-7	10	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
Benzo(a)anthracene	56-55-3	10	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
Chrysene	218-01-9	10	330
Di-n-octylphthalate	117-84-0	10	330
Benzo(b)fluoranthene	205-99-2	10	330
Benzo(k)fluoranthene	207-08-9	10	330
Benzo(a)pyrene	50-32-8	10	330
Indeno(1,2,3-cd)pyrene	193-39-5	10	330
Dibenz(a,h)anthracene	53-70-3	10	330
Benzo(g,h,i)perylene	191-24-2	10	330
Benzaldehyde	100-52-7	10	330
Caprolactam	105-60-2	10	330
1,1-Biphenyl	92-52-4	10	330
Atrazine	1912-24-9	10	330

[1] The STL North Canton primary standard is the standard normally used at F. Additional standards, such as the Appendix IX standard may be necessary to include all target analytes required for some clients.

[2] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

Table 2

STL North Canton Appendix IX[1] Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
2-Picoline	109-06-8	20	660
N-Nitrosomethylethylamine	10595-95-6	10	330
Methyl methanesulfonate	66-27-3	10	330
N-Nitrosodiethylamine	55-18-5	10	330
Ethyl methanesulfonate	62-50-0	10	330
Pentachloroethane	76-01-7	50	1600
Acetophenone	98-86-2	10	330
N-Nitrosopyrrolidine	930-55-2	10	330
N-Nitrosomorpholine	59-89-2	10	330
o-Toluidine	95-53-4	20	660
3-Methylphenol	108-39-4	10	330
N-Nitrosopiperidine	100-75-4	10	330
o,o,o-Triethyl-Phosphorothioate[2]	126-68-1	50	1600
a,a-Dimethyl-phenethylamine	122-09-8	50	1600
2,6-Dichlorophenol	87-65-0	10	330
Hexachloropropene	1888-71-7	100	3300
p-Phenylenediamine	106-50-3	100	3300
n-Nitrosodi-n-butylamine	924-16-3	10	330
Safrole	94-59-7	20	660
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330
Isosafrole	120-58-1	20	660
1,4-Dinitrobenzene	100-25-4	10	330
1,4-Naphthoquinone	130-15-4	50	1600
1,3-Dinitrobenzene	99-65-0	10	330
Pentachlorobenzene	608-93-5	10	330
1-Naphthylamine	134-32-7	10	330
2-Naphthylamine	91-59-8	10	330
2,3,4,6-Tetrachlorophenol	58-90-2	50	1600
5-Nitro-o-toluidine	99-55-8	20	660
Thionazin[2]	297-97-2	50	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp[2]	3689-24-5	50	1600
Phorate[2]	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Diallate[3]	2303-16-4	20	660
Dimethoate[2]	60-51-5	20	660
4-Aminobiphenyl	92-67-1	50	1600
Pentachloronitrobenzene	82-68-8	50	1600
Pronamide	23950-58-5	20	660
Disulfoton[2]	298-04-4	50	1600
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7	20	660
*Methyl Parathion[2]	298-00-0	50	1600

Table 2

STL North Canton Appendix IX[1] Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
4-Nitroquinoline-1-oxide	56-57-5	100	3300
*Parathion[2]	56-38-2	50	1600
Methapyrilene	91-80-5	50	1600
Aramite	140-57-8	20	660
*Isodrin[3]	465-73-6	10	330
*Kepone[2]	143-50-0	100	3300
Famphur[3]	52-85-7	100	3300
p-(Dimethylamino)azobenzene	60-11-7	20	660
p-Chlorobenzilate[3]	510-15-6	10	330
3,3'-Dimethylbenzidine	119-93-7	50	1600
2-Acetylaminofluorene	53-96-3	100	3300
Dibenz(a,j)acridine	224-42-0	20	660
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660
3-Methylcholanthrene	56-49-5	20	660

1 The Appendix IX standard contains additional analytes required for the Appendix IX list. The STL North Canton primary standard must also be analyzed to include all of the Appendix IX list

2 May also be analyzed by method 8140 or 8141, which can achieve lower reporting limits.

3 It is highly recommended that Famphur is analyzed by method 8081. It is a poor responder by 8270C.

* These compounds are analyzed by GC methods at STL North Canton.

Table 2A
STL North Canton Michigan Program[1]

Semivolatile	CAS Number	Michigan Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Acenaphthene	83-32-9	5	330
Acenaphthylene	208-96-8	5	330
Acetophenone	98-86-2	5	330
Anthracene	120-12-7	5	330
Atrzine	1912-24-9	5	330
Benzaldehyde	100-52-7	10	330
Benzo(a)anthracene	56-55-3	4	330
Benzo(a)pyrene	50-32-8	4	330
Benzo(b)fluoranthene	205-99-2	4	330
Benzo(g,h,i)perylene	191-24-2	5	330
Benzo(k)fluoranthene	207-08-9	5	330
1,1'-Biphenyl	92-52-4	10	330
4-Bromophenylphenyl ehter	101-55-3	5	330
Butylbenzylphthalate	85-68-7	5	330
di-n-Butylphthalate	84-74-2	5	330
Caprolactam	105-60-2	10	330
Carbazole	86-74-8	10	330
4-Chloroaniline	106-47-8	20	1700
bis(2-Chloroethoxy)methane	111-91-1	5	330
bis(2-Chloroethyl)ether	111-44-4	4	330
bis(2-Chloroisopropyl)ether	108-60-1	5	330
4-Chloro-3-Methylphenol	59-50-7	5	330
2-Chloronaphthalene	91-58-7	5	330
2-Chlorophenol	95-57-8	5	330
4-Chlorophenyl phenyl ether	7005-72-3	5	330
Chrysene	218-01-9	5	330
Dibenz(a,h)anthracene	53-70-3	4	330
Dibenzofuran	132-64-9	5	330
3,3'-Dichlorobenzidine	91-94-1	4	2000
2,4-Dichlorophenol	120-83-2	10	330
Diethylphthalate	84-66-2	5	330
2-4-Dimethylphenol	105-67-9	5	330
Dimethylphthalate	131-11-3	5	330
4,6-Dinitro-2-methylphenol	534-52-1	20	1700
2,4-Dinitrophenol	51-28-5	20	1700
2,4-Dinitrotoluene	121-14-2	5	330
2,6-Dinitrotoluene	606-20-2	5	330
bis(2-Ethylhexyl)phthalate	117-81-7	5	330
Fluoranthene	206-44-0	5	330
Fluorene	86-73-7	5	330
Hexachlorobenzene	118-74-1	5	330
Hexachlorobutadiene	87-68-3	5	330
Hexachlorocyclopentadiene	77-47-4	5	330

Table 2A
STL North Canton Michigan Program[1]

Semivolatile	CAS Number	Michigan Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
Hexachloroethane	67-72-1	5	330
Indeno(1,2,3-cd)pyrene	193-39-5	4	330
Isophorone	78-59-1	5	330
2-Methylnapthalene	91-57-6	5	330
2-Methylphenol	95-48-7	5	330
4-Methylphenol	106-44-5	5	330
Naphthalene	91-20-3	5	330
2-Nitroaniline	88-74-4	20	1700
3-Nitroaniline	99-09-2	20	1700
4-Nitroaniline	100-01-6	20	1700
Nitrobenzene	95-95-3	4	330
2-Nitrophenol	88-75-5	5	330
4-Nitrophenol	100-02-7	20	1700
N-Nitroso-di-n-propylamine	621-64-7	5	330
N-Nitrosodiphenylamine (diphenylamine)	62-75-9	5	330
di-n-Octylphthalate	117-84-0	5	330
Pentachlorophenol	87-86-5	20	800
Phenanthrene	85-01-8	5	330
Phenol	108-95-2	5	330
Pyrene	129-00-0	5	330
2,4,5-Trichlorophenol	95-95-4	5	330
2,4,6-Trichlorophenol	88-06-2	4	330

[1] Reporting Limits are only for samples performed under the Michigan program.

Table 3

Reportable Analytes for STL North Canton Standard Tests, Primary Standard

Analyte	CAS Number	STL North Canton Standard List	TCLP	TCL	Appendix IX
Pyridine	110-86-1		X		X
N-nitrosodimethylamine	62-75-9				X
Aniline	62-53-3				X
Phenol	108-95-2	X		X	X
Bis(2-chloroethyl)ether	111-44-4	X		X	X
2-Chlorophenol	95-57-8	X		X	X
1,3-Dichlorobenzene	541-73-1	X		X	X
1,4-Dichlorobenzene	106-46-7	X	X	X	X
Benzyl alcohol	100-51-6				X
1,2-Dichlorobenzene	95-50-1	X		X	X
2-Methylphenol	95-48-7	X	X	X	X
2,2'-oxybis(1-chloropropane)[1]	180-60-1	X		X	X

Table 3

Reportable Analytes for STL North Canton Standard Tests, Primary Standard

Analyte	CAS Number	STL North Canton Standard List	TCLP	TCL	Appendix IX
4-Methylphenol	106-44-5	X	X	X	X
N-Nitroso-di-n-propylamine	621-64-7	X		X	X
Hexachloroethane	67-72-1	X	X	X	X
Nitrobenzene	98-95-3	X	X	X	X
Isophorone	78-59-1	X		X	X
2-Nitrophenol	88-75-5	X		X	X
2,4-Dimethylphenol	105-67-9	X		X	X
Benzoic acid	65-85-0				
Bis(2-chloroethoxy)methane	111-91-1	X		X	X
2,4-Dichlorophenol	120-83-2	X		X	X
1,2,4-Trichlorobenzene	120-82-1	X		X	X
Naphthalene	91-20-3	X		X	X
4-Chloroaniline	106-47-8	X		X	X
Hexachlorobutadiene	87-68-3	X	X	X	X
4-Chloro-3-methylphenol	59-50-7	X		X	X
2-Methylnaphthalene	91-57-6	X		X	X
Hexachlorocyclopentadiene	77-47-4	X		X	X
2,4,6-Trichlorophenol	88-06-2	X	X	X	X
2,4,5-Trichlorophenol	95-95-4	X	X	X	X
2-Chloronaphthalene	91-58-7	X		X	X
2-Nitroaniline	88-74-4	X		X	X
Dimethyl phthalate	131-11-3	X		X	X
Acenaphthylene	208-96-8	X		X	X
3-Nitroaniline	99-09-2	X		X	X
Acenaphthene	83-32-9	X		X	X
2,4-Dinitrophenol	51-28-5	X		X	X
4-Nitrophenol	100-02-7	X		X	X
Dibenzofuran	132-64-9	X		X	X
2,4-Dinitrotoluene	121-14-2	X	X	X	X
2,6-Dinitrotoluene	606-20-2	X		X	X
Diethylphthalate	84-66-2	X		X	X
4-Chlorophenyl phenyl ether	7005-72-3	X		X	X
Fluorene	86-73-7	X		X	X
4-Nitroaniline	100-01-6	X		X	X
4,6-Dinitro-2-methylphenol	534-52-1	X		X	X
N-Nitrosodiphenylamine	86-30-6	X		X	X
Azobenzene[4]	103-33-3				
4-Bromophenyl phenyl ether	101-55-3	X		X	X
Hexachlorobenzene	118-74-1	X	X	X	X
Pentachlorophenol	87-86-5	X	X	X	X
Phenanthrene	85-01-8	X		X	X
Anthracene	120-12-7	X		X	X
Carbazole	86-74-8	X		X	
Di-n-butyl phthalate	84-74-2	X		X	X

Table 3

Reportable Analytes for STL North Canton Standard Tests, Primary Standard

Analyte	CAS Number	STL North Canton Standard List	TCLP	TCL	Appendix IX
Fluoranthene	206-44-0	X		X	X
Benzidine	92-87-5				
Pyrene	129-00-0	X		X	X
Butyl benzyl phthalate	85-68-7	X		X	X
3,3'-Dichlorobenzidine	91-94-1	X		X	X
Benzo(a)anthracene	56-55-3	X		X	X
Bis(2-ethylhexyl)phthalate	117-81-7	X		X	X
Chrysene	218-01-9	X		X	X
Di-n-octylphthalate	117-84-0	X		X	X
Benzo(b)fluoranthene	205-99-2	X		X	X
Benzo(k)fluoranthene	207-08-9	X		X	X
Benzo(a)pyrene	50-32-8	X		X	X
Indeno(1,2,3-cd)pyrene	193-39-5	X		X	X
Dibenz(a,h)anthracene	53-70-3	X		X	X
Benzo(g,h,i)perylene	191-24-2	X		X	X
Benzaldehyde	100-52-7			X	
Caprolactam	105-60-2			X	
1,1-Biphenyl	92-52-4			X	
Atrazine	1912-24-9			X	

[1] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

[2] Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 4

Reportable analytes for STL North Canton Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	STL North Canton Standard List	TCLP	TCL	Appendix IX
2-Picoline	109-06-8				X
N-Nitrosomethylethylamine	10595-95-6				X
Methyl methanesulfonate	66-27-3				X
N-Nitrosodiethylamine	55-18-5				X
Ethyl methanesulfonate	62-50-0				X
Pentachloroethane	76-01-7				X
Acetophenone	98-86-2			X	X
N-Nitrosopyrrolidine	930-55-2				X
N-Nitrosomorpholine	59-89-2				X
o-Toluidine	95-53-4				X
3-Methylphenol	108-39-4				X
N-Nitrosopiperidine	100-75-4				X
o,o,o-Triethyl-Phosphorothioate[2]	126-68-1				X
a,a-Dimethyl-phenethylamine	122-09-8				X
2,6-Dichlorophenol	87-65-0				X
Hexachloropropene	1888-71-7				X
p-Phenylenediamine	106-50-3				X
n-Nitrosodi-n-butylamine	924-16-3				X
Safrole	94-59-7				X
1,2,4,5-Tetrachlorobenzene	95-94-3				X
Isosafrole	120-58-1				X
1,4-Dinitrobenzene	100-25-4				
1,4-Naphthoquinone	130-15-4				X
1,3-Dinitrobenzene	99-65-0				X
Pentachlorobenzene	608-93-5				X
1-Naphthylamine	134-32-7				X
2-Naphthylamine	91-59-8				X
2,3,4,6-Tetrachlorophenol	58-90-2				X
5-Nitro-o-toluidine	99-55-8				X
Thionazin[2]	297-97-2				X
1,3,5-Trinitrobenzene	99-35-4				X
Sulfotepp[2]	3689-24-5				X
Phorate[2]	298-02-2				X
Phenacetin	62-44-2				X
Diallate	2303-16-4				X
Dimethoate[2]	60-51-5				X
4-Aminobiphenyl	92-67-1				X
Pentachloronitrobenzene	82-68-8				X
Pronamide	23950-58-5				X
Disulfoton[2]	298-04-4				X
2-secbutyl-4,6-dinitrophenol (Dinoseb)[2]	88-85-7				X
*Methyl parathion[2]	298-00-0				X

Table 4

Reportable analytes for STL North Canton Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	STL North Canton Standard List	TCLP	TCL	Appendix IX
4-Nitroquinoline-1-oxide	56-57-5				X
*Parathion[2]	56-38-2				X
*Isodrin[3]	465-73-6				X
*Kepone [2]	143-50-0				X
Famphur[2]	52-85-7				X
Methapyrilene	91-80-5				X
Aramite	140-57-8				X
p-(Dimethylamino)azobenzene	60-11-7				X
p-Chlorobenzilate[3]	510-15-6				X
3,3'-Dimethylbenzidine	119-93-7				X
2-Acetylaminofluorene	53-96-3				X
Dibenz(a,j)acridine	224-42-0				
7,12-Dimethylbenz(a)anthracene	57-97-6				X
3-Methylcholanthrene	56-49-5				X
Hexachlorophene[4]	70-30-4				X
Diphenylamine[5]	122-39-4				X

[2] May also be analyzed by method 8140 or 8141, which can achieve lower reporting limits.

[3] May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits

[4] Hexachlorophene is a required analyte for Appendix IX. This compound is not stable, and therefore not included in the calibration standard. The characteristic ions for hexachlorophene are searched for in the chromatogram. (See section 12.2.1)

[5] Diphenylamine is a required compound for Appendix IX. N-nitrosodiphenylamine decomposes in the injection port to form diphenylamine. Therefore these two compounds cannot be distinguished. Diphenylamine is not included in the calibration standard.

* These compounds are analyzed by GC methods at STL North Canton.

Table 5

Suggested Instrumental Conditions

Mass Range	35-500 amu
Scan Time	≤1 second/scan
Initial Column Temperature/Hold Time	40°C for 2 minutes
Column Temperature Program	40 - 320°C at 11.5°C/min
Final Column Temperature/Hold Time	320°C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature	250 - 300°C
Transfer Line Temperature	250 - 300°C
Source Temperature	According to manufacturer's specifications
Injector	Grob-type, split / splitless
Sample Volume	1 or 2 µl
Carrier Gas	Helium at 30 cm/sec

Table 6

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 - 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17 - 23% of mass 442

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
N-nitrosodimethylamine	74	42	
Pyridine	79	52	
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d5 (Surrogate Standard)	99	42	71
Benzaldehyde	77	105	106
Aniline	93	66	
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	79
2,2'-oxybis(1-chloropropane)[1]	45	77	79
4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d8 (Internal Standard)	136	68	54
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
Caprolactam	113	55	56
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	115
Hexachlorocyclopentadiene	237	235	272
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
1,1'-Biphenyl	154	153	76
2-Fluorobiphenyl (Surrogate Standard)	172	171	170
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
Acenaphthylene	152	151	153
2,6-Dinitrotoluene	165	63	89
Acenaphthene-d10 (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153	152	154
2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	84
4-Nitrophenol	109	139	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	182	77
N-Nitrosodiphenylamine	169	168	167
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141
Azobenzene	77	182	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Atrazine	200	173	215
Pentachlorophenol	266	264	268
Phenanthrene-d10 (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	100
Benzidine	184	92	185
Pyrene	202	101	100
Terphenyl-d14 (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Benzo(a)Anthracene	228	229	226
Chrysene-d12 (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d12 (Internal Standard)	264	260	265
Indeno(1,2,3-cd)pyrene	276	138	277
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
2-Picoline	93	66	92
N-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
N-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Pentachloroethane	117	119	167
Acetophenone	105	77	120
N-Nitrosopyrrolidine	100	41	42
N-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	
3-Methylphenol	108	107	77
N-Nitrosopiperidine	114	42	55
o,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
p-Phenylenediamine	108	80	
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole 1	162	104	131
Isosafrole 2	162	104	131
1,4-Dinitrobenzene	168	75	122
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	75	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	
2-Naphthylamine	143	115	
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	97	96	143
1,3,5-Trinitrobenzene	213	75	120
Sulfotepp	97	322	202
Phorate	75	97	121
Phenacetin	108	179	109
Diallate	86	234	
Dimethoate	87	93	125
4-Aminobiphenyl	169		
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263
4-Nitroquinoline-1-oxide	190	128	160

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
Parathion	109	97	291
Isodrin	193	66	195
Kepone	272	274	237
Famphur	218	125	93
Methapyrilene	97	58	
Aramite 1	185	319	
Aramite 2	185	319	
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	
2-Acetylaminofluorene	181	180	223
Dibenz(a,j)acridine	279	280	
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

Table 9

8270C LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract[1]
1,2,4-Trichlorobenzene	100
Acenaphthene	100
2,4-Dinitrotoluene	100
Pyrene	100
N-Nitroso-di-n-propylamine	100
1,4-Dichlorobenzene	100
Pentachlorophenol	150
Phenol	150
2-Chlorophenol	150
4-Chloro-3-methylphenol	150
4-Nitrophenol	150

[1] Levels are 50 and 75 ng/µL if 2 µL injection is used

Table 10

TCLP LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract[1]
1,4-Dichlorobenzene	100
2,4-Dinitrotoluene	100
Hexachlorobenzene	100
Hexachlorobutadiene	100
Hexachloroethane	100
2-Methylphenol	100
3-Methylphenol	100
4-Methylphenol	100
Nitrobenzene	100
Pentachlorophenol	100
Pyridine	100
2,4,5-Trichlorophenol	100
2,4,6-Trichlorophenol	100

[1] Levels are 50 ng/µL if 2 µL injection is used

Recovery limits for the LCS and for matrix spikes are generated from historical data and are maintained by the QA department.

Table 11

8270C Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract[2]
Nitrobenzene-d5	100
2-Fluorobiphenyl	100
Terphenyl-d14	100
1,2-Dichlorobenzene-d4[1]	100
Phenol-d5	150
2-Fluorophenol	150
2,4,6-Tribromophenol	150
2-Chlorophenol-d4[1]	150

[1] Included in standard mix, but not routinely evaluated for method 8270B

[2] Levels are 50 and 75 ng/µL if 2 µL injection is used

Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 12[3]
Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
Pyridine	10	25	40	60	80
N-nitrosodimethylamine	10	25	40	60	80
Aniline	10	25	40	60	80
Phenol	10	25	40	60	80
Bis(2-chloroethyl)ether	10	25	40	60	80
2-Chlorophenol	10	25	40	60	80
1,3-Dichlorobenzene	10	25	40	60	80
1,4-Dichlorobenzene	10	25	40	60	80
Benzyl alcohol	10	25	40	60	80
1,2-Dichlorobenzene	10	25	40	60	80
2-Methylphenol	10	25	40	60	80
2,2'-oxybis(1-chloropropane)[1]	10	25	40	60	80
4-Methylphenol	10	25	40	60	80
N-Nitroso-di-n-propylamine	10	25	40	60	80
Hexachloroethane	10	25	40	60	80
Nitrobenzene	10	25	40	60	80
Isophorone	10	25	40	60	80
2-Nitrophenol	10	25	40	60	80
2,4-Dimethylphenol	10	25	40	60	80
Benzoic acid	10	25	40	60	80
Bis(2-chloroethoxy)methane	10	25	40	60	80
2,4-Dichlorophenol	10	25	40	60	80
1,2,4-Trichlorobenzene	10	25	40	60	80
Naphthalene	10	25	40	60	80
4-Chloroaniline	10	25	40	60	80
Hexachlorobutadiene	10	25	40	60	80
4-Chloro-3-methylphenol	10	25	40	60	80
2-Methylnaphthalene	10	25	40	60	80
Hexachlorocyclopentadiene	10	25	40	60	80
2,4,6-Trichlorophenol	10	25	40	60	80
2,4,5-Trichlorophenol	10	25	40	60	80
2-Chloronaphthalene	10	25	40	60	80
2-Nitroaniline	10	25	40	60	80
Dimethyl phthalate	10	25	40	60	80
Acenaphthylene	10	25	40	60	80
3-Nitroaniline	10	25	40	60	80
Acenaphthene	10	25	40	60	80
2,4-Dinitrophenol	10	25	40	60	80
4-Nitrophenol	10	25	40	60	80
Dibenzofuran	10	25	40	60	80
2,4-Dinitrotoluene	10	25	40	60	80
2,6-Dinitrotoluene	10	25	40	60	80
Diethylphthalate	10	25	40	60	80
4-Chlorophenyl phenyl ether	10	25	40	60	80
Fluorene	10	25	40	60	80
4-Nitroaniline	10	25	40	60	80

Table 12[3]
Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5
4,6-Dinitro-2-methylphenol	10	25	40	60	80
N-Nitrosodiphenylamine	10	25	40	60	80
Azobenzene[2]	10	25	40	60	80
4-Bromophenyl phenyl ether	10	25	40	60	80
Hexachlorobenzene	10	25	40	60	80
Pentachlorophenol	10	25	40	60	80
Phenanthrene	10	25	40	60	80
Anthracene	10	25	40	60	80
Carbazole	10	25	40	60	80
Di-n-butyl phthalate	10	25	40	60	80
Fluoranthene	10	25	40	60	80
Benzidine	10	25	40	60	80
Pyrene	10	25	40	60	80
Butyl benzyl phthalate	10	25	40	60	80
3,3'-Dichlorobenzidine	10	25	40	60	80
Benzo(a)anthracene	10	25	40	60	80
Bis(2-ethylhexyl)phthalate	10	25	40	60	80
Chrysene	10	25	40	60	80
Di-n-octylphthalate	10	25	40	60	80
Benzo(b)fluoranthene	10	25	40	60	80
Benzo(k)fluoranthene	10	25	40	60	80
Benzo(a)pyrene	10	25	40	60	80
Indeno(1,2,3-cd)pyrene	10	25	40	60	80
Dibenz(a,h)anthracene	10	25	40	60	80
Benzo(g,h,i)perylene	10	25	40	60	80
Benzaldehyde	10	25	40	60	80
Caprolactam	10	25	40	60	80
1,1'-Biphenyl	10	25	40	60	80
Atrazine	10	25	40	60	80

[1] 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether

[2]Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

[3]Optional concentration levels = 1/5 of the listed concentrations for samples with a final volume of 5 mL.

Table 13[1]

Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5
2-Picoline	10	25	40	60	80
N-Nitrosomethylethylamine	10	25	40	60	80
Methyl methanesulfonate	10	25	40	60	80
N-Nitrosodiethylamine	10	25	40	60	80
Ethyl methanesulfonate	10	25	40	60	80
Pentachloroethane	10	25	40	60	80
Acetophenone	10	25	40	60	80
N-Nitrosopyrrolidine	10	25	40	60	80
N-Nitrosomorpholine	10	25	40	60	80
o-Toluidine	10	25	40	60	80
3-Methylphenol	10	25	40	60	80
N-Nitrosopiperidine	10	25	40	60	80
o,o,o-Triethyl-Phosphorothioate	10	25	40	60	80
a,a-Dimethyl-phenethylamine	10	25	40	60	80
2,6-Dichlorophenol	10	25	40	60	80
Hexachloropropene	10	25	40	60	80
p-Phenylenediamine	10	25	40	60	80
n-Nitrosodi-n-butylamine	10	25	40	60	80
Safrole	10	25	40	60	80
1,2,4,5-Tetrachlorobenzene	10	25	40	60	80
Isosafrole 1 + 2	10	25	40	60	80
1,4-Dinitrobenzene	10	25	40	60	80
1,4-Naphthoquinone	10	25	40	60	80
1,3-Dinitrobenzene	10	25	40	60	80
Pentachlorobenzene	10	25	40	60	80
1-Naphthylamine	10	25	40	60	80
2-Naphthylamine	10	25	40	60	80
2,3,4,6-Tetrachlorophenol	10	25	40	60	80
5-Nitro-o-toluidine	10	25	40	60	80
Thionazin	10	25	40	60	80
1,3,5-Trinitrobenzene	10	25	40	60	80
Sulfotepp	10	25	40	60	80
Phorate	10	25	40	60	80
Phenacetin	10	25	40	60	80
Diallate 1 + 2	10	25	40	60	80
Dimethoate	10	25	40	60	80
4-Aminobiphenyl	10	25	40	60	80
Pentachloronitrobenzene	10	25	40	60	80
Pronamide	10	25	40	60	80
Disulfoton	10	25	40	60	80
2-secbutyl-4,6-dinitrophenol (Dinoseb)	10	25	40	60	80
*Methyl parathion	10	25	40	60	80
4-Nitroquinoline-1-oxide	10	25	40	60	80
*Parathion	10	25	40	60	80

Table 13[1]

Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5
*Isodrin	10	25	40	60	80
*Kepone	10	25	40	60	80
Famphur	10	25	40	60	80
Methapyrilene	10	25	40	60	80
Aramite 1 and 2	10	25	40	60	80
p-(Dimethylamino)azobenzene	10	25	40	60	80
p-Chlorobenzilate	10	25	40	60	80
3,3'-Dimethylbenzidine	10	25	40	60	80
2-Acetylaminofluorene	10	25	40	60	80
Dibenz (a,j)acridine	10	25	40	60	80
7,12-Dimethylbenz(a)anthracene	10	25	40	60	80
3-Methylcholanthrene	10	25	40	60	80

* These compounds are not in the STL North Canton Standard Mix,

[2]Optional concentration levels = 1/5 of the listed concentrations for samples with a final volume of 5 mL.

Table 14
Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
Acenaphthene	60	27.6	60.1-132.3
Acenaphthylene	60	40.2	53.5-126.0
Aldrin[1]	60	39.0	7.2-152.2
Anthracene	60	32.0	43.4-118.0
Benz(a)anthracene	60	27.6	41.8-133.0
Benzo(b)fluoranthene	60	38.8	42.0-140.4
Benzo(k)fluoranthene	60	32.3	25.2-145.7
Benzo(a)pyrene	60	39.0	31.7-148.0
Benzo(ghi)perylene	60	58.9	D-195.0
Benzylbutyl phthalate	60	23.4	D-139.9
B-BHC[1]	60	31.5	41.5-130.6
d-BHC[1]	60	21.6	D-100.0
Bis(2-chloroethyl) ether	60	55.0	42.9-126.0
Bis(2-chloroethoxy)methane	60	34.5	49.2-164.7
Bis(2-chloroisopropyl) ether	60	46.3	62.8-138.6
Bis(2-ethylhexyl) phthalate	60	41.1	28.9-136.8
4-Bromophenyl phenyl ether	60	23.0	64.9-114.4
2-Chloronaphthalene	60	13.0	64.5-113.5
4-Chlorophenyl phenyl ether	60	33.4	38.4-144.7
Chrysene	60	48.3	44.1-139.9
4,4'-DDD[1]	60	31.0	D-134.5

Table 14
Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
4,4'-DDE[1]	60	32.0	19.2-119.7
4,4'-DDT[1]	60	61.6	D-170.6
Dibenzo(a,h)anthracene	60	70.0	D-199.7
Di-n-butyl phthalate	60	16.7	8.4-111.0
1,2-Dichlorobenzene	60	30.9	48.6-112.0
1,3-Dichlorobenzene	60	41.7	16.7-153.9
1,4-Dichlorobenzene	60	32.1	37.3-105.7
3,3'-Dichlorobenzidine	60	71.4	8.2-212.5
Dieldrin[1]	60	30.7	44.3-119.3
Diethyl phthalate	60	26.5	D-100.0
Dimethyl phthalate	60	23.2	D-100.0
2,4-Dinitrotoluene	60	21.8	47.5-126.9
2,6-Dinitrotoluene	60	29.6	68.1-136.7
Di-n-octylphthalate	60	31.4	18.6-131.8
Endosulfan sulfate[1]	60	16.7	D-103.5
Endrin aldehyde	60	32.5	D-188.8
Fluoranthene	60	32.8	42.9-121.3
Fluorene	60	20.7	71.6-108.4
Heptachlor[1]	60	37.2	D-172.2
Heptachlor epoxide[1]	60	54.7	70.9-109.4
Hexachlorobenzene	60	24.9	7.8-141.5
Hexachlorobutadiene	60	26.3	37.8-102.2
Hexachloroethane	60	24.5	55.2-100.0
Indeno(1,2,3-cd)pyrene	60	44.6	D-150.9
Isophorone	60	63.3	46.6-180.2
Naphthalene	60	30.1	35.6-119.6
Nitrobenzene	60	39.3	54.3-157.6
N-Nitrosodi-n-propylamine	60	55.4	13.6-197.9
PCB-1260[1]	60	54.2	19.3-121.0
Phenanthrene	60	20.6	65.2-108.7
Pyrene	60	25.2	69.6-100.0
1,2,4-Trichlorobenzene	60	28.1	57.3-129.2
4-Chloro-3-methylphenol	60	37.2	40.8-127.9
2-Chlorophenol	60	28.7	36.2-120.4
2,4-Chlorophenol	60	26.4	52.5-121.7
2,4-Dimethylphenol	60	26.1	41.8-109.0
2,4-Dinitrophenol	60	49.8	D-172.9
2-Methyl-4,6-dinitrophenol	60	93.2	53.0-100.0
2-Nitrophenol	60	35.2	45.0-166.7
4-Nitrophenol	60	47.2	13.0-106.5
Pentachlorophenol	60	48.9	38.1-151.8
Phenol	60	22.6	16.6-100.0

Table 14
Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
2,4,6-Trichlorophenol	60	31.7	52.4-129.2

[1]Since the organochlorine pesticides and PCBs are normally determined by method 8080 at STL North Canton, they will not be included in the initial demonstration of capability for method 8270B.

ATTACHMENT A

MODIFICATIONS REQUIRED FOR ANALYSIS OF WASTEWATER FOLLOWING METHOD 625

18. REQUIREMENTS FOR METHOD 625

18.1 Method 625 is required for demonstration of compliance with NPDES wastewater discharge permits. The standard analyte list and reporting limits are listed in Table A-1.

18.2 This method can be applied only to aqueous matrices.

18.3 The tune period for this method is defined as 24 hours.

18.4 Initial calibration curve requirements:

18.4.1 The initial calibration curve for this method requires at least three points.

18.4.2 Target compounds must have RSD ≤ 35%.

18.4.3 If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds.

18.5 Continuing calibration verification requirements: All target compounds must have %D ≤ 20%.

18.6 Matrix Spike and LCS requirements:

18.6.1 A full analyte spike is required for method 625. The spiking levels are given in Table A-2.

Table A-1. STL North Canton Method 625 standard reporting list and reporting limits.

Analytes	CAS Number	Aqueous µg/L
Phenol	108-95-2	10
Bis(2-chloroethyl)ether	111-44-4	10
2-Chlorophenol	95-57-8	10
1,3-Dichlorobenzene	541-73-1	10
1,4-Dichlorobenzene	106-46-7	10
1,2-Dichlorobenzene	95-50-1	10
2,2'-oxybis(1-chloropropane)	108-60-1	10
N-Nitroso-di-n-propylamine	621-64-7	10
Hexachloroethane	67-72-1	10
Nitrobenzene	98-95-3	10
Isophorone	78-59-1	10
2-Nitrophenol	88-75-5	10
2,4-Dimethylphenol	105-67-9	10
Bis(2-chloroethoxy)methane	111-91-1	10
2,4-Dichlorophenol	120-83-2	10
1,2,4-Trichlorobenzene	120-82-1	10
Naphthalene	91-20-3	10
Hexachlorobutadiene	87-68-3	10
4-Chloro-3-methylphenol	59-50-7	10
Hexachlorocyclopentadiene	77-47-4	50
2,4,6-Trichlorophenol	88-06-2	10
2-Chloronaphthalene	91-58-7	10
Dimethyl phthalate	131-11-3	10
Acenaphthylene	208-96-8	10
Acenaphthene	83-32-9	10
2,4-Dinitrophenol	51-28-5	50
4-Nitrophenol	100-02-7	50
2,4-Dinitrotoluene	121-14-2	10
2,6-Dinitrotoluene	606-20-2	10
Diethylphthalate	84-66-2	10
4-Chlorophenyl phenyl ether	7005-72-3	10
Fluorene	86-73-7	10
4,6-Dinitro-2-methylphenol	534-52-1	50
N-Nitrosodiphenylamine	86-30-6	10
4-Bromophenyl phenyl ether	101-55-3	10
Hexachlorobenzene	118-74-1	10
Pentachlorophenol	87-86-5	50
Phenanthrene	85-01-8	10
Anthracene	120-12-7	10
Di-n-butyl phthalate	84-74-2	10
Fluoranthene	206-44-0	10
Benzidine	92-87-5	100
Pyrene	129-00-0	10

Analytes	CAS Number	Aqueous µg/L
Butyl benzyl phthalate	85-68-7	10
3,3'-Dichlorobenzidine	91-94-1	50
Benzo(a)anthracene	56-55-3	10
Bis(2-ethylhexyl)phthalate	117-81-7	10
Chrysene	218-01-9	10
Di-n-octylphthalate	117-84-0	10
Benzo(b)fluoranthene	205-99-2	10
Benzo(k)fluoranthene	207-08-9	10
Benzo(a)pyrene	50-32-8	10
Indeno(1,2,3-cd)pyrene	193-39-5	10
Dibenz(a,h)anthracene	53-70-3	10
Benzo(g,h,i)perylene	191-24-2	10

Table A-2. Method 625 LCS and MS compounds and spike concentrations.

LCS Compounds	Spiking Level, ng/µL in extract[1]
Phenol	100
Bis(2-chloroethyl)ether	100
2-Chlorophenol	100
1,3-Dichlorobenzene	100
1,4-Dichlorobenzene	100
1,2-Dichlorobenzene	100
2,2'-oxybis(1-chloropropane)	100
N-Nitroso-di-n-propylamine	100
Hexachloroethane	100
Nitrobenzene	100
Isophorone	100
2-Nitrophenol	100
2,4-Dimethylphenol	100
Bis(2-chloroethoxy)methane	100
2,4-Dichlorophenol	100
1,2,4-Trichlorobenzene	100
Naphthalene	100
Hexachlorobutadiene	100
4-Chloro-3-methylphenol	100
Hexachlorocyclopentadiene	100
2,4,6-Trichlorophenol	100
2-Chloronaphthalene	100
Dimethyl phthalate	100
Acenaphthylene	100
Acenaphthene	100
2,4-Dinitrophenol	100
4-Nitrophenol	100
2,4-Dinitrotoluene	100
2,6-Dinitrotoluene	100
Diethylphthalate	100
4-Chlorophenyl phenyl ether	100
Fluorene	100
4,6-Dinitro-2-methylphenol	100
N-Nitrosodiphenylamine	100
4-Bromophenyl phenyl ether	100
Hexachlorobenzene	100
Pentachlorophenol	100
Phenanthrene	100
Anthracene	100
Di-n-butyl phthalate	100
Fluoranthene	100
Benzidine	100
Pyrene	100
Butyl benzyl phthalate	100
3,3'-Dichlorobenzidine	100

LCS Compounds	Spiking Level, ng/µL in extract[1]
Benzo(a)anthracene	100
Bis(2-ethylhexyl)phthalate	100
Chrysene	100
Di-n-octylphthalate	100
Benzo(b)fluoranthene	100
Benzo(k)fluoranthene	100
Benzo(a)pyrene	100
Indeno(1,2,3-cd)pyrene	100
Dibenz(a,h)anthracene	100
Benzo(g,h,i)perylene	100

[1] **Levels are 50 and 75 ng/µL if 2 µL injection is used**

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STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: GAS CHROMATOGRAPHIC ANALYSIS BASED ON METHOD 8000B, 8021B, 8081A, 8082, 8151A, 8310, 8141A, 8015B, 608, 610 and Wisconsin DNR Modified DRO Method

(SUPERSEDES: Revision 5.5 Dated 03/16/01)

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1. SCOPE AND APPLICATION

This SOP describes procedures for analysis of organic analytes by Gas Chromatography (GC). The procedures are based on SW-846 methodology and are applicable for measurements made to comply with the Resource Conservation and Recovery Act (RCRA). Individual analytes and methods are described in the appendices. Appendices B, C, and E include criteria for the analysis of wastewater by Methods 608 and 610. Appendix G describes procedures for the analysis of petroleum hydrocarbons by SW-846 methodology and the Wisconsin DNR Modified DRO method. Appendix H includes criteria for the analysis of non-halogenated organic compounds by Method 8015B, Direct Injection. Appendix I describes the analysis of Phillips 66 analytes by Method 8015B.

2. SUMMARY OF METHOD

In general, semivolatile analytes in aqueous samples are prepared for analysis using continuous or separatory funnel liquid / liquid extraction or solid phase extraction (SOP # CORP-OP-0001NCNC) Solid samples are prepared using sonication, Soxhlet or pressurized fluid extraction (SOP # CORP-OP-0001NC). Volatile analytes are prepared for analysis using purge and trap methodology (Appendix A).

After the initial preparation step, the sample is introduced to the GC and concentrations of target analytes are measured by the detector response within a defined retention time window, relative to the response to standard concentrations. Internal or external standardization procedures are used as specified in the method appendices.

3. DEFINITIONS

Definitions of terms used in this SOP may be found in the glossary of the STL North Canton Laboratory Quality Manual (LQM), current version.

4. INTERFERENCES

Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. In addition, some purge and trap autosamplers are susceptible to port specific contamination. Co-elution of target analytes with non-targets can occur, resulting in false positives or biased high results. In particular, this is a problem with non-selective detectors such as the Flame Ionization Detector (FID). See the appendices for interferences specific to individual tests and suggested corrective actions.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL North Canton associates. The following requirements must be met:

Eye protection that prevents spalsh, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have become contaminated will be removed and discarded; other gloves will be cleaned immediately. Refer to the STL North Canton Chemical Hygiene plan for a complete description of personal protection equipment.

The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. Specific hazards are covered in the appendices.

5.1.1. Opened containers of neat standards will be handled in a fume hood.

5.2. Sample extracts and standards, which are in a flammable solvent, shall be stored in an explosion-proof refrigerator.

5.3. When using hydrogen gas as a carrier, all precautions listed in the CHP shall be observed.

5.4. Standard preparation and dilution shall be performed inside an operating fume hood.

6. EQUIPMENT AND SUPPLIES

An analytical system complete with a gas chromatograph is required. A data system capable of measuring peak area and/or height is required. Recommended equipment and supplies for individual methods are listed in each method appendix.

7. REAGENTS AND STANDARDS

7.1. Stock Standards

Stock standards are purchased as certified solutions or prepared from pure solutions. Stock standards for method 8021B are stored at -10 to -20°C. Other stock standard solutions are stored as recommended by the manufacturer. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.

Semivolatile stock standard solutions must be replaced after one year. Stock standards of gases must be replaced at least every week, unless the acceptability of the standard is demonstrated (Less than 20% drift from the initial calibration is an acceptable demonstration). Other volatile stock standards must be replaced every 6 months or sooner if comparison with check standards prepared from an independent source indicates a problem.

7.1.1. Expiration times for all standards are measured from the time the standard is prepared or from the time that the standard ampoule is opened, if the standard is supplied in a sealed ampoule. If a vendor-supplied standard has an earlier expiration date then that date is used.

7.2. Calibration Standards

7.2.1. Volatile Calibration Standards

The procedure for preparation of volatile standards is given in Appendix A.

7.2.2. Semivolatile Calibration Standards

Semivolatile calibration standards are prepared as dilutions of the stock standards. Surrogates and internal standards are used as specified in the method appendices. Semivolatile calibration solutions must be refrigerated at $\leq$6°C and protected from light. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.

7.3. Gases for carrier and make-up: Hydrogen, Helium, Nitrogen, Argon/Methane.

7.4. Quality control (QC) Standards

QC standards (matrix spiking and LCS standards) are prepared and stored in the same way as calibration standards. They must be made from a stock independent from the calibration standards.

8. SAMPLE PRESERVATION AND STORAGE

Semivolatile extracts must be refrigerated at $\leq$6°C and analyzed within 40 days of the end of the extraction. Volatile sample storage conditions and holding times are given in Appendix A.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2. Batch Definition

Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the STL North Canton QC Program document (QA-003) for further details of the batch definition.

9.2.1. Quality Control Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. If insufficient sample is available for an MS/MSD a LCSD may be substituted.

9.3. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery +/- 3 standard deviations, unless that limit is tighter than the calibration criteria, in which case limits may be widened. Refer to policy QA-003 for more details.

9.3.1. These limits do not apply to dilutions (except for tests without a separate extraction), but surrogate and matrix spike recoveries will be reported unless the dilution is more than 5X.

9.3.2. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.3.3. Refer to the QC Program document (QA-003) for further details of control limits.

9.4. Surrogates

All methods must use surrogates to the extent possible. Surrogate recoveries in samples and QC samples must be assessed to ensure that recoveries are within established limits. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveal a problem.

- Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem. Repreparation is not necessary is there is obvious chromatographic interference.
- The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.4.1. If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in-control result is reported.

9.4.2. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then repreparation or flagging of the data is required.

9.4.3. Refer to the STL North Canton QC Program document (QA-003) for further details of the corrective actions.

9.5. Method Blanks

For each batch of samples, analyze a method blank. The method blank consists of reagent water for aqueous semivolatile samples, and sodium sulfate for semivolatile soils tests (Refer to SOP No. CORP-OP-0001NCNC for details). For low level volatiles, the method blank consists of reagent water. For medium level volatiles, the method blank consists of methanol as described in Appendix A. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone, phthalate esters) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

Re-extraction and reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.

If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be taken in consultation with the client.

9.5.1. Refer to the STL North Canton QC Program document (QA-003) for further details of the corrective actions.

9.6. Instrument Blanks

9.6.1. An instrument blank must be analyzed during any 12-hour period of analysis that does not contain a method blank.

9.6.2. An instrument blank consists of the appropriate solvent with internal standards added. If internal standards are not used the surrogates should be added.

9.6.3. Control criteria are the same as for the method blank, except that only reanalysis of affected samples would be required, not re-extraction.

9.7. Laboratory Control Samples (LCS)

For each batch of samples, analyze a LCS. The LCS contains a representative subset of the analytes of interest, and must contain the same analytes as the matrix spike. The LCS may also contain the full set of analytes with a subset of control analytes. If any control analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and reanalysis of the batch; however, if the matrix spike and matrix spike duplicate are within limits; the batch may be acceptable.

9.7.1. Refer to the STL North Canton QC Program document (QA-003) for further details of the corrective action.

9.7.2. If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in control result is reported.

9.7.3. LCS compound lists are included in the appendices.

9.8. Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in the appendices. Compare the percent recovery and relative percent difference (RPD) to those in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed.
- If the recovery for any component is outside QC limits for both the Matrix spike / spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include repreparation and reanalysis of the batch.
- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed.
- The matrix spike / duplicate must be analyzed at the same dilution as the unspiked sample, unless the matrix spike components would then be above the calibration range.

9.8.1. If dual column analysis is used the choice of which result to report is made in the same way as for samples (Section 12.1.2) unless one column is out of control, in which case the in control result is reported.

9.9. Quality Assurance Summaries

Certain clients may require specific project or program QC that may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.10. STL North Canton QC Program

Further details of QC and corrective action guidelines are presented in the STL QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION AND STANDARDIZATION

Internal or external calibration may be used. Internal calibration is recommended unless the sample matrix is likely to interfere with the quantitation of the internal standard. In either event prepare standards containing each analyte of interest at a minimum of five concentration levels. The low level standard should be at or

below the reporting limit. The other standards define the working range of the detector. Recommended calibration levels are given in the appendices.

10.1. A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include new columns, changing PID lamps or FID jets or replacing the ECD detector. A new calibration is not required after clipping the column, replacing the septum or syringe, or other minor maintenance.

10.2. With the exception of 10.3 below, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. In any event, at least 5 points must be included in the calibration curve. Quadratic (second order) calibrations require at least six points. Third order calibrations require at least seven points.

10.3. A level may be removed from the calibration if the reason can be clearly documented, for example a broken vial or no purge run. A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration. All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

10.4. External standard calibration

Quantitation by the external standard method assumes a proportional relationship between the calibration run and the analyte in the sample. To use this approach, introduce each calibration standard into the GC using the technique that will be used for samples. The ratio of the peak height or area response to the mass or concentration injected may be used to prepare a calibration curve.

$$Calibration\ Factor\,(CF) = \frac{Area\ or\ Height\ of\ Peak}{Mass\ Injected\,(ng)}$$

Some data systems may use the inverse of this formula. This is acceptable so long as the same formula is used for standards and samples. It is also possible to use the concentration of the standard rather than the mass injected. (This would require changes in the equations used to calculate the sample concentrations). Use of peak area or height must be consistent. However, if matrix interferences would make quantitation using peak area inaccurate for a particular sample, then peak height may be used as a substitute.

10.5. Internal standard calibration

10.5.1. The internal standard approach assumes that variations in instrument sensitivity, amount injected etc. can be corrected by determining the ratio of the response of the analyte to the response of an internal standard that has been added to the extract. To use this approach, select one or more internal standard(s) that are similar in analytical behavior to the compounds of interest. Recommended internal standards are given in the appendices. The analyst must demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. If the sample matrix interferes with quantitation of the internal standard, then the external standard approach must be used instead. In this event use the response factors from the previous continuing calibration to quantitate the analytes in the sample with the interference (applies only to the sample with the interference).

10.5.2. Introduce each calibration standard into the GC using the technique that will be used for samples. Response factors (RF) for each compound are calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

A_s = Response for the analyte to be measured
A_{is} = Response for the internal standard
C_{is} = Concentration of internal standard
C_s = Concentration of the analyte to be determined in the standard

10.6. Calibration curve fits

Average response factor, linear regression, or quadratic curves may be used to fit the data. Average response factor may be used if the average % RSD of the response factors or calibration factors of all the analytes in the calibration standard taken together is $\leq$ 20%. The average %RSD is calculated by summing the RSD value for each analyte and dividing by the total number of analytes.

10.6.1. In general, for environmental analysis, average response factors are the most appropriate calibration model. Linear or curved regression fits should only be used if the analyst has reason to believe that the average RF model does not fit the normal concentration/response behavior of the detector.

10.6.2. Average response factor

The average response factor may be used if the average percent relative standard deviation (%RSD) of all the response factors taken together is $\leq$ 20%.

The equation for average response factor is:

$$Average\ response\ factor = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where: n = Number of calibration levels

$\sum_{i=1}^{n} RF_i$ = Sum of response factors for each calibration level

10.6.3. Linear regression

The linear fit uses the following functions:

10.6.3.1. External Standard

$$y = ax + b$$

or

$$x = \frac{(y - b)}{a}$$

Where: y = Instrument response
x = Concentration
a = Slope
b = Intercept

10.6.3.2. Internal Standard

$$C_s = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{a}$$

Where: C_s = Concentration in the sample
A_s = Area of target peak in the sample
A_{is} = Area of internal standard in the sample
C_{is} = Concentration of the internal standard

10.6.4. Quadratic curve

The quadratic curve uses the following functions:

10.6.4.1. External standard

$$y = ax + cx^2 + b$$

Where c is the curvature

10.6.4.2. Internal Standard

$$y = a\left(\frac{A_s \times C_{is}}{A_{is}}\right) + c\left(\frac{A_s \times C_{is}}{A_{is}}\right)^2 + b$$

10.7. Evaluation of calibration curves

10.7.1. The percent relative standard error (%RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation.

10.7.2. The least squares regression line is calculated and used to calculate the predicted concentration for each level. The percent relative standard error is calculated as follows:

$$\%\ RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:
N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
C_i = True concentration for level i
PC_i = Predicted concentration for level i

Note that when average response factors are used, %RSE is equivalent to %RSD.

10.8. The following requirements must be met for any calibration to be used:

- Response must increase with increasing concentration.
- If a curve is used, the intercept of the curve at zero response must be less than $\pm$ the reporting limit for the analyte.
- The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be $\leq$ 20%.
- Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (r) and Coefficient of Determination (r^2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on r. As a result a curve may have a very good correlation coefficient (>0.995), while also having > 100% error at the low point.

10.9. Weighting of data points

10.9.1. In linear and quadratic calibration fits, the points at the lower end of the calibration curve have less absolute variance than points at the high concentration end of the curve. This can cause severe errors in quantitation at the low end of the calibration. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. 1/Concentration^2 weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.10. Non-standard analytes are sometimes requested. For these analytes, it may be acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. This action must be with client approval. If the analyte is detected in any of the samples, a five point initial calibration must be generated and the sample(s) reanalyzed for quantitation.

10.11. Calibration Verification

10.11.1. 12 hour Calibration

The working calibration curve or RF must be verified by the analysis of a mid point calibration standard at the beginning, after every 12 hours, and at the end of the analysis sequence. The center of each retention time window is updated with each 12-hour calibration or calibration verification.

10.11.2. Calibration Verification

It may be appropriate to analyze a mid point standard more frequently than every 12 hours. If these calibration verification standards are analyzed, requirements are the same as the 12-hour calibration with the exception that retention times are not updated.

10.11.3. Any individual compounds with %D < 15% meet the calibration criteria. The calibration verification is also acceptable if the average of the %D for all the analytes is < 15%. This average is calculated by summing the entire absolute %D results in the calibration (including surrogates) and dividing by the number of analytes. Any analyte that is reportable as found must have a % difference of < 15% in the calibration verification or 12 hour calibration, on the column used for quantitation. Refer to section 12.1.2 for which result to report.

10.11.4. It is not necessary to run a calibration verification standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.11.5. Samples quantitated by external standard methods must be bracketed by calibration verification standards that meet the criteria listed above. The bracketing standards on the column used for calibration must meet the same criteria as the opening standards. Bracketing is not necessary for internal standard methods.

10.11.6. If the analyst notes that a CCV has failed and can document the reason for failure (e.g. no purge, broken vial, carryover from the previous sample etc.) then a second CCV may be analyzed without any adjustments to the instrument. If this CCV meets criteria then the preceding samples have been successfully bracketed. If adjustments to the instrument are performed before the repeat CCV then the proceeding samples have not been successfully bracketed but analysis may continue.

10.11.7. In general, it is not advisable to analyze repeat CCVs on unattended runs. If repeat CCVs are analyzed then the first will serve as the bracketing standard for the preceding samples and the last will serve as the CCV for the following samples.

10.11.8. If highly contaminated samples are expected it is acceptable to analyze blanks or primers at any point in the run.

10.11.9. % Difference calculation

% Difference for internal and external methods is calculated as follows

Internal Standard:

$$\%D = \frac{RF_c - \overline{RF}}{\overline{RF}} \times 100$$

External standard:

$$\%D = \frac{CF_c - \overline{CF}}{\overline{CF}} \times 100$$

Where RF_c and CF_c are the response and calibration factors from the continuing calibration

$\overline{RF}$ and $\overline{CF}$ are the average response and calibration factors from the initial calibration

10.11.10. % Drift calculation

% Drift is used for comparing the continuing calibration to a linear or quadratic curve. The criteria for % drift are the same as for % difference

$$\%\ Drift = \frac{Calculated\ Conc.\ -\ Theoretical\ Conc.}{Theoretical\ Conc.} \times 100\%$$

10.11.11. Corrective Actions for Continuing Calibration

If the overall average %D of all analytes is greater than ± 15% corrective action must be taken. This may include clipping the column, changing the liner or other minor instrument adjustments, followed by reanalyzing the standard. If the overall average %D still varies by more than ± 15%, a new calibration curve must be prepared.

10.11.12. Corrective Action for Samples

For internal standard methods, any samples injected after a standard not meeting the calibration criteria must be re-injected.

For external standard methods, any samples injected after the last good continuing calibration standard must be re-injected.

If the average %D for all the analytes in the calibration is over 15%, but all of the analytes requested for a particular sample have %D ≤ 15%, then the analysis is acceptable for that sample.

11. PROCEDURE

11.1. Extraction

Extraction procedures are referenced in the appendices.

11.2. Cleanup

Cleanup procedures are referenced in the appendices.

11.3. Gas Chromatography

Chromatographic conditions for individual methods are presented in the appendices.

11.4. Sample Introduction

In general, volatiles analytes are introduced using purge and trap as described in Appendix A. Semivolatile analytes are introduced by direct injection of the extract. Samples, standards, and QC must be introduced using the same procedure.

11.5. Analytical Sequence

An analytical sequence starts with an initial calibration or a daily calibration. Refer to the individual method appendices for method specific details of daily calibrations and analytical sequences.

11.5.1. The daily calibration includes analysis of standards containing all single response analytes and updating the retention time windows.

11.5.2. If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a daily calibration.

11.6. Retention Time Windows

11.6.1. Retention time windows must be determined for all analytes. Make an injection of all analytes of interest each day over a three-day period. Calculate the standard deviation of the three retention times for each analyte (relative retention times may also be used). For multi-response analytes (e.g., Aroclors) use the retention time of major peaks. Plus or minus three times the standard deviation of the retention times of each analyte defines the retention time window.

11.6.2. The center of the retention time window is the retention time from the last of the three standards. The centers of the windows are updated with the mid- point of the initial calibration and each 12-hour calibration. The widths of the windows will remain the same until new windows are generated following the installation of a new column.

11.6.3. If the retention time window as calculated above is less than +/- 0.05 minutes, use +/- 0.05 minutes as the retention time window. This allows for slight variations in retention times caused by sample matrix.

11.6.4. The laboratory must calculate new retention time windows each time a new column is installed. The new windows must be generated within one week of the installation of the new column. Until these standards have been run on the new column, the retention time windows from the old column may be used, updated with the retention times from the new initial calibration.

11.6.5. Corrective Action for Retention Times

The retention times of all compounds in the 12 hour calibration or calibration verification standard must be within the retention time window. If this condition is not met, all samples analyzed after the last compliant standard must be reanalyzed unless the following conditions are met for any compound that elutes outside the retention time window:

The retention time of that compound in the standard must be within a retention time range equal to twice the original window.

No peak that would be reportable may be present on the sample chromatogram within an elution time range equal to three times the original retention time window.

11.7. Daily Retention Time Windows

The center of the retention time windows determined in Section 11.6 are adjusted to the retention time of each analyte as determined in the 12 hour calibration standards or continuing calibration verification standards. (See the method 8081A and 8082 appendices for exceptions for multi-response components.) The retention time windows must be updated at the beginning of each analytical sequence and with each 12-hour calibration or continuing calibration verification.

11.8. Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP CORP-OP-0001NC for determination of percent moisture.

11.9. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a supervisor and QA/QC manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file. The nonconformance is also addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative Identification

12.1.1. Tentative identification occurs when a peak is found within the retention time window for an analyte, at a concentration above the reporting limit, or above the MDL if J flags are required. Normally confirmation is required on a second column, but if the detector is sufficiently specific or if the sample matrix is well enough defined, single column analysis may be adequate. In some cases GC/MS confirmation may be required. Client specific requirements may also define the need for second column confirmation and / or GC/MS confirmation. Refer to the appendices for test specific requirements for confirmation. Identification is confirmed if a peak is also present in the retention time window for that analyte on the confirmatory column, at a concentration greater than the reporting limit (MDL if J flag confirmation required).

12.1.2. Dual column quantitation

For confirmed results, two approaches are available to the analyst.
A) The primary column approach
Or
B) The better result approach
Both are acceptable to avoid the reporting of erroneous or unconfirmed data.

12.1.2.1. Primary column approach:

The result from the primary column is normally reported. The result from the secondary column is reported if any of the following three bulleted possibilities are true.

- There is obvious chromatographic interference on the primary column
- The result on the primary column is > 40% greater than the result on the secondary column
- Continuing or bracketing standard fails on the primary column but is acceptable on the secondary column. (If the primary column result is > 40% higher than the secondary and the primary column calibration fails, then the sample must be evaluated for reanalysis.)

12.1.2.2. Better result approach

The lower of the two results is normally reported. The lower result is considered better because the higher result is generally higher because of chromatographic interference. The higher result is reported if any of the following two bulleted possibilities are true.

- There is obvious chromatographic interference on the column with the lower result
- The continuing or bracketing calibration on the column with the lower result fails. (If the higher result is > 40% higher and the calibration on the column with the lower result fails, then the sample must be evaluated for reanalysis.)

12.1.3. If the Relative percent difference (RPD) between the response on the two columns is greater than 40%, or if the opinion of an experienced analyst is that the complexity of the matrix is resulting in false positives, the confirmation is suspect and the results are qualified. RPD is calculated using the following formula:

$$RPD = \frac{R_1 - R_2}{\frac{1}{2}(R_1 + R_2)}$$

Where R=Result

12.1.4. Multi-response Analytes

For multi-response analytes, the analyst should use the retention time window, but should rely primarily on pattern recognition. The pattern of peaks will normally serve as confirmation.

12.1.5. The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times.

12.2. Calibration Range

If concentrations of any analytes exceed the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. It may be necessary to dilute samples due to matrix.

12.3. Dilutions

Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.3.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and only minor matrix peaks are, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgement is required to determine the most concentrated dilution that will not result in instrument contamination.

12.3.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions may be reported at client request, if the lower dilutions will not cause detector saturation, column overload, or carryover. Analyst judgement and client site history will factors in the reporting of dual dilutions.

12.4. Interferences

If peak detection is prevented by interferences, further cleanup should be attempted. If no further cleanup is reasonable, then elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.5. Internal Standard Criteria for Continuing Calibration

If internal standard calibration is used, then the internal standard response in a continuing calibration standard must be within 50 to 150% of the response in the mid level of the initial calibration.

12.6. Calculations

Capabilities of individual data systems may require the use of different formulas than those presented here. When this is the case, the calculations used must be shown to be equivalent and must be documented in an appendix attached to this document.

12.6.1. External Standard Calculations

12.6.1.1. Aqueous samples

$$Concentration\ (mg/L) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times V_s)}$$

Where:

A_x = Response for the analyte in the sample
V_i = Volume of extract injected, µL
D_f = Dilution factor
V_t = Volume of total extract, µL
V_s = Volume of sample extracted or purged, mL
CF = Calibration factor, area or height/ng, Section 10.1

12.6.1.2. Non-aqueous Samples

$$Concentration\ (mg/kg) = \frac{(A_x \times V_t \times D_f)}{(CF \times V_i \times W \times D)}$$

Where:

W = Weight of sample extracted or purged, g

$$D = \frac{100 - \%\,Moisture}{100}$$ (D = 1 if wet weight is required)

12.6.2. Internal Standard Calculations

12.6.2.1. Aqueous Samples

$$Concentration\ (mg/L) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times V_s)}$$

Where:

C_{is} = Amount of internal standard added, ng
A_{is} = Response of the internal standard
RF = Response factor for analyte

12.6.2.2. Non-aqueous Samples

$$Concentration\ (mg/kg) = \frac{(A_x \times C_{is} \times D_f)}{(A_{is} \times RF \times W \times D)}$$

12.6.3. Surrogate Recovery

Concentrations of surrogate compounds are calculated using the same equations as for the target compounds. The response factor from the initial calibration is used. Surrogate recovery is calculated using the following equation:

$$\%\ \text{Recovery} = \frac{Concentration\ (or\ amount)\ found}{Concentration\ (or\ amount)\ spiked} \times 100$$

13. METHOD PERFORMANCE

13.1. Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: QA-005.

13.2. Initial Demonstration

Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these results with the acceptance criteria given in each appendix.

13.2.3. If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3. Training Qualification

The group/team leader has the responsibility to ensure that an analyst who has been properly trained in its use and has the required experience performs this procedure.

14. POLLUTION PREVENTION

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, and Section 8000B

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the Method Detection Limit. This SOP states that the Method Blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed to be up to 5 times the reporting limit in the blank following consultation with the client.

17.2. Modifications from Previous Revision

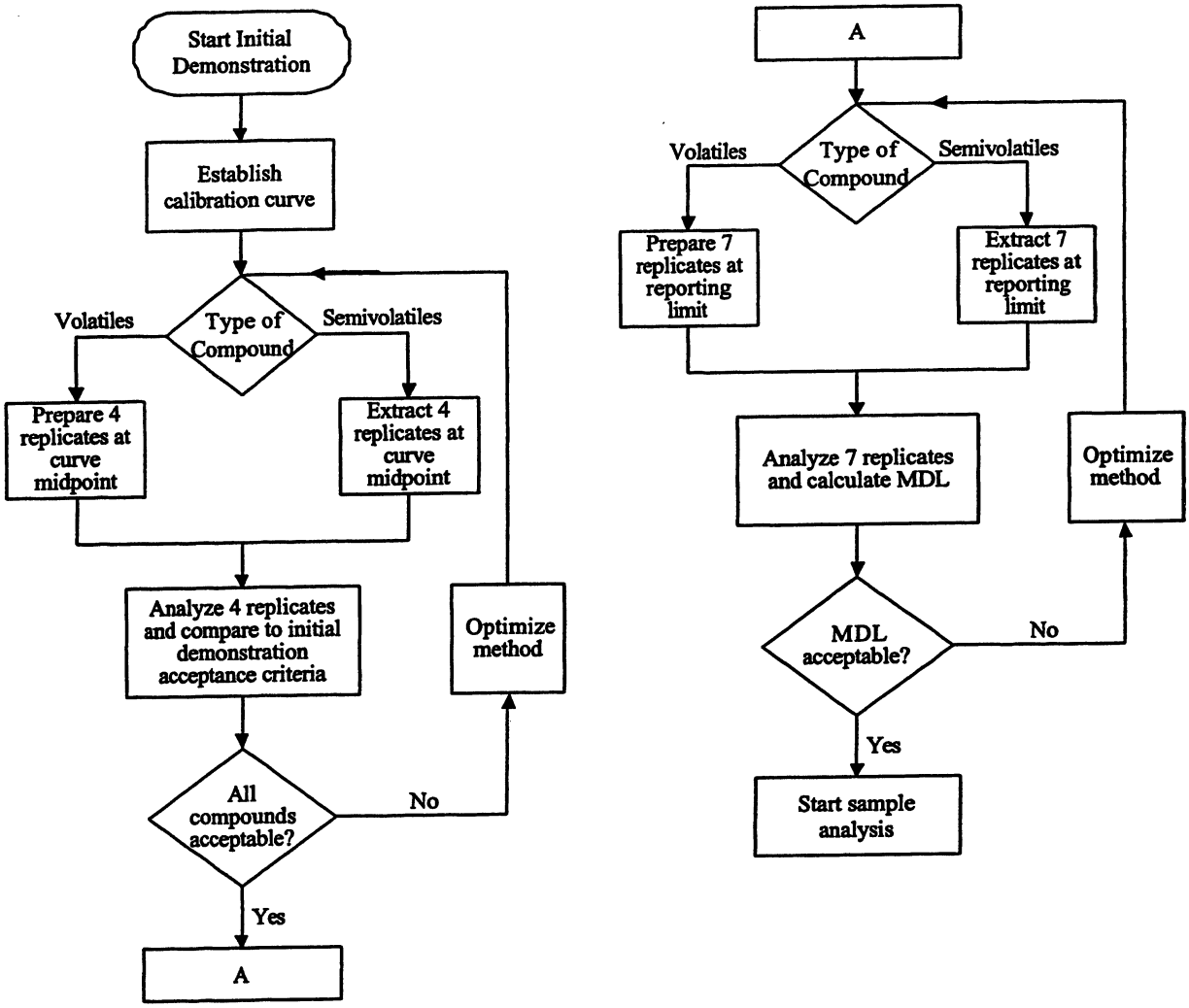
The calibration criteria in section 10.11 have been rewritten to improve consistency with SW-846 and to improve clarity.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

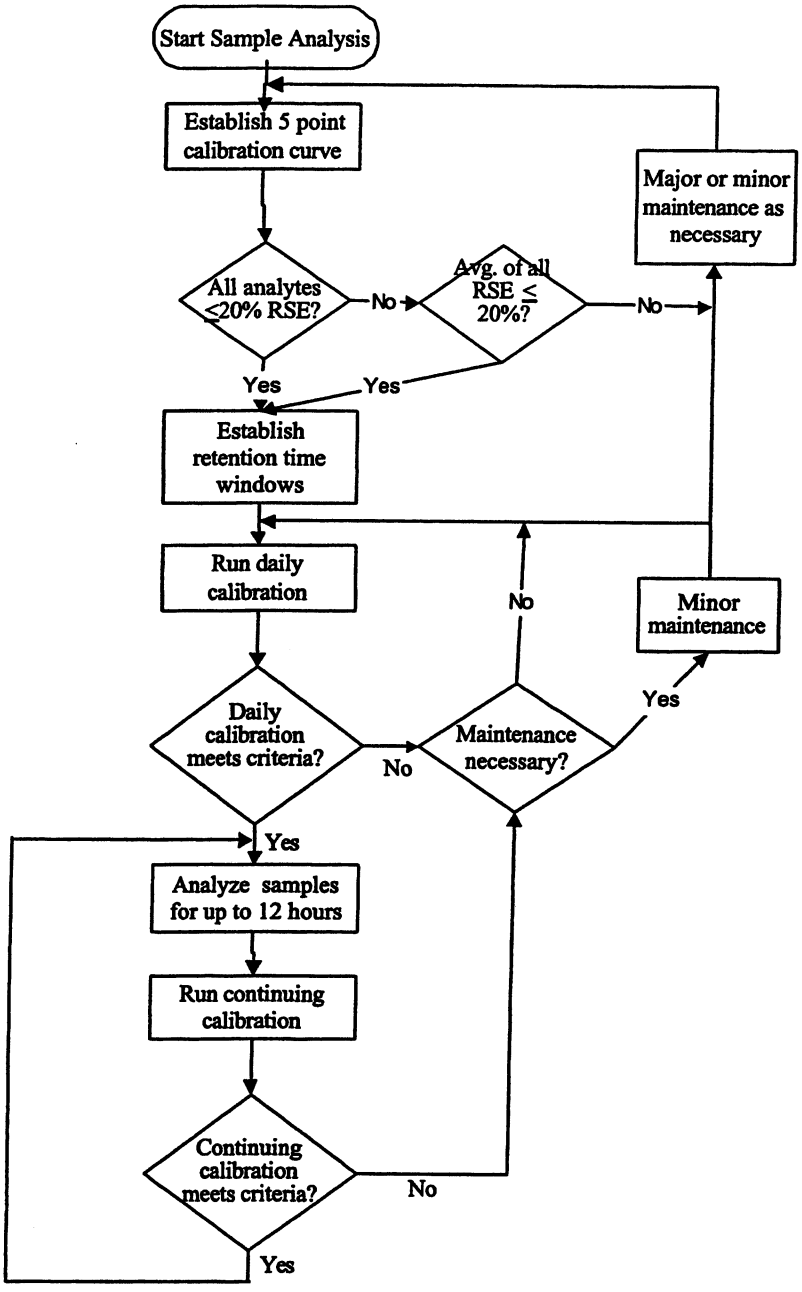
17.4. Flow Diagrams

17.4.1. Initial demonstration and MDL[1]



[1] This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.

17.4.2. Sample Analysis[1]



[1] This flow diagram is for guidance and cannot cover all eventualities. Consult the SOP text and a supervisor if in doubt.

1. SCOPE AND APPLICATION

1.1. This method describes sample preparation and extraction for the analysis of volatile organics by a purge and trap procedure, following method 8021B. However, where required by a client QAPP this section may also be used to analyze aromatic volatiles by discontinued methods 8020A and 8010B. All requirements of the 8000B section of this SOP must be met except when superseded by this Appendix. Refer to Table A-1 for the individual analytes normally determined by these procedures.

1.2. Compounds within the scope of this method have boiling points below 200°C and are soluble or slightly soluble in water. Classes of compounds best suited to purge-and-trap analysis include low molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.

1.3. Water samples and soils samples with low levels of contamination may be analyzed directly by purge-and-trap extraction and gas chromatography. Higher concentrations of these analytes in soil may be determined by the medium level methanol extraction procedure.

1.4. This method also describes the preparation of water-miscible liquids, non-water-miscible liquids, solids, wastes, and soils/sediments for analysis by the purge-and-trap procedure.

1.5. The associated LIMS method code is QR.

2. SUMMARY OF METHOD

2.1. An inert gas is bubbled through the sample at ambient temperature or at 40°C (40°C required for low-level soils), and the volatile components are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and back-flushed with inert gas to desorb the components onto a gas chromatographic column. Analytes are detected using a photoionization Detector, an electrolytic conductivity detector or a combination of both.

2.2. For soil samples, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water. It is then analyzed by purge-and-trap GC following the normal water method. If very low detection limits are needed for soil samples then direct purge using sodium bisulfate preservation may be necessary.

3. DEFINITIONS

Refer to the STL North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this SOP.

4. INTERFERENCES

4.1. Refer to section 4 of the method 8000B part of this SOP for general information on chromatographic interferences.

4.2. Impurities in the purge gas, and from organic compounds out-gassing from the plumbing ahead of the trap, account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

4.3. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank

prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination.

4.4. Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. Whenever an unusually concentrated sample is analyzed, it should be followed by an analysis of organic-free reagent water to check for cross-contamination. The trap and other parts of the system are subject to contamination. Therefore, frequent bake-out and purging of the system may be required.

4.5. When utilizing an autosampler system, which has multiple ports for sample analysis, it is likely that only a single stage or port may be contaminated by a highly concentrated sample. If a port is suspect, a water blank should be analyzed to verify lack of contamination. If the water blank and subsequent blanks on that port show contamination consistent with the concentrated sample, further maintenance is required. This may include replacing or cleaning the multi-port valve, transfer lines, etc.

4.6. A holding blank is kept in the sample refrigerator. This is analyzed and replaced every 14 days. If the holding blank does not meet the method blank criteria, the source of contamination must be found and corrected. Evaluation of all samples analyzed in the 14-day period prior to the analysis of the contaminated holding blank is required.

4.7. Acidification of samples may result in hydrolysis of 2-chloroethyl-vinyl ether.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B section of this SOP for general safety requirements.

5.2. Often, purge vessels on purge-and-trap instrumentation are pressurized by the time analysis is completed. Therefore, vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.3. The toxicity or carcinogenicity of each chemical used in this procedure has not been fully defined. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

Methanol -- Flammable and toxic

5.4. The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: Benzene, Carbon Tetrachloride, 1,4-Dichlorobenzene, 1,2-Dichloroethane, Hexachlorobutadiene, 1,1,2,2-Tetrachloroethane, 1,1,2-Trichloroethane, Chloroform, 1,2-Dibromoethane, Tetrachloroethene, Trichloroethene, Vinyl Chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood.

5.5. Methanol shall not be used in a CaptAir hood.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes -- 10µL, 25µL, 100µL, 250µL, 500µL, and 1000µL. These should be equipped with a 20 gauge (0.006" ID) needle. These will be used to measure and dispense methanolic solutions and aqueous samples.

6.2. Gas tight syringes -- 5 mL and 25 mL. Used for measuring sample volumes.

6.3. Purge and Trap Apparatus -- A device capable of extracting volatile compounds, trapping on a sorbent trap, and introducing onto a gas chromatograph.

6.4. Purge and Trap Autosampler -- In order to maintain high sample throughput, an autosampler is highly recommended.

6.5. Trap -- The trap used is dependent on the class of compound to be analyzed. Refer to Table A-2 for suggested traps for specific tests.

6.6. Purge Vessels -- These are dependent on the purge and trap unit/autosampler used. Both disposable culture tubes (needle sparge units) and specially designed vessels with fritted bottoms may be used. Follow the manufacturer's suggestions for configuration.

6.7. Columns - Refer to Table A-2 for details of columns.

6.8. Volumetric flasks, Class A: 5 mL to 250 mL

6.9. pH paper

6.10. Balance capable of weighing to 0.01g for samples.

7. REAGENTS AND SUPPLIES

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.

7.2. Organic Free Water

Organic free water is defined as water in which an interferent is not observed at the reporting limit of the compounds of interest. Suggested methods for generating organic free water include:

- Filtration through a carbon bed.
- Continuously sparging water with helium or nitrogen.
- Use of commercial water purification systems.

Other methods may be used, so long as the requirement that the water show no interference is met. The procedure used should be documented in a lab specific attachment.

7.3. Sodium Bisulfate

7.4. Methanol -- Purge and Trap Grade

7.5. Standards

Refer to tables A-5 and A-6 for details of surrogate, matrix spiking and internal standards. Calibration standard levels are not specified, since they may depend on the sensitivity and linear range of specific detectors. However, the low level standard must be equivalent to the reporting limits specified in Table A-1.

7.5.1. Volatile standards are prepared by injecting a measured volume of the stock standard into a syringe containing the appropriate volume of organic free water. The calibration standard is then loaded into the purge device.

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1. Holding times for all volatile analysis are 14 days from sample collection.

8.2. Water samples are normally preserved at pH < 2 with 1:1 hydrochloric acid. If residual chlorine is present, 2 drops of 10% sodium thiosulfate are added.

8.3. Solid samples are field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. At specific client request, unpreserved soil samples may be accepted.

8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take an EnCore sample. (The 5 g or 25 g sampler can be used, depending on client preference). Following shipment back to the lab the soil is preserved in methanol. This is the medium level procedure. If very low detection limits are needed (< 50 µg/kg for most analytes) then it will be necessary to use two additional 5 g EnCore samplers or to use field preservation.

8.5. Sample collection for medium level analysis using EnCore samplers.

8.5.1. Ship one 5 g (or 25 g) EnCore sampler per field sample position.

8.5.2. An additional bottle must be shipped for percent moisture determination.

8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25 g) sample into a tared VOA vial containing 5 mL methanol (25 mL methanol for the 25 g sampler). Obtain the weight of the soil added to the vial and note on the label.

8.5.4. Add the correct amount of surrogate spiking mixture. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.)

8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.) The addition of spike introduces a slight error, (0.4%) which can be neglected, into the calculations.

8.5.6. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (100 µL of spike to 25 mL methanol or 20 µL spike to 5 mL methanol).

8.5.7. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.5.8. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4+2°C until analysis.

8.6. Sample collection for medium level analysis using field methanol preservation

8.6.1. Prepare a VOA vial by adding 5 mL purge and trap grade methanol. (If a 25 g sample is to be used, add 25 mL methanol to the VOA vial).

8.6.2. Seal the bottle and attach a label.

8.6.3. Weigh the bottle to the nearest 0.01g and note the weight on the label.

8.6.4. Ship with appropriate sampling instructions.

8.6.5. Each sample will require an additional bottle with no preservative for percent moisture determination.

8.6.6. At client request, the methanol addition and weighing may also be performed in the field.

8.6.7. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.

8.6.8. Add the correct amount of surrogate spiking mixture. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.)

8.6.9. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 100 µL of 250 µg/mL solution for a nominal 25 g sample, 20µL for a nominal 5 g sample.) The addition of spike introduces a slight error, (0.4%) which can be neglected, into the calculations.

8.6.10. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (100 µL of spike to 25 mL methanol or 20 µL spike to 5 mL methanol).

8.6.11. Shake the samples for two minutes to distribute the methanol throughout the soil.

8.6.12. Allow to settle, then remove a portion of methanol and store in a clean Teflon capped vial at 4+2°C until analysis.

8.7. Low level procedure

8.7.1. If low detection limits are required (typically < 50 µg/kg) sodium bisulfate preservation must be used. However, it is also necessary to take a sample for the medium level (methanol preserved) procedure, in case the concentration of analytes in the soil is above the calibration range of the low-level procedure.

8.7.2. A purge and trap autosampler capable of sampling from a sealed vial is required for analysis of samples collected using this method. (Varian Archon or O.I. 4552).

8.7.3. The soil sample is taken using a 5g EnCore sampling device and returned to the lab. It is recommended that two EnCore samplers be used for each field sample position, to allow for any reruns than may be necessary. A separate sample for % moisture determination is also necessary.

8.7.4. Prepare VOA vials by adding a magnetic stir bar, approximately 1 g of sodium bisulfate and 5 mL of reagent water.

8.7.5. Seal the vial and attach a label. The label must not cover the neck of the vial or the autosampler will malfunction.

8.7.6. Weigh the vial to the nearest 0.01g and note the weight on the label.

8.7.7. Extrude the soil sample from the EnCore sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil and note on the label.

8.7.8. **Note:** Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case at a specific site, add 5 mL of water instead, and freeze at ≥ -10°C until analysis.

8.7.9. Alternatively the sodium bisulfate preservation may be performed in the field. Ship at least two vials per sample. The field samplers must determine the weight of soil sampled. Each sample will require an additional bottle with no preservative for percent moisture determination, and an additional bottle preserved with methanol for the medium level procedure.

8.8. Aqueous samples are stored in glass containers with Teflon lined septa at 4°C +/- 2°C, with minimum headspace.

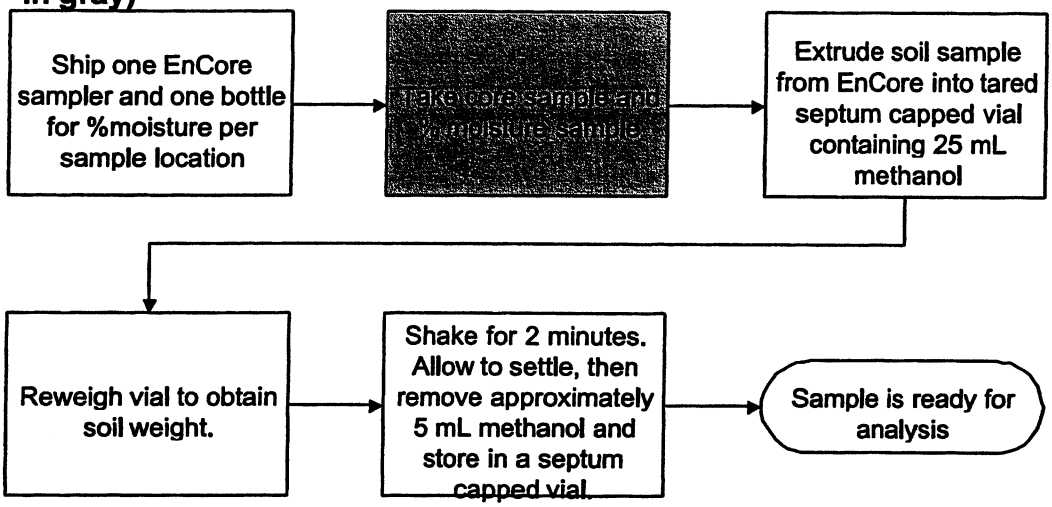
8.9. Medium level solid extracts are aliquoted into 2 - 5 mL glass vials with Teflon lined caps and stored at 4°C +/- 2°C. The extracts are stored with minimum headspace.

8.10. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14 day holding time. However they should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative). Maximum holding time for the EnCore sampler (before the sample is added to methanol or sodium bisulfate) is 48 hours.

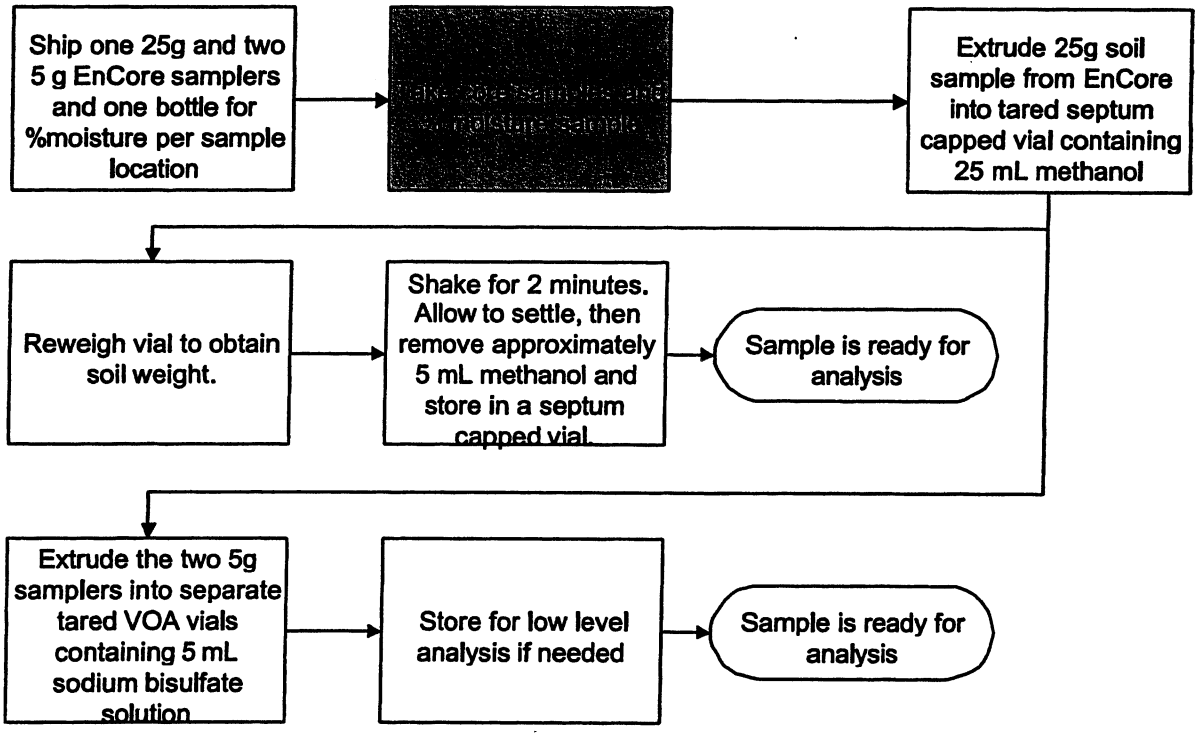
8.11. A holding blank is stored with the samples. This is analyzed and replaced if any of the trip blanks show any contamination. Otherwise it is replaced every 14 days.

Note: Freezing is not allowed for Ohio VAP solids.

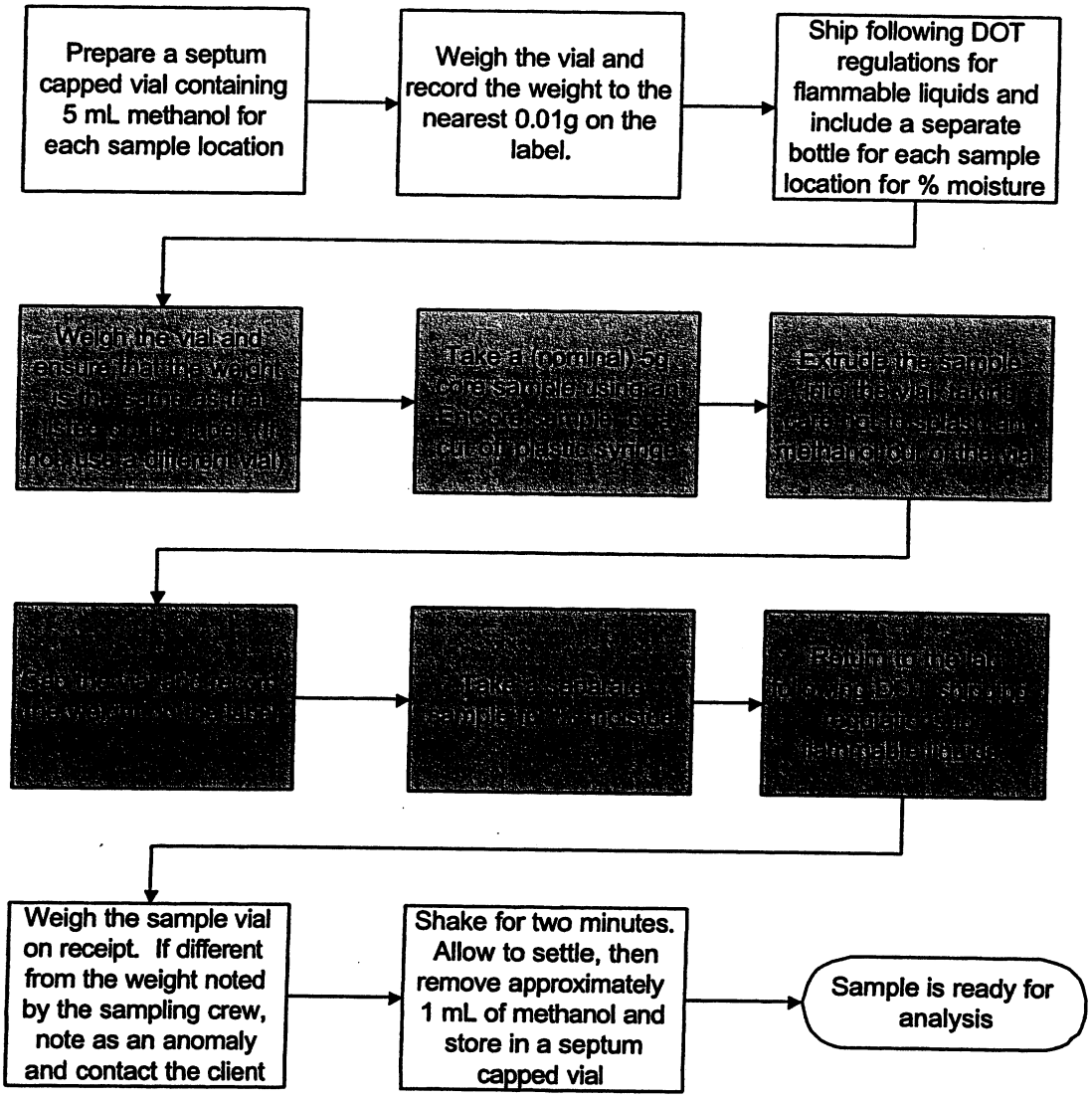
EnCore procedure when low level is not required (field steps in gray)



EnCore procedure when low level is required



Field methanol extraction procedure (field steps in gray)



9. QUALITY CONTROL

9.1. Refer to the method 8000B section of this SOP, section 9, for general quality control procedures, including batch definition, requirements for method blanks, LCS, matrix spikes, surrogates, and control limits.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to the method 8000B section of this SOP, section 10, for general calibration procedures.

10.2. Gas Chromatograph Operating Conditions

Various column configurations are possible. If dual column confirmation is necessary, the sample may be split using a Y splitter at the injector end to direct the sample to two columns and two detectors. For simultaneous determination of aromatic and halogenated volatiles, a single column is used and the PID and ELCD detectors are connected in series.

10.2.1. Refer to Table A-2, A-3 and A-4 for GC operating conditions.

10.3. Initial Calibration

10.3.1. Refer to Section 10 of the 8000B section of this SOP for details of initial calibration criteria.

10.3.2. Low level soil samples must be purged at 40°C; therefore the calibration curve must also be purged at 40°C. In addition, the low level soil calibration solutions should contain approximately the same amount of sodium bisulfate as the samples.

10.3.3. The low level calibration must be at the reporting limit or below. The remaining standards encompass the working range of the detector.

10.3.4. Calibrate the instrument using the same volume that will be used during sample analysis.

10.4. Calibration Verification

10.4.1. A mid level calibration standard is used for the calibration verification. The gases have 20 % D criteria rather than the 15% used for other analytes.

10.4.2. A calibration verification run is performed after every 10 samples for this method.

10.4.3. Bracketing of samples with calibration verification runs is only necessary for external standard analysis.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for general procedural requirements.

11.2. Analytical Sequence

The analytical sequence starts with an initial calibration of at least five points, or a 12 hour calibration that meets % difference criteria from an existing initial calibration.

11.3. Confirmation

The PID and ELCD detectors are sufficiently selective that second column confirmation is not always necessary. Requirements for second column confirmation should be decided in consultation with the

client. If the PID and ELCD are used in series confirmatory information for many analytes can be gained by comparing the relative response from the two detectors.

11.4. Aqueous Sample Analysis (Purge and Trap units using sparge vessels)

11.4.1. Depending on the sensitivity of the instrument and capabilities of the purge and trap device, 5, 10, 20, or 25 mL sample volumes may be analyzed. A 5 mL sample volume is recommended.

11.4.2. Rinse a 5 mL (or 25 mL for larger sample volumes) gas-tight syringe with organic free water. Fill the syringe with the sample to be analyzed, and compress to volume.

11.4.3. Check and document the pH of the sample remaining in the VOA vial after loading the syringe.

11.4.4. This procedure invalidates the contents of the VOA vial for further analysis, unless an aliquot is transferred to a smaller VOA vial with no headspace (e.g., 20 mL) at the same time the analysis aliquot is removed.

11.4.5. Spike with the appropriate volume of surrogate/internal standard solution and spike solution (if required) through the barrel of the syringe. The method blank is spiked with surrogates only, the LCS and matrix spikes with the surrogate and matrix spiking solutions. Refer to Tables A-5 and A-6 for volumes and concentrations of spiking solutions.

11.4.6. Load onto the purge and trap device and start the run.

11.4.7. If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. When a sample has a high response for a compound, an organic free water blank should follow analysis. It is recognized that during automated unattended analysis, this may not occur. If any potential carryover hits are present in samples following highly contaminated samples, the sample must be reanalyzed to determine if any of these hits are a result of carryover or are actually present in the sample.

11.4.8. Dilutions may be made in gas tight syringes unless the volume of sample used is less than 5 µL, in which case dilution in volumetric flasks will be necessary.

11.4.8.1. Spike with the same volume of surrogate/internal standard solution as used for undiluted samples prior to loading onto the purge and trap device.

11.4.8.2. For Matrix spike / matrix spike duplicates where the sample requires dilution, the sample is spiked after the dilution is performed.

11.5. Aqueous and Soil Sample Analysis (Purge and Trap units that sample directly from the VOA vial)

11.5.1. Units, which sample from the VOA vial, should be equipped with a module, which automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

11.5.2. If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise the internal and surrogate standards must be added to the vial. *Note:* Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to analyzed as soils.

11.5.3. Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

11.5.4. Check the pH of the sample remaining in the VOA vial after analysis is completed.

11.6. Low-Level Solids Analysis using discrete autosamplers

Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

This method is based on purging a heated sediment/soil sample mixed with reagent water containing the surrogate and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.

11.6.1. Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.

11.6.2. Weigh out 5 g (or other appropriate aliquot) of sample into a disposable culture tube or other purge vessel. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 1.0 g. If the sample is contaminated with analytes such that a purge amount less than 1.0 g is appropriate, use the medium level method described in section 11.7.

11.6.3. Connect the purge vessel to the purge and trap device.

11.6.4. Rinse a 5 mL gas-tight syringe with organic free water, and fill. Compress to 5 mL. Add surrogate/internal standard (and matrix spike solutions if required.) (See Tables A-5, A-6, A-7 and A-8.) Add directly to the sample from 11.6.2.

11.6.5. The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

11.6.6. Add the heater jacket or other heating device and start the purge and trap unit.

11.6.7. Soil samples that have low IS recovery when analyzed (<50%) should be reanalyzed once to confirm matrix effect. If external standard calibration is used, samples with surrogate recovery below the control limit should be reanalyzed once to confirm matrix effect.

11.7. Methanol Extract Soils

11.7.1. Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100 µL for a 5 mL purge) methanolic extract (from Section 8.5 or 8.6) to the syringe. Add internal standard (if used). Load the sample onto the purge and trap device and analyze as for aqueous samples. If less than 5µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5µL will be added to the water in the syringe.

12. DATA ANALYSIS AND CALCULATIONS

Refer to section 12 of the 8000B section of this SOP.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the 8000B section of this SOP.

14. POLLUTION PREVENTION

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Final Update III, December 1996, Sections 5000, 5030B, 5035 and 8021B

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.2. Modifications from previous revision

17.2.1. No revisions were made to this appendix.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

17.4. Tables

Table A-1

Standard Analyte List

Test	Compound	CAS number	Reporting Limit, µg/L or µg/kg		
			Aqueous	Low Soil	Medium Soil
Halogenated volatiles by 8021B	Bromodichloromethane	75-27-4	1.0	1.0	50
	Bromoform	75-25-2	1.0	1.0	50
	Bromomethane	74-83-9	1.0	1.0	50
	Carbon Tetrachloride	56-23-5	1.0	1.0	50
	Chlorobenzene	108-90-7	1.0	1.0	50
	Chloroethane	70-00-3	1.0	1.0	50
	2-Chloroethyl vinyl ether	110-75-8	5.0	5.0	250
	Chloroform	67-66-3	1.0	1.0	50
	Chloromethane	74-87-3	1.0	1.0	50
	Dibromochloromethane	124-48-1	1.0	1.0	50
	1,2-Dichlorobenzene	95-50-1	1.0	1.0	50
	1,3-Dichlorobenzene	541-73-1	1.0	1.0	50
	1,4-Dichlorobenzene	106-46-7	1.0	1.0	50
	Dichlorodifluoromethane	75-71-8	1.0	1.0	50
	1,1-Dichloroethane	75-34-3	1.0	1.0	50
	1,2-Dichloroethane	107-06-2	1.0	1.0	50
	1,1-Dichloroethene	75-45-4	1.0	1.0	50
	cis-1,2 Dichloroethene	156-59-4	1.0	1.0	50
	trans-1,2-Dichloroethene	156-60-5	1.0	1.0	50
	Dichloromethane(DCM)	75-09-2	5.0	5.0	250
	1,2-Dichloropropane	78-87-5	1.0	1.0	50
	cis-1,3-Dichloropropene	10061-01-5	1.0	1.0	50
	trans-1,3-Dichloropropene	10061-02-6	1.0	1.0	50
	1,1,2,2-Tetrachloroethane	79-34-5	1.0	1.0	50
	Tetrachloroethene	127-18-4	1.0	1.0	50
	1,1,1-Trichloroethane	71-55-6	1.0	1.0	50
	1,1,2-Trichloroethane	79-00-5	1.0	1.0	50
	Trichloroethene	79-01-6	1.0	1.0	50
	Trichlorofluoromethane	75-69-4	1.0	1.0	50
	Vinyl Chloride	75-01-4	1.0	1.0	50
Additional halogenated volatiles	Benzyl Chloride	100-44-7	5.0	5.0	250
	Bromobenzene	108-86-1	1.0	1.0	50
	Dibromomethane	74-95-3	1.0	1.0	50
	1,1,1,2-Tetrachloroethane	630-20-6	1.0	1.0	50
	Freon 113	76-13-1	1.0	1.0	50
	1,2,3-Trichloropropane	96-18-4	1.0	1.0	50
BTEX by 8021B	Benzene	71-43-2	1.0	1.0	50
	Ethyl Benzene	100-41-4	1.0	1.0	50
	Toluene	108-88-3	1.0	1.0	50
	Xylenes (total)	1330-20-7	1.0	1.0	50
Aromatic	Benzene	71-43-2	1.0	1.0	50

Table A-1

Standard Analyte List

Test	Compound	CAS number	Reporting Limit, µg/L or µg/kg		
			Aqueous	Low Soil	Medium Soil
volatiles by 8021B					
	Chlorobenzene	108-90-7	1.0	1.0	50
	1,2-Dichlorobenzene	75-34-3	1.0	1.0	50
	1,3-Dichlorobenzene	107-06-2	1.0	1.0	50
	1,4-Dichlorobenzene	75-45-4	1.0	1.0	50
	Ethyl Benzene	100-41-4	1.0	1.0	50
	Toluene	108-88-3	1.0	1.0	50
	Xylenes (total)	1330-20-7	1.0	1.0	50
Additional aromatic and unsaturated volatiles	1,2,4 Trimethylbenzene	95-63-6	1.0	1.0	50
	1,3,5 Trimethylbenzene	108-67-8	1.0	1.0	50
	Acetone	67-64-1	10	10	500
	MEK (2-butanone)	78-93-3	5.0	5.0	250
	MIBK (4-methyl-2-pentanone)	108-10-1	5.0	5.0	250
	Naphthalene	91-20-3	2.0	2.0	250
	Styrene	100-42-5	1.0	1.0	50
	Methyl tert-butyl ether (MTBE)	1634-04-4	1.0	1.0	50
Combined halogenated and aromatic volatiles by 8021B	Benzene	71-43-2	1.0	1.0	50
	Bromobenzene	108-86-1	1.0	1.0	50
	Bromochloromethane	74-97-5	1.0	1.0	50
	Bromodichloromethane	75-27-4	1.0	1.0	50
	Bromoform	75-25-2	1.0	1.0	50
	Bromomethane	74-83-9	1.0	1.0	50
	n-butylbenzene	104-51-8	1.0	1.0	50
	sec-Butylbenzene	135-98-8	1.0	1.0	50
	tert-Butylbenzene	98-06-6	1.0	1.0	50
	Carbon Tetrachloride	56-23-5	1.0	1.0	50
	Chlorobenzene	108-90-7	1.0	1.0	50
	Chlorodibromomethane	124-48-1	1.0	1.0	50
	Chloroethane	75-00-3	1.0	1.0	50
	Chloroform	67-66-3	1.0	1.0	50
	Chloromethane	74-87-3	1.0	1.0	50
	2-Chlorotoluene	95-49-8	1.0	1.0	50
	4-Chlorotoluene	106-43-4	1.0	1.0	50
	1,2-Dibromo-3-Chloropropane(DBCP)	96-12-8	1.0	1.0	50
	1,2-Dibromoethane(EDB)	106-93-4	1.0	1.0	50
	Dibromomethane	74-95-3	1.0	1.0	50
	1,2-Dichlorobenzene	95-50-1	1.0	1.0	50
	1,3-Dichlorobenzene	541-73-1	1.0	1.0	50
	1,4-Dichlorobenzene	106-46-7	1.0	1.0	50
	Dichlorodifluoromethane	75-71-8	1.0	1.0	50
	1,1-Dichloroethane	75-34-3	1.0	1.0	50
	1,2-Dichloroethane	107-06-2	1.0	1.0	50
	1,1-Dichloroethene	75-35-4	1.0	1.0	50

Table A-1
Standard Analyte List

Test	Compound	CAS number	Reporting Limit, µg/L or µg/kg		
			Aqueous	Low Soil	Medium Soil
	cis-1,2-Dichloroethene	156-59-4	1.0	1.0	50
	trans-1,2-Dichloroethene	156-60-5	1.0	1.0	50
	1,2-Dichloropropane	78-87-5	1.0	1.0	50
	1,3-Dichloropropane	142-28-9	1.0	1.0	50
	2,2-Dichloropropane	590-20-7	1.0	1.0	50
	1,1-Dichloropropene	563-58-6	1.0	1.0	50
	cis-1,3-Dichloropropene	10061-01-5	1.0	1.0	50
	trans-1,3-Dichloropropene	10061-02-6	1.0	1.0	50
	Ethylbenzene	100-41-4	1.0	1.0	50
	Hexachlorobutadiene	87-68-3	1.0	1.0	50
	Isopropylbenzene	98-82-8	1.0	1.0	50
	p-Isopropyltoluene	99-87-6	1.0	1.0	50
	Methylene Chloride	75-09-2	5.0	5.0	250
	Naphthalene	91-20-3	2.0	2.0	250
	n-Propylbenzene	10306501	1.0	1.0	50
	Styrene	100-42-5	1.0	1.0	50
	1,1,1,2-Tetrachloroethane	630-20-6	1.0	1.0	50
	1,1,2,2-Tetrachloroethane	79-34-5	1.0	1.0	50
	Tetrachloroethene	127-18-4	1.0	1.0	50
	Toluene	108-88-3	1.0	1.0	50
	1,2,3-Trichlorobenzene	87-61-6	1.0	1.0	50
	1,2,4-Trichlorobenzene	120-82-1	1.0	1.0	50
	1,1,1-Trichloroethane	71-55-6	1.0	1.0	50
	1,1,2-Trichloroethane	79-00-5	1.0	1.0	50
	Trichloroethene	79-01-6	1.0	1.0	50
	Trichlorofluoromethane	75-69-4	1.0	1.0	50
	1,2,3-Trichloropropane	96-18-4	1.0	1.0	50
	1,2,4-Trimethylbenzene	95-63-6	1.0	1.0	50
	1,3,5-Trimethylbenzene	108-67-8	1.0	1.0	50
	Vinyl Chloride	75-01-4	1.0	1.0	50
	Xylenes (total)	1330-20-7	1.0	1.0	50

Table A-2
Recommended Conditions for Aromatic Volatiles

Parameter	Recommended Conditions
Temperature program	50°C, 1min, 10°C/min to 200°C,1min
Column 1	Rtx-502.2 or DB-502.2 60m x 0.53mm 3.0um
Column 2	Rtx-1 or DB-1 60m x 0.53mm 3.0 um
Carrier gas	Helium or hydrogen
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-3 Recommended Conditions for Method Halogenated Volatiles	
Parameter	Recommended Conditions
Temperature program	35°C, 12 min, then 4°C/min to 200°C, hold for 5 min
Column 1	DB-VRX or RTX-502.2 105m x 0.53 mm id df = 3.0um
Column 2	DB-1 or RTX-1 105m x 0.53 mm ID df = 3.0um
Column 3	Rtx - Volatiles 120m x 0.53mm ID df=2.0um
Carrier gas	Helium
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-4 Recommended Conditions for Method Combined Aromatic and Halogenated Volatiles	
Parameter	Recommended Conditions
Temperature program	35°C, 12 min, then 4°C/min to 200°C, hold for 5 min
Column 1	DB-VRX or RTX-502.2 105m x 0.53 mm id df = 3.0um
Column 2	DB-1 or RTX-1 105m x 0.53 mm ID df = 3.0um
Column 3	Rtx - Volatiles 120m x 0.53mm ID df=2.0um
Carrier gas	Helium
Purge Flow / time	40 mL/min, 11 minutes
Desorb Temp / time	180°C, 2 minutes (220°C for Vocarb 3000)
Bake Time / temp	200°C, 12 minutes (230°C for Vocarb 3000)
Transfer line / valve temp	115°C

Table A-5
Surrogate and Internal Standard Concentrations

Standard	Components	Working Solution µg/mL	Spike amount µL (for 5 mL purge)	Final concentration µg/L (µg/kg)
Aromatic volatiles IS/SS	4-Chlorotoluene (SS) 1-Chloro-4-fluorobenzene (IS)	20 40	5	20 40
Halogenated volatiles IS/SS	4-chlorotoluene (SS) 1-Chloro-4-fluorobenzene (IS)	20 40	5	20 40
Combined Aromatic and halogenated volatiles IS/SS	Fluorobenzene (SS)	20	5	20
	1,4-Dichlorobutane (SS)	20	5	20
	1-Chloro-4-fluorobenzene (IS)	40	10	40

It may be necessary to select different surrogates in order to minimize sample interferences. 1-chloro-4-fluorobenzene and 4-chlorotoluene are fairly well resolved from analytes listed in this SOP. However 4-chlorotoluene may sometimes be requested as a target analyte. Other surrogates that may be considered, and issues associated with their use are:

Bromochloromethane: Elutes very close to chloroform and cis-1, 2-dichloroethene on the 502.2 column. May be a target analyte.

1,2-Bromochloroethane:

1-Chloro-2-fluorobenzene: Elutes close to ethylbenzene on DB-1 or Rtx-1 and close to m,p-xylene on 502.2

a,a,a-Trifluorotoluene: Good for aromatic volatiles, coelutes or very close to trichloroethene

Bromofluorobenzene: Close to 1,1,2,2-trichloroethane and 1,2,3-trichloropropane on the 502.2 column. Good on DB-1 or Rtx-1.

2-Bromo-1-chloropropane: May coelute with 1,1,2-trichloroethane

Table A-6
Concentrations for LCS and MS/MSD compounds

Standard	Components	Working Solution µg/mL	Spike amount µL (5 mL purge)	Final concentration µg/L (µg/kg)
Aromatic	Benzene	20	5	20
	Toluene	20		20
	Chlorobenzene	20		20
Halogenated	Chlorobenzene	20	5	20
	1,1-Dichloroethene	20		20
	Trichloroethene	20		20
Combination aromatic / halogenated	Benzene	20	5	20
	Toluene	20		20
	Chlorobenzene	20		20
	1,1-Dichloroethene	20		20
	Trichloroethene	20		20

1. SCOPE AND APPLICATION

This SOP Appendix describes procedures to be used when SW-846 Method 8081A is applied to the analysis of organochlorine pesticides by GC/ECD. This Appendix may also to be applied when discontinued SW-846 Method 8080A is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate STL North Canton sample extraction SOPs. (CORP-OP-0001NC) Criteria for the analysis of wastewater by Method 608 is also included in this appendix.

Table B-1 lists compounds, which are routinely determined by this method, and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

At client request, this method may also be used for the analysis of PCBs (Arochlors) in combination with pesticides, although these are normally analyzed following method 8082, as described in Appendix C of this SOP. In any event, if samples for PCB analysis do not need the acid clean up procedure, then the same injection may be used for method 8081B and 8082, assuming all calibration and QC requirements for both methods are met. Extracts that have been acid cleaned may not be analyzed for pesticides, since several of the pesticides will be degraded.

1.1. The associated LIMS method codes are QJ (8081A) and DM (608).

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of organochlorine pesticides. The pesticides are injected onto the column and separated and detected by electron capture detection. Quantitation may be by internal or external standard methods.

3. DEFINITIONS

Refer to the STL North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Avoiding contact with any plastic materials minimizes interferences from phthalates.

4.3. Sulfur will interfere and can be removed using procedures described in SOP CORP-OP-0001NC.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), and Sulfur cleanup (Method 3660). These cleanup procedures are included in SOP # CORP-OP-0001NC. Using hexane / acetone as the extraction solvent (rather than hexane / methylene chloride) will reduce the amount of interferences extracted.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

5.2. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

5.3. The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, and the BHCs. Primary standards of these toxic compounds should be prepared in a hood.

5.4. All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

5.5. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the Director, EH&S shall be immediately notified and a letter sent to the NRC or local state agency.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. A ^{63}Ni electron capture detector is required.

6.2. Refer to Table B-2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies.

7.2. Refer to Table B-3 for details of calibration standards.

7.3. Surrogate Standards

Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Refer to tables B-5 and B-6 for details of surrogate standards.

7.4. Column Degradation Evaluation Mix

A mid-level standard containing 4,4'-DDT and Endrin and not containing any of their breakdown products must be prepared for evaluation of degradation of these compounds by the GC column and injection port. This mix must be replaced after one year, or whenever corrective action to columns fails to eliminate the breakdown of the compounds, whichever is shorter. This solution also contains the surrogates. Refer to Table B-4 for details of the column degradation evaluation mix.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

Refer to Section 9 of the 8000B section of this SOP.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. Refer to Table B-2 for details of GC operating conditions. The conditions listed should result in resolution of all analytes listed in Table B-1 on both columns. Closely eluting pairs are DDE and Dieldrin on the Rtx-5 or DB-5 column and Endosulfan II and DDD on the 1701 column.

Note: Method 608 requires a minimum of three concentration levels for initial calibrations. The relative standard deviation, RSD must be less than 10%.

10.3. Column Degradation Evaluation

Before any calibration runs, either initial or 12 hour, The column evaluation mix must be injected before each initial or daily calibration. The degradation of DDT and endrin must be calculated (see equations 9 and 10) and each shown to be less than 15% before calibration can proceed. This is only necessary if the target compound list includes DDT, Endrin, or any of their degradation products.

If the breakdown of DDT and/or endrin exceeds the limits given above, corrective action must be taken. This action may include:

- Replacement of the injection port liner or the glass wool.
- Cutting off a portion of the injection end of a capillary column.
- Replacing the GC column.

10.4. Initial Calibration

Refer to Section 10 of the 8000B section of this SOP for details of calibration procedures.

10.4.1. Refer to Table B-7 for the initial calibration analytical sequence.

10.4.2. The response for each single-peak analyte will be calculated by the procedures described in the general method for GC analysis.

10.4.3. The surrogate calibration curve is calculated from the Individual AB mix. Surrogates in the other calibration standards are used only as retention time markers. If there are resolution problems, then the A and B mixes may be analyzed separately.

10.4.4. For multi-component pesticides:

Single point calibration is used for multi-component pesticides (typically toxaphene and technical chlordane). Two options are possible; the same quantitation option must be used for standards and samples. Refer to section 12.3 for guidance on which option to use.

10.4.5. For multi-component analytes, the mid level standard must be analyzed as part of the initial calibration. This single point calibration is used to quantitate multi-component analytes.

10.4.6. The analyst may include a full 5 point calibration for any of the multi-component analytes with the initial calibration.

10.5. 12 hour Calibration Verification

The 12 hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration. A mid level calibration standard is used for the 12 hour calibration. Refer to the 8000B section of this SOP for acceptance criteria.

Note : Method 608 requires that the working calibration curve must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ± 15%, the test must be repeated using a fresh calibration standard.

10.5.1. At a minimum, the 12 hour calibration includes analysis of the breakdown mix followed by mid level standards of any single and multi-component analytes.

10.5.2. The retention time windows for any analytes included in the 12 hour calibration are updated.

10.6. Continuing Calibration

The AB calibration mix is analyzed as the continuing calibration standard. At a minimum, this is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. If 12 hours elapse analyze the 12 hour standard sequence instead. The continuing calibration standard need not include multi-component analytes. If instrument drift is expected due to sample matrix or other factors, it may be advisable to analyze the continuing calibration standard more frequently.

10.6.1. A mid level calibration standard is used for the continuing calibration.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for general procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP No. CORP-OP-0001NC.

11.3. Cleanup

Cleanup procedures are described in SOP No. CORP-OP-0001NC.

11.4. Suggested gas chromatographic conditions are given in Table B-2.

11.5. Allow extracts to warm to ambient temperature before injection.

11.6. The suggested analytical sequence is given in Table B-7.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. Identification of Multi-component Analytes

Retention time windows are also used for identification of multi-component analytes, but the "fingerprint" produced by major peaks of those compounds in the standard is used in tandem with the retention times to identify the compounds. The ratios of the areas of the major peaks are also taken into consideration. Identification of these compounds may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.3. Quantitation of Multi-component Analytes

Use 3-10 major peaks or total area for quantitation as described in section 10.4.4, initial calibration of multi-component analytes.

12.3.1. If there are no interfering peaks within the envelope of the multi-component analyte, the total area of the standards and samples may be used for quantitation. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area.

12.3.1.1. Multiple peak option

This option is particularly valuable if toxaphene is identified but interferences make quantitation based on total area difficult. Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks. Alternatively, find the response of each of the 3-10 peaks per multi-peak pesticide, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be coeluting with contaminant peaks from the quantitation. (i.e. peaks which are significantly larger than would be expected from the rest of the pattern.)

Chlordane may be quantitated either using the multiple peak option (12.3.1.1) total area option (12.3.1.2.) or by quantitation of the major components, α-chlordane, γ-chlordane and heptachlor.

12.3.1.2. Total area option

The total area of the standards and samples may be used for quantitation of multi-component analytes. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area. This option should not be used if there are significant interference peaks within the multi-component pattern in the samples. The retention time window for total area measurement must contain at least 90% of the area of the analyte.

12.4. Second column confirmation multi-component analytes will only be performed when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence.

12.5. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX). Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

12.6. Calculation of Column Degradation/% Breakdown (%B)

Equation 9

$$DDT\,\%B = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100$$

where:
A_{DDD}, A_{DDE}, and A_{DDT} = the response of the peaks for 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT in the column degradation evaluation mix.

Equation 10

$$Endrin\,\%B = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_{E}} \times 100$$

where:
A_{EK}, A_{EA}, and A_E = the response of endrin ketone, endrin aldehyde, and endrin in the column degradation evaluation mix.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required under Section 13.1 of the main body of this SOP. Example performance limits are listed in Table B-8. The spiking level should be equivalent to a mid level calibration.

14. POLLUTION PREVENTION

Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, Update III, December 1996, Method 8081A

16.2. CFR136A, Method 608 – Organochlorine Pesticides and PCBs

17. MISCELLANEOUS

17.1. Modifications from Reference Method

None

17.2. Modifications from Previous Revisions

17.2.1. No revisions were made to this appendix.

17.3. Tables

Table B-1
Standard Analyte list and Reporting Limits

Compound	Reporting Limit, µg/L or µg/kg		
	water	soil	waste
Aldrin	0.05	1.7	50
α-BHC	0.05	1.7	50
β-BHC	0.05	1.7	50
δ-BHC	0.05	1.7	50
γ-BHC (Lindane)	0.05	1.7	50
α-Chlordane	0.05	1.7	50
γ-Chlordane	0.05	1.7	50
Chlordane (technical)	0.5	17	500
4,4'-DDD	0.05	1.7	50
4,4'-DDE	0.05	1.7	50
4,4'-DDT	0.05	1.7	50
Dieldrin	0.05	1.7	50
Endosulfan I	0.05	1.7	50
Endosulfan II	0.05	1.7	50
Endosulfan Sulfate	0.05	1.7	50
Endrin	0.05	1.7	50
Endrin Aldehyde	0.05	1.7	50
Heptachlor	0.05	1.7	50
Heptachlor Epoxide	0.05	1.7	50
Methoxychlor	0.1	3.3	100
Toxaphene	2.0	67	2000
APPENDIX IX ADD ONs			
Diallate	1.0	33	1000
Isodrin	0.1	3.3	100
Chlorobenzilate	0.1	3.3	100
Kepone[1]	1.0	33	1000

[1] Kepone is sometimes requested for analysis by method 8081A. However kepone may produce peaks with broad tails that elute later than the standard by up to a minute (presumably due to hemi-acetal formation). As a result kepone analysis by 8081A is unreliable and not recommended. Analysis by method 8270C is a possible alternative. Note: alpha chlordane, gamma chlordane, and endrin ketone are not required for some projects.

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.		Final Vol.
Ground water	1000 mL	10 mL	
Low-level Soil	30 g		10 mL
High-level soil / waste	1 g		10 mL

Table B-2	
Parameter	**Recommended Conditions**
Injection port temp	220°C
Detector temp	325°C
Temperature program	120°C for 1 min, 8.5°C/min to 285°C, , 6 min hold
Column 1	Rtx-CLPesticides 30m x 0.32mm id, 0.5µm
Column 2	Rtx-35 30m x 0.32 mm id, 0.5µm
Column 3	DB-608, 30m X 0.32 mm, 0.25µm
Injection	2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table B-3						
Calibration Levels ng/mL						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[2]
Individual Mix AB[1]						
Aldrin	5	10	25	50	100	200
g-BHC (Lindane)	5	10	25	50	100	200
Heptachlor	5	10	25	50	100	200
Methoxychlor	10	20	50	100	200	400
Dieldrin	5	10	25	50	100	200
Endosulfan I	5	10	25	50	100	200
Endosulfan II	5	10	25	50	100	200
4,4'-DDT	5	10	25	50	100	200
Endrin Aldehyde	5	10	25	50	100	200
Endrin Ketone	5	10	25	50	100	200
β-BHC	5	10	25	50	100	200
δ-BHC	5	10	25	50	100	200
α-BHC	5	10	25	50	100	200
4,4'-DDD	5	10	25	50	100	200
4,4'-DDE	5	10	25	50	100	200
Endosulfan Sulfate	5	10	25	50	100	200
Endrin	5	10	25	50	100	200
α-Chlordane[3]	5	10	25	50	100	200
γ-Chlordane[3]	5	10	25	50	100	200
Multi-component Standards						
Chlordane (Technical)			250[4]			
Toxaphene			1000[5]			
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl	5	10	25	50	100	200

[1] Standards may be split into an A and B mix if resolution of all compounds on both columns is not obtained.

[2] Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.

[3] Compounds may be used in lieu of running a daily technical Chlordane standard for samples that are non-detect for technical Chlordane.

[4] This standard may be used for quantitation of technical chlordane between 50 and 1000 ng/mL. If the chlordane is more concentrated, the extract must be diluted and reanalyzed.

[5] This standard may be used for quantitation of toxaphene between 200 and 4000 ng/mL. If the toxaphene is more concentrated, the extract must be diluted and reanalyzed.

Table B-4	
Column Degradation Evaluation Mix ng/mL	
Component	Concentration
4,4'-DDT	25
Endrin	25
Tetrachloro-m-xylene (Surrogate)	20
Decachlorobiphenyl (Surrogate)	20

Table B-5			
LCS/Matrix Spike and Surrogate Spike levels µg/L or µg/kg			
	Aqueous	Soil	Waste
gamma BHC (Lindane)	0.20	33.3	200
Aldrin	0.20	33.3	200
Heptachlor	0.20	33.3	200
Dieldrin	0.50	33.3	500
Endrin	0.50	33.3	500
4,4'DDT	0.50	33.3	500
Tetrachloro-m-xylene (Surrogate)	0.20	33.3	200
Decachlorobiphenyl (Surrogate)	0.20	33.3	200

Table B-6		
LCS/Matrix Spike and Surrogate Spike levels for TCLP µg/L or µg/kg		
	Aqueous	Waste
Heptachlor	5	500
Heptachlor epoxide	5	500
Lindane	5	500
Endrin	5	500
Methoxychlor	10	1000

Table B-7
Suggested Analytical Sequence

Initial Calibration

Solvent blank (optional)	
Breakdown Mix	
Individual mix AB	All levels
Technical Chlordane	Level 3[1]
Toxaphene	Level 3[1]
Up to 20 samples unless 12 hours comes first)	
Solvent blank (optional)	
Individual mix AB	Mid level (Continuing calibration)
Samples	
After 12 hours:	
Breakdown mix	
Individual mix AB	
Any other single component analytes	

Any multi-component analytes

[1] A five point curve for any of the multi-component analytes may be included
If Arochlors are included, a 5 point calibration for Arochlor 1016/1260 should be included with the initial calibration and a single point for the other Arochlors. The mid point 1016/1260 mix is included with the daily calibration (every 12 hours).

Note: A solvent blank or primer may be analyzed at any time during the sequence when highly contaminated samples are expected. A solvent blank or primer may not be analyzed as routine immediately prior to standards.

12 hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Individual mix AB, and the breakdown mix must be run before the continuing calibration.

Table B-8		
Example Performance limits, four replicate initial demonstration of capability		
Compound	Initial demonstration, mean recovery limits	Initial demonstration, RSD limits
Aldrin	46-112	21
alpha-BHC	51-122	24
beta-BHC	61-120	32
delta-BHC	49.5-118.5	36
gamma-BHC	57-116	23
Chlordane	44.8-108.6	20
4,4'-DDD	52-126	28
4,4'-DDE	46-120	27.5
4,4'-DDT	54-137	36
Dieldrin	42.5-124.5	38
Endosulfan I	43-141	24.5
Endosulfan II	78-171	61
Endosulfan Sulfate	62-132	27
Endrin	49-126	37
Heptachlor	57-100	20
Heptachlor Epoxide	43.5-131.5	25.4
Toxaphene	44.4-111.2	20

1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8000B is applied to the analysis of polychlorinated biphenyls (PCB) by GC/ECD. This Appendix is to be applied when SW-846 Method 8082 is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate STL sample extraction SOPs. (CORP-OP-0001NC). The PCBs are determined and quantitated as Arochlor mixes. Criteria for the analysis of wastewater by Method 608 is also included in this appendix.

Table C-1 lists compounds, which are routinely determined by this method, and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed.

Note: SW-846 method 8082 provides incomplete guidance for determination of individual PCB congeners. This SOP does not include directions for congener specific analysis.

1.2. The associated LIMS method codes are QH (8082) and DM (608).

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of PCBs. The PCBs are injected onto the column and separated and detected by electron capture detection. Quantitation is by the external standard method.

3. DEFINITIONS

Refer to the STL North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for information regarding chromatographic interferences.

4.2. Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Avoiding contact with any plastic materials minimizes interferences from phthalates.

4.3. Sulfur will interfere and can be removed using procedures described in SOP CORP-OP-0001NC.

4.4. Interferences co-extracted from samples will vary considerably from source to source. The presence of interferences may raise quantitation limits for individual samples. Specific cleanups may be performed on the sample extracts, including florisil cleanup (Method 3620), Gel Permeation Chromatography (Method 3640), and Sulfur cleanup (Method 3660). These cleanup procedures are included in SOP # CORP-OP-0001NC.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

5.2. Aroclors have been classified as a potential carcinogen under OSHA. Concentrated solutions of Aroclors must be handled with extreme care to avoid excess exposure. Contaminated gloves and clothing must be removed immediately. Contaminated skin surfaces must be washed thoroughly.

5.3. All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license.

5.4. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the Director, EH&S shall be immediately notified and a letter sent to the NRC or local state agency.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. A ^{63}Ni electron capture detector is required.

6.2. Refer to Table C-2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to the method 8000B section of this SOP for general requirements for reagents and supplies. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.

7.2. Refer to Table C-3 for details of calibration standards.

7.3. Surrogate Standards

Tetrachloro-m-xylene and decachlorobiphenyl are the surrogate standards. Other surrogates may be used at client request. Refer to Table C-4 for details of surrogate standards.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

Refer to Section 9 of the 8000B section of this SOP.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. Initial Calibration

10.2.1. Refer to Table C-5 for the initial calibration analytical sequence.

10.2.2. The response for each Arochlor will be calculated by the procedures described in the general method for GC analysis, with the following modifications.

10.2.3. A five point calibration of the Arochlor 1016/1260 mix is generated with at least mid level single points for the other Aroclor mixes. The average response factor is used to quantitate Arochlors 1260 and 1016, other Arochlors are quantitated from the mid level single point.

Note: Method 608 requires a minimum of three concentration levels for initial calibrations. The relative standard deviation, RSD must be less than 10%.

10.2.4. The analyst may include a full 5 point calibration for any of the Arochlors with the initial calibration.

10.2.5. The high and low standards for the initial 5 point calibration of 1016 / 1260 define the acceptable quantitation range for the other Arochlors. If any Arochlor is determined above this concentration the extract must be diluted and reanalyzed.

10.2.6. If the analyst knows that a specific Arochlor is of interest for a particular project, that Arochlor may be used for the five point calibration rather than the 1016 / 1260 mix.

10.2.7. The surrogate calibration curve is calculated from the Aroclor 1016/1260 mix. Surrogates in the other calibration standards are used only as retention time markers.

10.2.8. Two options are possible for quantitation of Aroclors. The same quantitation option must be used for standards and samples.

10.2.8.1. Multiple peak option

Select 3-10 major peaks in the analyte pattern. Calculate the response using the total area or total height of these peaks. Alternatively, find the response of each of the 3-10 peaks per Aroclor, and use these responses independently, averaging the resultant concentrations found in samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be coeluting with contaminant peaks from the quantitation. (i.e. peaks which are significantly larger than would be expected from the rest of the pattern.)

10.2.8.2. Total area option

The total area of the standards and samples may be used for quantitation of multi-component analytes. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area. This option should not be used if there are significant interference peaks within the multi-component pattern in the samples. The retention time window for total area measurement must contain at least 90% of the area of the analyte.

10.3. 12 hour Calibration

The 12 hour calibration verification must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter if samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12 hour calibration.

10.3.1. At a minimum, the 12 hour calibration includes analysis of the Arochlor 1260 / 1016 mix.

10.3.2. It is adequate to verify calibration with a mixture of Aroclors 1016 and 1260. If a specific Aroclor is expected, it may be included in the daily calibration check.

10.3.3. The retention time windows for any analytes included in the daily calibration and CCVs are updated.

10.3.4. For this method samples must be bracketed with successful calibration verification runs.

10.4. Calibration verification

The Arochlor 1260/1016 calibration mix is analyzed as the calibration verification standard. This is analyzed after every 20 samples, including matrix spikes, LCS, and method blanks. (Depending on the type of samples, it may be advisable to analyze verifications more frequently in order to minimize reruns.).

10.4.1. A mid level standard is used for the calibration verification.

Note : Method 608 requires that the working calibration curve must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ± 15%, the test must be repeated using a fresh calibration standard.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for general procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP No. CORP-OP-0001NC.

11.3. Cleanup

Cleanup procedures are described in SOP No. CORP-OP-0001NC.

11.4. Suggested gas chromatographic conditions are given in Table C-2.

11.5. Allow extracts to warm to ambient temperature before injection.

11.6. The suggested analytical sequence is given in Table C-5.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Identification of Arochlors

Retention time windows are used for identification of Arochlors, but the "fingerprint" produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

A clearly identifiable Arochlor pattern serves as confirmation of single column GC analysis. Dual column confirmation may be used for specific program requirements or by client request.

12.2. Quantitation of Arochlors

Use 3-10 major peaks or total area for quantitation

If the analyst believes that a combination of Aroclor 1254 and 1260, or a combination of 1242, 1248 and 1232 is present, then only the predominant Arochlor is quantitated and reported, but the suspicion of multiple Aroclors is discussed in the narrative. If well separated Aroclor patterns are present, and then both Aroclors are quantitated and reported.

12.3. If there are no interfering peaks within the envelope of the Arochlor, the total area of the standards and samples may be used for quantitation. Any surrogate or extraneous peaks within the envelope must be subtracted from the total area.

12.4. Second column confirmation of Aroclors will only be performed when requested by the client. The appearance of the multiple peaks in the sample usually serves as a confirmation of Aroclor presence.

12.5. Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX). Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

13.2. Method detection limits (MDL) are determined for all Arochlors.

14. POLLUTION PREVENTION

Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, Update III, December 1996, Method 8082

16.2. CFR136A, Method 608, Organochlorine Pesticides and PCBs.

17. MISCELLANEOUS

17.1. Modifications from Reference Method

17.1.1. Method 8082 includes limited direction for congener specific quantitation. This is outside the scope of this SOP.

17.2. Modifications from Previous Revisions

No changes were made to this Appendix

17.3. Tables

Table C-1
Standard Analyte list and Reporting Limits

Compound	Reporting Limit, µg/L or µg/kg		
	water	soil	waste
Aroclor-1016	1.0	33	1000
Aroclor-1221	1.0	33	1000
Aroclor-1232	1.0	33	1000
Aroclor 1242	1.0	33	1000
Aroclor-1248	1.0	33	1000
Aroclor-1254	1.0	33	1000
Aroclor-1260	1.0	33	1000

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.
Ground water	1000 mL	10 mL
Low-level Soil	30 g	10 mL
High-level soil / waste	1 g	10 mL

Table C-2

Parameter	Recommended Conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	70°C for 0.5min, 30°C/min to 190°C, 2.5°C/min to 225, 18°C/min to 280°C, 3 min hold
Column 1	DB-5 or Rtx-5 30m x 0.32mm id, 0.5µm
Column 2	DB-1701 or Rtx 1701 30m x 0.32 mm id, 0.25µm
Column 3	DB-608, 30m X 0.32 mm, 0.25µm
Injection	1-2µL
Carrier gas	Helium or Hydrogen
Make up gas	Nitrogen
Y splitter	Restek or J&W or Supelco glass tee

Table C-3						
Calibration Levels ng/mL						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6[1]
Aroclor 1016/1260	100	200	500	1000	2000	4000
Aroclor 1242[2]			500			
Aroclor 1221 +1254[2]			500			
Aroclor 1232[2]			500			
Aroclor 1248[2]			500			
Surrogates are included with all the calibration mixes at the following levels:						
Tetrachloro-m-xylene	5	10	25	50	100	200
Decachlorobiphenyl	5	10	25	50	100	200

[1] Level 6 is optional and should only be used if linearity can be maintained on the instrument to this level.

[2] Aroclors may be quantitated within the range 100 to 2000 ng/mL (4000ng/mL if the level 6 1016/1260 standard is included). If the Aroclor is more concentrated, it must be reanalyzed at a dilution.

Table C-4			
LCS/Matrix Spike and Surrogate Spike levels for Aroclor analysis with Acid Cleanup µg/L or µg/kg			
	Aqueous	Soil	Waste
Aroclor 1016/1260	10	333	10,000
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	200

Table C-5 Michigan Analyte List and Reporting Limits[1]		
	Reporting Limit	
Compound	water (µg/L)	soil (µg/Kg)
Aroclor-1016	0.2	330
Aroclor-1221	0.2	330
Aroclor-1232	0.4	330
Aroclor 1242	0.2	330
Aroclor-1248	0.2	330
Aroclor-1254	0.2	330
Aroclor-1260	0.2	330

[1] Reporting Limits are only for samples performed under the Michigan program

Table C-5
Suggested Analytical Sequence

Initial Calibration

Injection #		
1	Solvent blank (optional)	
2	Aroclor 1016/1260	Level 1
3	Aroclor 1016/1260	Level 2
4	Aroclor 1016/1260	Level 3
5	Aroclor 1016/1260	Level 4
6	Aroclor 1016/1260	Level 5
7	Aroclor 1232	Level 3
8	Aroclor 1242	Level 3
9	Aroclor 1248	Level 3
10	Aroclor 1221/1254	Level 3
11-30	Sample 1-20 (or as many samples as can be analyzed in 12 hours Solvent blank (optional)	
32	Aroclor 1016/1260	Level 3

etc

Note: A solvent blank or primer may be analyzed at any time during the sequence when highly contaminated samples are expected. A solvent blank or primer may not be analyzed as routine immediately prior to standards.

12 hour Calibration

At least every 12 hours, counting from the start of the initial calibration, or from the start of the last daily calibration, the retention time windows must be updated using the Aroclor 1260 / 1016 mix. Mid level standards of any other Aroclors expected to be present in the samples are also injected.

1. SCOPE AND APPLICATION

This method is applicable to the gas chromatographic determination of Chlorinated phenoxy acid herbicides in extracts prepared by SOP CORP-OP-0001NC. The herbicides listed in Table D1 are routinely analyzed. Other chlorinated acids may be analyzed by this method if the quality control criteria in Section 9 and the initial demonstration of method performance in Section 13 are met.

1.1. The associated LIMS method code is QS.

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of phenoxy acid herbicides by gas chromatography. The herbicides, as their methyl esters, are injected onto the column, separated, and detected by electron capture detectors. Quantitation is by the external standard method.

3. DEFINITIONS

Refer to the STL North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

4.2. Chlorinated acids and phenols cause the most direct interference with this method.

4.3. Sulfur may interfere and may be removed by the procedure described in SOP#CORP-OP-0001NC.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. A Ni_{63} electron capture detector is required.

6.2. Refer to Table D2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to section 7 of the 8000B section of this SOP for general information on reagents and standards.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).

9.2. Refer to Table D-3 for the components and levels of the LCS and MS mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. Calibration standards are prepared from purchased standards in the methyl ester form.

10.3. The low level standard must be at or below the laboratory reporting limit. Other standards are chosen to bracket the expected range of concentrations found in samples, without saturating the detector or leading to excessive carryover.

10.4. Refer to Table D-2, for details of GC operating conditions.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP #CORP-OP-0001NC.

11.3. Cleanup

The alkaline hydrolysis and subsequent extraction of the basic solution described in the extraction procedure provides an effective cleanup.

11.4. Analytical Sequence

The analytical sequence starts with an initial calibration of at least five points, or a daily calibration that meets % difference criteria from an existing initial calibration.

11.4.1. The daily calibration must be analyzed at least once every 24 hours when samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 24 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a daily calibration.

11.4.2. The daily calibration consists of mid level standards of all analytes of interest. Retention time windows must be updated with the daily calibration.

11.4.3. After every 12 hours a continuing calibration is analyzed. The continuing calibration consists of mid level standards of all analytes of interest. Retention time windows are updated with continuing calibrations.

11.5. Gas Chromatography

Chromatographic conditions are listed in Table D-2.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. The herbicides are analyzed as their methyl esters, but reported as the free acid. For this reason it is necessary to correct the results for the molecular weight of the ester versus the free acid. This is achieved through the concentrations of the calibration standards. For example the 20µg/L calibration standard for 2,4-D contains 21.3 µg/L of the methyl ester. No further correction is necessary.

12.3. A routine 10X dilution occurs on final extracts for all samples. Due to a QuantIMS limitation, the dilution factor field in QuantIMS cannot be used when a dilution is routine, because the dilution factor is automatically applied to all reference values creating reporting problems. For the herbicide analysis, the extract volume will be 10mL and an aliquot at 10X dilution will be analyzed. The final extract volume recorded on the laboratory bench sheet will be recorded as 100mL to avoid using the dilution factor field in QuantIMS.

13. METHOD PERFORMANCE

13.1. The EPA for this method has not published multiple laboratory performance data. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

14. POLLUTION PREVENTION

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

Method 8151A, SW-846, Update III, December 1996

17. MISCELLANEOUS

17.1. Modifications from Reference Method

Refer to the method 8000B section of this SOP for modifications from the reference method.

17.2. Modifications from Previous Revision

The calibration procedure has been changed to require esterification of the calibration standards

17.3. Tables

Table D-1				
Standard Analyte list				
Compound	CAS Number	Reporting Limit, µg/L or µg/kg		
		Aqueous	Soil	Waste
2,4-D	94-75-7	4	80	4000
2,4-DB	94-82-6	4	80	4000
2,4,5-TP (Silvex)	93-72-1	1	20	1000
2,4,5-T	93-76-5	1	20	1000
Dalapon	75-99-0	2	40	2000
Dicamba	1918-00-9	2	40	2000
Dichloroprop	120-36-5	4	80	4000
Dinoseb	88-85-7	0.6	12	600
MCPA	94-74-6	400	8000	400,000
MCPP	93-65-2	400	8000	400,000

The following concentration factors are assumed in calculating the Reporting Limits:

	Extraction Vol.	Final Vol.	Dilution Factor
Ground water	1000 mL 10 mL	20	
Low-level Soil without GPC	50 g	10 mL	20
High-level soil / waste	1 g	10 mL	20

Specific reporting limits are highly matrix dependent. The reporting limits listed above are provided for guidance only and may not always be achievable. For special projects, the extracts may be analyzed without any dilution, resulting in reporting limits 20 times lower than those in Table D-1.

Table D-2	
Instrumental Conditions	
PARAMETER	Recommended conditions
Injection port temp	220°C
Detector temp	325°C
Temperature program	80,2/30/170,0/1/180,1
Column 1	DB-5MS or RTX 5 30x0.32, 0.5um
Column 2	DB-1701 or Rtx-1701
Injection	1-2µL
Carrier gas	Helium / Hydrogen
Make up gas	Nitrogen

Recommended conditions should result in resolution of all analytes listed in Table D-1.

The reporting limits listed in Table D-1 will be achieved with these calibration levels and a 20 fold dilution of the sample extract. Lower reporting limits can be achieved with lesser dilutions of the sample extract.

Table D-3			
LCS/Matrix Spike and Surrogate Spike levels µg/L or µg/kg[1]			
	Aqueous	Soil	Waste
2,4-D	16	800	16000
Silvex	4	200	4000
2,4,5-T	4	200	4000
2,4-DB	16	800	16000
Dalapon	8	400	8000
DCAA (surrogate)	16	800	16000

[1] LCS, MS and SS spikes are as the free acid.

1. SCOPE AND APPLICATION

1.1. This SOP Appendix describes procedures to be used when SW-846 Method 8000B is applied to the analysis of polynuclear aromatic hydrocarbons (PAH) by High Performance Liquid Chromatography. This Appendix is to be applied when SW-846 Method 8310 or EPA Method 610 is requested, and is applicable to extracts derived from any matrix which are prepared according to the appropriate STL North Canton sample extraction SOPs. (CORP-OP-0001NC).

Table E-1 lists compounds, which are routinely determined by this method, and gives the Reporting Limits (RL) for each matrix. RLs given are based on the low level standard and the sample preparation concentration factors. Matrix interferences may result in higher RLs than those listed. Note: Reporting limits are subject to change due to compound sensitivity, calibration levels, MDLs, and other factors.

1.2. The associated LIMS method codes are SG (8310) and VT (610).

2. SUMMARY OF METHOD

This method presents conditions for the analysis of prepared extracts of polynuclear aromatic hydrocarbons by high performance liquid chromatography. The compounds are injected onto a column, separated, and detected by ultraviolet and fluorescence detectors. Quantitation is by the external standard method.

3. DEFINITIONS

Refer to the STL North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

5.2. The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: Benzo(a)anthracene, Benzo(a)pyrene, and Dibenzo(a,h)anthracene. Primary standards of these toxic compounds should be prepared in a hood.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP. An Ultraviolet detector and Fluorescence detector is required.

6.2. Refer to Table E2 for analytical columns.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to section 7 of the 8000B section of this SOP for general information on reagents and standards.

7.2. Refer to Table E-3 and E-4 for details of calibration and other standards.

7.3. Surrogate Standards

Terphenyl-d14 and Benzo(e)pyrene are the surrogate standards. Refer to table E-3 for details of surrogate standards.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

Refer to section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).

9.2. Refer to Table E-3 for the components and levels of the LCS and MS mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

Note: The Initial Calibration requirement for Method 610 is as follows: If the ratio of response to amount injected (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

10.2. Calibration standards are prepared from purchased solutions. Table E-4 lists the calibration levels.

10.3. The low level standard must be at or below the laboratory reporting limit. Other standards are chosen to bracket the expected range of concentrations found in samples, without saturating the detector or leading to excessive carryover.

10.4. Refer to Table E-2, for details of LC operating conditions.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP #CORP-OP-0001NC.

11.3. Cleanup

11.4. Analytical Sequence

The analytical sequence starts with an initial calibration of at least five points, or a daily calibration that meets % difference criteria from an existing initial calibration.

Note: Method 610 requires that continuing calibration verifications may not vary from the predicted response for any parameter by more than ± 15%. The average response may not be used for Method 610.

11.4.1. The daily calibration must be analyzed at least once every 24 hours when samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 24 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a daily calibration.

11.4.2. The daily calibration consists of mid level standards of all analytes of interest. Retention time windows must be updated with the daily calibration.

11.4.3. After every 12 hours a continuing calibration is analyzed. The continuing calibration consists of mid level standards of all analytes of interest. Retention time windows are updated with continuing calibrations.

11.5. High Performance Liquid Chromatography

Chromatographic conditions are listed in Table E-2.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. Surrogate recovery results are calculated and reported for Terphenyl-d14 and Benzo(e)pyrene unless it is determined that sample interference has adversely affected the quantitation of one of the surrogates. One surrogate must be within QC criteria. Corrective action is only necessary if Terphenyl-d14 and Benzo(e)pyrene are both outside of acceptance limits.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability required as referenced under Section 13.1

14. POLLUTION PREVENTION

Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, Update III, December 1996, Method 8310.

16.2. CFR136 Appendix A Method 610-Polynuclear Aromatic Hydrocarbons.

17. MISCELLANEOUS

17.1. Reporting limits

17.1.1. The lower standard reporting limits are listed in Table

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Elution time

17.2.1. For any positive detection in a sample, the chromatogram is overlaid on screen with the nearest standard for elution time matching and pattern recognition using the Target system.

17.3. Analyte detection

17.3.1. Some compounds are detected only on the ultraviolet detector. For other compounds, the lower of the concentrations calculated from both detectors is reported, the second detector serving as confirmation of the analytes presence.

17.3.2. For any compound that is detected only on the ultraviolet detector, confirmation analysis is not possible. These detected compounds must be narrated that the quantitation is not confirmed by a second detector (Fluorescence).

17.3.3. Some compounds, due to calibration levels and detector limitations, can only be confirmed using the Fluorescence detector. The result will always be reported from the UV detector. These compounds will be narrated, when applicable. Table E-5 lists the compounds and respective detectors.

17.4. Troubleshooting guide

17.4.1. Consult the instrument manufacturer's operating manual for guidance.

17.5. Tables

Table E-1
Standard Analyte list

Compound	CAS Number	Reporting Limit, µg/L or µg/kg	
		Aqueous	Soil
Acenaphthene	83-32-9	1	100
Acenaphthylene	208-96-8	1	100
Anthracene	120-12-7	2	100
Benzo(a)anthracene	56-55-3	0.1	5
Benzo(a)pyrene	50-32-8	0.1	5
Benzo(b)fluoranthene	205-99-2	0.1	5
Benzo(g,h,i)perylene	191-24-2	0.1	10
Benzo(k)fluoranthene	207-08-9	0.1	1.7
Chrysene	218-01-9	0.1	5
Dibenzo(a,h)anthracene	53-70-3	0.1	5
Fluoranthene	206-44-0	0.1	10
Fluorene	86-73-7	1	100
Indeno(1,2,3-cd)pyrene	193-39-5	0.1	5
Naphthalene	91-20-3	2	100
Phenanthrene	85-01-8	1	100
Pyrene	129-00-0	0.1	5
1-Methylnaphthalene*	90-12-0	2	100
2-Methylnaphthalene*	91-57-6	2	100

Table E-2
Instrumental Conditions

PARAMETER	Recommended conditions			
Injection volume	20 uL			
Detector	HP Series 1100 Fluorescence Detector			
Detector	HP Series 1100 Variable Wavelength Detector (UV)			
Solvent Program	Time	% Water	% Acetonitrile	Flow
	0 min.	50	50	1.5 mL/m
	5 min.	45	55	1.5 mL/m
	10 min.	35	65	2.0 mL/m
	15 min.	25	75	2.0 mL/m

	20 min. 25 min. 30 min.	15 5 45	85 95 55	2.0 mL/m 2.0 mL/m 2.0 mL/m
Column 1	PAH Hypersil 5 micron			
Injection	20 uL			
Solvent	Acetonitrile, HPLC grade			
Solvent	Reagent Water, HPLC grade			

Table E-3 LCS/Matrix Spike and Surrogate Spike Compounds

Compound	Concentration	Compound	Concentration
Acenaphthene	10 ug/mL	Dibenzo(a,h)anthracene	2 ug/mL
Acenaphthylene	10 ug/mL	**Fluoranthene**	2 ug/mL
Anthracene	10 ug/mL	Fluorene	2 ug/mL
Benzo(a)anthracene	2 ug/mL	**Indeno(1,2,3-cd)pyrene**	2 ug/mL
Benzo(a)pyrene	2 ug/mL	**Naphthalene**	10 ug/mL
Benzo(b)fluoranthene	2 ug/mL	**Phenanthrene**	2 ug/mL
Benzo(g,h,i)perylene	2 ug/mL	Pyrene	2 ug/mL
Benzo(k)fluoranthene	2 ug/mL	Terphenyl-d14*	1 ug/mL
Chrysene	2 ug/mL	Benzo(e)pyrene*	5 ug/mL

Bold print denotes control compounds in the LCS/LCSD and MS/MSD.

* Denotes surrogate compounds

Table E-4 Calibration Standards

Component	ST	1:2	1:5	1:10	1:20	1:50	1:100
Naphthalene	50	25	10	5	2.5	1	0.5
Acenaphthylene	50	25	10	5	2.5	1	0.5
Terphenyl d14 (surrogate)	50	25	10	5	2.5	1	0.5
Acenaphthene	10	5	2	1	0.5	0.2	0.1
Fluorene	10	5	2	1	0.5	0.2	0.1
Phenanthrene	10	5	2	1	0.5	0.2	0.1
Anthracene	10	5	2	1	0.5	0.2	0.1
Chrysene	10	5	2	1	0.5	0.2	0.1
1-Methylnaphthalene	50	25	10	5	2.5	1	0.5
Fluoranthene	10	5	2	1	0.5	0.2	0.1
Pyrene	10	5	2	1	0.5	0.2	0.1
Benzo(a)anthracene	10	5	2	1	0.5	0.2	0.1
Benzo(e)pyrene (surrogate)	50	25	10	5	2.5	1	0.5
Benzo(b)fluoranthene	10	5	2	1	0.5	0.2	0.1
Benzo(k)fluoranthene	10	5	2	1	0.5	0.2	0.1

Table E-4 Calibration Standards

Component	ST	1:2	1:5	1:10	1:20	1:50	1:100
Benzo(a)pyrene	10	5	2	1	0.5	0.2	0.1
Dibenzo(a,h)anthracene	10	5	2	1	0.5	0.2	0.1
Benzo(g,h,i)perylene	10	5	2	1	0.5	0.2	0.1
Indeno(1,2,3-cd)pyrene	10	5	2	1	0.5	0.2	0.1
2-Methylnaphthalene	50	25	10	5	2.5	1	0.5

Table E-5 Compound/Detector

Compound	Ultraviolet 254 nm	Fluorescence 280 nm
Acenaphthene	X	X
Acenaphthylene	X	
Anthracene	X	Retention Time confirmation only
Benzo(a)anthracene	X	X
Benzo(a)pyrene	X	X
Benzo(b)fluoranthene	X	X
Benzo(g,h,i)perylene	X	X
Benzo(k)fluoranthene	X	X
Chrysene	X	X
Dibenzo(a,h)anthracene	X	X
Fluoranthene	X	X
Fluorene	X	X
Indeno(1,2,3-cd)pyrene	X	X
Naphthalene	X	X
Phenanthrene	X	X
Pyrene	X	X
1-Methylnaphthalene	X	X
2-Methylnaphthalene	X	X

1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of the concentration of certain Organophosphorus Pesticides in waters, wastewaters, oils, soils, and sludges. It is based on SW846 Method 8141A. Table F1 shows reporting limits for compounds routinely analyzed by this method. The compounds include, but are not limited to, those shown in Table F1.

1.2. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

1.3. The associated LIMS method code is P2.

2. SUMMARY OF METHOD

2.1. An aliquot of prepared sample is injected in a gas chromatograph (GC) and compounds in the effluent are detected by a flame photometric detector. Appropriate preparation techniques are described in SOP CORP-OP-0001NC. Ultrasonic Extraction (Method 3550) is **NOT** an appropriate sample preparation for Method 8141 and should not be used because of the potential for destruction of target analytes during the ultrasonic extraction process.

3. DEFINITIONS

Refer to the STL North Canton Laboratory Quality Manual (LQM), current version, for definitions of terms used in this document.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

4.2. Analytical difficulties encountered for target analysis include:

4.2.1. The water solubility of Dichlorvos (DDVP) is 10 g/L at 20°C, and recovery is poor from aqueous solution.

4.2.2. Naled is converted to Dichlorvos (DDVP) on column by debromination. This reaction may also occur during sample workup.

4.2.3. Trichlorfon rearranges and is dehydrochlorinated in acidic, neutral, or basic media to form Dichlorvos (DDVP) and hydrochloric acid. If this method is to be used for the determination of organophosphates in the presence of Trichlorfon, the analyst should be aware of the possibility of rearrangement to Dichlorvos to prevent misidentification.

4.2.4. Merphos is a single component pesticide that is readily oxidized to Merphos oxone. Chromatographic analysis of Merphos almost always results in two peaks.

5. SAFETY

5.1. Refer to Section 5 of the Method 8000B SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP.

6.2. Refer to Table F2 for Instrument settings.

6.3. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to Section 7 of the 8000B section of this SOP for general information on reagents and standards.

7.2. Refer to Table F-3 for details of calibration and other standards.

7.3. Surrogate Standards

Triphenyl phosphate is the surrogate standard. Refer to table F-4 for details of the surrogate standard.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

Refer to section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).

9.2. Refer to Table F-4 for the components and levels of the LCS and MS mixes.

10. CALIBRATION AND STANDARDIZATION

10.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

10.2. Calibration standards are made up from purchased solutions. Table F-3 lists the calibration levels.

10.3. The low level standard must be at or below the laboratory reporting limit. Other standards are chosen to bracket the expected range of concentrations found in samples, without saturating the detector or leading to excessive carryover.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP #CORP-OP-0001NC.

11.3. Cleanup

11.4. Analytical Sequence

The analytical sequence starts with an initial calibration of at least five points, or a daily calibration that meets % difference criteria from an existing initial calibration.

11.4.1. The daily calibration must be analyzed at least once every 24 hours when samples are being analyzed. If there is a break in the analytical sequence of greater than 12 hours, then a new continuing calibration run must be analyzed before proceeding with the sequence. If more than 24 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a daily calibration.

11.4.2. The daily calibration consists of mid level standards of all analytes of interest. Retention time windows must be updated with the daily calibration.

11.4.3. After every 12 hours a continuing calibration is analyzed. The continuing calibration consists of mid level standards of all analytes of interest. Retention time windows are updated with continuing calibrations.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. Surrogate recovery results are calculated and reported for Triphenylphosphate unless it is determined that sample interference has adversely affected the quantitation of one of the surrogates. The surrogate must be within QC criteria. Corrective action is only necessary if Triphenylphosphate is outside of acceptance limits.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

14. POLLUTION PREVENTION

Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

SW846, Update III, December 1996, Method 8141A.

17. MISCELLANEOUS

17.1. Reporting limits

17.1.1. The lower standard reporting limits are listed in Table F-1

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.1.2.1. The nature of the FPD detector contributes to high dilutions for Method 8141A. There is a phenomenon known as quenching that occurs. This happens when light absorption occurs in the flame of the FPD due to hydrocarbons, sulfur, and certain light absorbing compounds.

When this happens the analytes of interest do not reach the photomultiplier tube and are not detected even though they may be present.

17.2. Elution time

17.2.1. For any positive detection in a sample, the chromatogram is overlaid on screen with the nearest standard for elution time matching and pattern recognition using the Target system.

17.3. Troubleshooting guide

17.3.1. Consult the instrument manufacturer's operating manual for guidance.

Table F1: Organophosphorus Pesticides Routinely Analyzed and Reporting Limits

Compound	CAS Number	Reporting Limits	
		Water, µg/L	Solid, µg/kg
Azinphos methyl	86-50-0	1.0	33
Bolstar (Suprofos)	35400-43-2	1.0	33
Chlorpyrifos	2921-88-2	1.0	33
Coumaphos	56-72-4	1..0	33
Demeton, O and S	8065-48-3	1.0	33
Diazinon	333-41-5	1.0	33
Dichlorvos	62-73-7	1.0	33
Disulfoton	298-04-4	1.0	33
Ethoprop	13194-48-4	1.0	33
Fensulfothion	115-90-2	1.0	33
Fenthion	55-38-9	1.0	33
Malathion	121-75-5	1.0	33
Merphos	150-50-5	1.0	33
Methyl Parathion	298-00-0	1.0	33
Mevinphos	7786-34-7	1.0	33
Naled	300-76-5	1.0	33
Phorate	298-02-2	1.0	33
Ronnel	299-84-3	1.0	33
Stirophos	22248-79-9	1.0	33
Tokuthion	34643-46-4	1.0	33
Trichloronate	327-98-0	1.0	33
O,O,O-Trientyl phosphorothioate	126-68-1	1.0	33
Thionazin	297-97-2	1.0	33
Sulfotepp	3689-24-5	1.0	33
Dimethoate	60-51-5	1.0	33
Parathion	56-38-2	1.0	33
Famphur	52-85-7	1.0	33

Table F2: Instrumental Conditions	
PARAMETER	Recommended conditions
Injection port temp	175°C
Detector temp	230°C
Initial temp	135°C
Temperature program	(A) 5°C/minute (B) 20°C/minute
Final Temp	(A) 245°C (B) 295°C
Final Hold Time	(A) 0 minutes (B) 7 minutes
Column 1	RTX-OPP, 30 meter, 0.32 mm, 0.5 µm film
Column 2	RTX-1, 30 meter, 0.32 mm, 0.5 µm film
Injection	1-2µL
Carrier gas	Helium / Hydrogen
Make up gas	Nitrogen

Table F3 Initial Calibration Concentrations (ng/uL)

COMPOUND	LEVEL1	LEVEL2	LEVEL3	LEVEL4	LEVEL5	LEVEL6	LEVEL7
o,o,o-Triethylp-hosphate	0.2	0.5	1	2	5	10	20
Dichlorvos	0.2	0.5	1	2	5	10	20
Mevinphos	0.2	0.5	1	2	5	10	20
Thionazin	0.2	0.5	1	2	5	10	20
Ethoprop	0.2	0.5	1	2	5	10	20
Naled	0.2	0.5	1	2	5	10	20
Sulfotepp	0.2	0.5	1	2	5	10	20
Phorate	0.2	0.5	1	2	5	10	20
Demeton	0.2	0.5	1	2	5	10	20
Dimethoate	0.2	0.5	1	2	5	10	20
Diazinon	0.2	0.5	1	2	5	10	20
Disulfoton	0.2	0.5	1	2	5	10	20
Methyl Parathion	0.2	0.5	1	2	5	10	20
Ronnel	0.2	0.5	1	2	5	10	20
Fenthion	0.2	0.5	1	2	5	10	20
Chlorpyrofos	0.2	0.5	1	2	5	10	20
Parathion	0.2	0.5	1	2	5	10	20
Malathion	0.2	0.5	1	2	5	10	20
Trichloronate	0.2	0.5	1	2	5	10	20
Merphos	0.2	0.5	1	2	5	10	20
Stirophos	0.2	0.5	1	2	5	10	20
Tokuthion	0.2	0.5	1	2	5	10	20
Fensulfothion	0.2	0.5	1	2	5	10	20
Bolstar	0.2	0.5	1	2	5	10	20
Famphur	0.2	0.5	1	2	5	10	20
Azinphos methyl	0.2	0.5	1	2	5	10	20
Coumaphos	0.2	0.5	1	2	5	10	20

Table F4: LCS/Matrix Spike and Surrogate Spike Compounds – 20 ug/mL

Compound	Compound
Thinazin	Sulfotepp
Phorate	Disulfoton
Methyl Parathion	Parathion
Famphur	O,O,O-Triethylphosphate
Dimethoate	Triphenyl Phosphate - Surrogate

1. Scope and Application

1.1. This method is applicable to the determination of the concentration and tentative identification of petroleum hydrocarbon mixes in waters, wastewaters, soils, and sludges.

1.2. This SOP is based on SW-846 Method 8015B, Modified, Revision 3, December 1996 and Wisconsin DNR Modified DRO method.

1.3. The associated LIMS method codes are HS (8015 MOD), KI (8015B), and C6 (Wisconsin DRO).

2. Summary of Method

2.1. This method provides gas chromatographic conditions for detection and identification of total petroleum hydrocarbons. Prior to the use of this method, appropriate sample preparation techniques are used.

2.2. Wisconsin DRO Method is designed to measure the concentration of diesel range organics in water and soil. This corresponds to a hydrocarbon range of C_{10}-C_{28} and a boiling point range between approximately 170 °C and 430 °C.

2.3. An aliquot of the prepared sample is injected into a gas chromatograph (GC) and compounds in the effluent are detected by a flame ionization detector (FID).

3. Definitions

3.1. Refer to the glossary in the STL North Canton Laboratory Quality Manual (LQM), current version.

3.2. Wisconsin DNR DRO Method Definition - Diesel Range Organics (DRO) – All the chromatographic response falling between the onset of the n-decane (n-C_{10}) peak and the conclusion of the n-octacosane (n-C_{28}) peak. Quantitation is based on a direct comparison of the total area within this range to the total area of the Diesel Component Standard.

3.3. Wisconsin DNR DRO Method Definition –Diesel Component Standard: A ten component blend of typical diesel compounds. This standard serves as a quantitation standard and is used to establish a retention time window for diesel range organics.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

5. SAFETY

5.1. Refer to Section 5 of the Method 8000B SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP.

6.2. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to Section 7 of the 8000B section of this SOP.

7.2. The petroleum hydrocarbons are purchased from a chemical supplier when available. When no chemical supplier is available, the fuels are purchased from public sources.

7.3. The Diesel Component Stock Standard for the Wisconsin DRO method may be commercially prepared standards if the standards are certified by the manufacturer or by an independent source.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

8.1. Refer to Section 8 of the 8000B section of this SOP.

8.2. Wisconsin DNR DRO aqueous samples must be preserved with HCL such that the pH is < 2.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).

9.2. LCS recoveries are calculated from a Diesel Calibration. The Wisconsin DNR DRO method specifies the recovery of the LCS-water spike must be between 75%-115% and the RPD <20%. LCS Soil recoveries must fall between 70%-120% of the known concentration and the RSD must be <20%.

9.2.1. The Wisconsin DNR DRO Method requires a Duplicate Laboratory Control Spike with each batch. MS/MSD samples are not required for this method. One LCS must be run at the beginning of the batch of samples and the other at the end.

9.3. MS/MSD recoveries are calculated from a Diesel calibration.

9.4. Surrogates

9.4.1. Because of the nature of the TPH analysis, whereas certain petroleum mixtures can override the C9 surrogate, the C9 surrogate recoveries are advisory. Re-extraction due to surrogate recoveries is determined by analyst judgement.

NOTE: Ohio VAP rules require reanalysis when surrogate recoveries are outside of control limits.

10. CALIBRATION AND STANDARDIZATION

10.1. Recommended Instrument Conditions

10.1.1. Hydrogen carrier gas - flow rate 5 - 6 mL/min

10.1.2. Detector gas mixture - air hydrogen mixture in a 10:1 ratio, air 80 - 120 mL/min, hydrogen 8 -12 mL/min

10.1.3. Temperature Program - refer to Appendix

10.1.4. Injection volume - 1 µL

10.2. Initial Calibration

10.2.1. Analyze a five point Diesel calibration standard and a five point DRO calibration standard referring to the recommended instrument conditions. The calibration concentrations are 100, 200, 500, 1000, and 2000 ng/uL. A 5000ng/uL standard may be analyzed if needed. The retention time window of C10-C32 shall be used for the Diesel calibration.

10.2.2. The Wisconsin DNR DRO method requires the retention time window be defined as beginning 0.1 minutes before the onset of the n-decane peak and ending 0.1 minutes after the conclusion of the n-octacosane peak in the calibration run.

10.2.3. Wisconsin DNR DRO quantitation is based on a direct comparison of the total area within the retention time window to the total area of the Diesel Component Standard.

10.2.4. Wisconsin DNR DRO integration must be "baseline to baseline" as opposed to a "valley to valley". Baseline to baseline is defined here as a flat baseline drawn parallel to the x-axis of chromatographic graph that includes all responses within the retention time window.

10.2.5. A linear regression calibration curve must be prepared for Wisconsin DNR DRO analysis. The curve must have a correlation coefficient of at least 99%.

10.3. Continuing Calibration

10.3.1. The calibration verification must bracket the LCS, MS, MSD extracts and must meet passing criteria. The Wisconsin DNR DRO method requires the calibration verification must not vary from the calibration curve by more than 20%.

10.4. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for procedural requirements.

11.2. Extraction

The extraction procedure is described in SOP #CORP-OP-0001NC.

11.3. Cleanup

11.4. Analytical Sequence – Refer to Section 11 in the 8000B Section of this SOP.

11.5. Petroleum Hydrocarbon Identification and/or Fingerprinting

11.5.1. To identify the type of petroleum hydrocarbon, compare the chromatographic peak pattern to the patterns of known petroleum hydrocarbons analyzed under identical chromatographic conditions. Samples are quantified against diesel, but fingerprinting may be done when client requested.

11.5.2. Positive matching may not be possible, even using site-specific hydrocarbons. Degradation of the pattern can occur during environmental exposure of the fuel. See Table 2 for possible fingerprints.

11.6. Sample Quantification

11.6.1. Samples are quantified against the initial calibration of diesel or DRO on a single column.

11.6.2. The total height or area of the hydrocarbon is determined in the same manner used for the hydrocarbon standard.

11.6.3. If the amount of sample injected into the GC exceeds the working range of the calibration curve, an appropriate dilution is performed before reanalysis.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. Surrogate recovery results are calculated and reported for Nonane (C-9) unless it is determined that sample interference has adversely affected the quantitation of one of the surrogates. The surrogate must be within QC criteria. Corrective action is only necessary if Nonane (C-9) is outside of acceptance limits.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

14. POLLUTION PREVENTION

Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, Method 8015B, Nonhalogenated Organics Using GC/FID, Test Methods for Evaluating Solid Waste, Third Edition, USEPA

16.2. Method for Determining Diesel Range Organics, Modified DRO, Wisconsin DNR, Public Hearing Draft, September 1995.

16.3. Related SOP

16.3.1. CORP-OP-0001NC, Extraction and Cleanup of Organic Compounds from Waters and Soils, Based on SW846 3500 Series, 3600 Series, 8150, 8151, and 600 Series Methods

Table G1: Suggested GC Temperature Program for TPH analysis

Initial Temperature	40°C
Initial Hold Time	4 minutes
Temperature Program	10°C/minute
Final Temperature	280°C
Final Hold Time	10 minutes

Table G2: Reporting Limits for TPH Analysis

Analyte	Reporting Limits		
	Water (µg/L)	Solids (mg/kg)	Waste Dilution (mg/kg)
TPH (as Diesel) or DRO	100	3.3	200
C10-C32 (Ohio VAP only)	100	3.3	200
Fingerprint Compounds[1]			
Mineral Spirits	Kerosene	Motor Oil	
Hydraulic Oil	Jet Fuel	Stoddard Solvent	
DRO Spiking Solution			
Decane	Dodecane	Tetradecane	
Hexadecane	Octobecane	Eicosane	
Docosane	Tetracosane	Hexacosane	
Octacosane			

[1] This list represents most of the common petroleum hydrocarbons. The list may be expanded to include other petroleum hydrocarbons.

1. Scope and Application

1.1. This method is applicable to the determination of the concentration of various Non-halogenated Organic Compounds in waters, wastes, sludges, and solids. It is based on SW-846 Method 8015B.

1.2. The applicable LIMs method codes are J7 (GC/FID 8015) and QU (Semivolatile Organics, 8015B). The preparation code is 88.

2. Summary of Method

2.1. This method provides gas chromatographic conditions for the detection of various nonhalogenated organic compounds. Samples are introduced to the GC by direct injection. Detection is achieved by a flame ionization detector (FID).

3. Definitions

3.1. Refer to the glossary in the STL North Canton Laboratory Quality Manual (LQM).

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP.

6.2. Recommended Columns:

6.2.1. RTX Stabilwax, fused silica, 60 m x 0.53, 0.5 µm film thickness, or equivalent column.

6.2.2. Stabilwax-DA, fused silica, 60 m x 0.32, 0.5 µm film thickness, or equivalent column.

6.3. Detectors: Flame ionization (FID)

6.4. Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

7. REAGENTS AND STANDARDS

7.1. Refer to Section 7 of the 8000B section of this SOP.

7.1.1. Reagent water

7.2. Standards

7.2.1. Refer to Table H2.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

8.1. Refer to section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).

10. CALIBRATION AND STANDARDIZATION

10.1. Recommended Instrument Conditions

10.1.1. The following conditions are recommended. The following table lists specific information for various compounds.

10.1.2. RTX- Stabilwax and Stabilwax-DA Columns:

Carrier Gas:	Hydrogen
Initial Temp:	45 °C
Initial Hold:	3 mins
Ramp Rate A:	5 °C/min
Ramp Rate B:	30 °C/min
Final Hold A:	0 min
Final Hold B:	3 mins
Analysis Time:	17.83 mins
Injector Temp:	275 °C
FID Temp:	300 °C
Injection Vol:	1 µL

10.2. Initial Calibration

10.2.1. For each non-halogenated organic compound and surrogate standard, analyze five or more calibration standards referring to the recommended GC conditions in Section 10.1. One of the standards analyzed should be at or near the concentration which corresponds to the calibration range.

10.2.2. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for procedural requirements.

11.2. Sample Preparation Summary

11.2.1. Samples received fall into three general categories: waters, soils, or wastes.

11.3. Sample Preparation Procedure

11.3.1. Waters

11.3.1.1. Add surrogate solution to the sample to achieve a concentration of 100 mg/L.

11.3.2. Sediments/soils and waste

11.3.2.1. Mix the contents of the sample container with a narrow metal or wood spatula. Weigh 5 g (wet weight) into a tarred culture tube. Use a top-loading balance. Record the weight to 0.01 gram.

11.3.2.2. Quickly add 5 mL of reagent water. Add surrogate standard. Cap the vial and vortex to mix for two minutes.

11.3.2.3. If extract is cloudy or has suspended sediment particles, refrigerate and allow sample to sit for a maximum of 24 hours. Filter sample if necessary.

11.4. Sample Analysis

11.4.1. Preliminary Evaluation

11.4.1.1. The sample or sample extract is introduced to the GC column by direct inject techniques. The concentration of the sample components is then calculated from the resulting chromatograms.

11.4.2. Analytical Sequence – Refer to Section 11 in the 8000B Section of this SOP.

11.4.3. Inject 1 µL of the sample extract or diluted sample into the GC using the same operating conditions and techniques as those used in the calibration of the instrument.

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.5.2. All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

12.2. Surrogate recovery results are calculated and reported unless it is determined that sample interference has adversely affected the quantitation of the surrogate. The surrogate must be within QC criteria. Corrective action is only necessary if the surrogate is outside of acceptance limits.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

14. POLLUTION PREVENTION

Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, Method 8015B, Nonhalogenated Organics Using GC/FID, Test Methods for Evaluating Solid Waste, Third Edition, USEPA

17. Miscellaneous (Tables, Appendices, Etc...)

Table H1 Non-halogenated Organic Compounds, Reporting Limits[1]

Compound	CAS Number	Reporting Limits	
		Water, mg/L	Solid, mg/kg
2-Methoxyethanol	109-86-4	1.0	1.0
Methanol	67-56-1	1.0	0.5
Isopropyl alcohol	67-63-0	1.0	0.5
n-Propyl alcohol	71-23-8	1.0	0.5
Ethanol	64-17-5	1.0	0.5
n-Butanol	71-36-3	1.0	0.5
1,4-Dioxane	123-91-1	1.0	0.5

Compound	CAS Number	Reporting Limits	
		Water, mg/L	Solid, mg/kg
Ethylene oxide	75-21-8	1.0	0.5
iso-Butanol	78-83-1	1.0	0.5

[1] If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

Table H2 Non-halogenated Organic Compounds Working Standards

COMPOUND	LEVEL1	LEVEL2	LEVEL3	LEVEL4	LEVEL5	LEVEL 6
2-Methoxyethanol	1.0	2.0	5.0	10.0	20.0	50.0
Methanol	1.0	2.0	5.0	10.0	20.0	50.0
Isopropyl alcohol	1.0	2.0	5.0	10.0	20.0	50.0
n-Propyl alcohol	1.0	2.0	5.0	10.0	20.0	50.0
Ethanol	1.0	2.0	5.0	10.0	20.0	50.0
n-Butanol	1.0	2.0	5.0	10.0	20.0	50.0
1,4-Dioxane	1.0	2.0	5.0	10.0	20.0	50.0
Ethylene oxide	1.0	2.0	5.0	10.0	20.0	50.0
iso-Butanol	1.0	2.0	5.0	10.0	20.0	50.0

1. Scope and Application

1.1. This method is applicable to the determination of the concentration of Sulfolane and N-Methyl-2-pyrrolidone in water and solid samples. It is based on SW846 Method 8015B. The working linear range is 50 to 1000 µg/L. Table I1 lists the reporting limits associated with this method.

1.2. The applicable LIMS method code is KU.

2. Summary of Method

2.1. This method provides gas chromatographic conditions for the detection of mg/L levels of Phillips 66 compounds in water. Prior to use of this method, appropriate sample extraction techniques must be used.

3. Definitions

3.1. Refer to the glossary in the STL North Canton Laboratory Quality Manual (LQM), current version.

4. INTERFERENCES

4.1. Refer to the method 8000B section of this SOP for general information regarding chromatographic interferences.

5. SAFETY

5.1. Refer to section 5 of the Method 8000B SOP for general safety requirements.

5.2. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.2.1. Chemicals that have been classified as **carcinogens**, or **potential carcinogens**, under OSHA include: **Methylene Chloride.**

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP.

6.2. Gas Chromatograph

6.2.1. Gas Chromatograph: Modified to accept capillary columns

6.2.2. Data System: Capable of peak integration

6.2.3. Gas Chromatographic Column: 30 m x 0.32 mm ID RTX-5 fused silica capillary column

6.2.4. Autosampler: Capable of reproducible injections

6.2.5. Carrier Gas: Hydrogen

6.2.6. Detector: Flame ionization (FID)

6.3. Volumetric Flasks: 10, 50, and 100 mL

6.4. Microsyringe: 10 µL

6.5. Pipettes: Disposable µL, Pasteur

6.6. Autosampler Vials: 1 mL with 11 mm crimp cap, Teflon®/silicone septum liner.

7. REAGENTS AND STANDARDS

7.1. Refer to Section 7 of the 8000B section of this SOP.

7.2. Reagents

7.2.1. Methylene Chloride: Pesticide grade or equivalent

7.3. Standards

7.3.1. Refer to Section 7 of the 8000B section of this SOP.

8. SAMPLE PREPARATION, PRESERVATION AND STORAGE

8.1. Refer to Section 8 of the 8000B section of this SOP.

9. QUALITY CONTROL

9.1. Refer to Section 9 of the 8000B section of this SOP for quality control requirements, including the initial demonstration of capability, definition of a batch, surrogate limits, method blanks, laboratory control samples (LCS), and matrix spikes (MS).

9.2. Surrogates are not used for this analysis.

10. CALIBRATION AND STANDARDIZATION

10.1. Initial Calibration

10.1.1. Refer to Section 10 of the 8000B section of this SOP for general calibration requirements.

11. PROCEDURE

11.1. Refer to the method 8000B section of this SOP for procedural requirements.

11.2. Sample Analysis

11.2.1. Summary

11.2.1.1. The sample extract is injected onto the GC column. The compounds are then identified and quantitated.

11.2.2. Recommended Instrument Conditions

11.2.2.1. GC Conditions

Initial Temperature:	45°C
Initial Hold Time:	4 minutes
Temperature Program:	15°C/minute
Final Temperature:	300°C, hold 2 minutes
Final Time:	23 minutes
Carrier Gas:	Hydrogen
Injection Volume:	1 µL

11.2.3. Sample Analysis Procedure

11.2.3.1. Preliminary Evaluation

The sample extracts may be screened to determine the level of analyte present. If the level of analyte exceeds the working range of the calibration curve, an appropriate dilution is performed to bring the level within the calibration range.

11.2.4. Inject 1 µL of the sample extract or diluted sample into the GC using the same conditions as those used in calibration.

11.2.5. Identification

11.2.5.1. Analytes of interest are identified by comparing retention times with known standards.

11.2.5.2. A single column is used for identification.

11.2.6. Sample Quantification

11.2.6.1. Refer to Section 11 in the 8000B Section of this SOP

11.3. Analytical Documentation

11.3.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.3.2. All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.3.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Refer to the 8000B section of this SOP for identification and quantitation of single component analytes.

13. METHOD PERFORMANCE

13.1. Performance limits for the four replicate initial demonstration of capability are required as referenced under Section 13.1 of the main body of this SOP.

14. POLLUTION PREVENTION

14.1. Refer to section 14 of the 8000B section of this SOP.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure will be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, Method 8015B, Nonhalogenated Organics Using GC/FID, Test Methods for Evaluating Solid Waste, Third Edition, USEPA

17. Miscellaneous (Tables, Appendices, Etc...)

17.1. Reporting limits

17.1.1. The lower reporting limits are shown in Table I1

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

TABLE I1
PHILLIPS 66 REPORTING LIMITS

Compound	Reporting Limits, µg/L
Tetramethylene sulfone (Sulfolane)	50
N-Methyl-2-pyrrolidone	50

Controlled Copy
Copy No. _____
Implementation Date: _____

SOP No. CORP-IP-0003NC
Revision No. 1.3
Revision Date: 09/25/01

STL STANDARD OPERATING PROCEDURE

TITLE: ACID DIGESTION OF AQUEOUS SAMPLES BY SW846 AND MCAWW 200 SERIES METHODS

(Supersedes: Revision 1.2 Dated 03/20/00)

Prepared by: _____ 9/26/01
Date

Reviewed by: _____ 9.26.01
Technology Specialist Date

Reviewed by: _____ 9/26/01
Quality Assurance Manager Date

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Reviewed by: _____ 9-28-01
Laboratory Director Date

Reviewed by: _____
Corporate Technology

Date

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of aqueous samples for the analysis of certain metals by Graphite Furnace Atomic Absorption (GFAA), Flame Atomic Absorption (FLAA), Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP), and Inductively Coupled Plasma-Mass Spectrometry (ICP/MS) using the MCAWW 200 series methods (NPDES) and SW846 Methods 3005A, 3010A, 3020A and 7060A/7740 (RCRA).

1.2. The applicability of each of these preparation protocols to specific analytes is detailed in Tables I and II (Appendix A) and the applicable determinative methods are illustrated by Figures 6 and 7 (Section 17). Additional elements may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.

1.3. This SOP provides procedures applicable to the preparation of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, certain aqueous sludges, wastes, and biological tissues, and leachates/extracts.

1.4. SW-846 Method 3005A is used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by FLAA, ICP and GFAA (antimony only).

1.5. MCAWW Method 200.7 Section 9.4 is used to prepare surface water, domestic and industrial waste samples for total recoverable and dissolved metals determination by ICP.

1.6. SW-846 Method 3010A is used to prepare aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for total metals analysis by FLAA or ICP.

1.7. MCAWW Method 200.7 Section 9.3 is used to prepare surface water and wastes that contain suspended solids for total metals analysis by ICP.

1.8. SW-846 Method 3020A is used to prepare aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for total metals by GFAA, or ICP/MS.

1.9. MCAWW Method 200.0 Section 4.1.3 is used to prepare surface water and wastes that contain suspended solids for total metals analysis by GFAA.

1.10. MCAWW Method 200.0 Section 4.1.4 is used to surface water, domestic and industrial waste samples for total recoverable and dissolved metals determination by GFAA.

1.11. SW-846 Methods 7060A and 7740, respectively, contain the procedure for the preparation of aqueous samples for arsenic and selenium.

1.12. MCAWW Methods 206.2 and 270.2, respectively, contain the procedure for the preparation of aqueous samples for arsenic and selenium.

1.13. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators do require digestion of dissolved samples and this must be clarified before project initiation.

2. SUMMARY OF METHOD

2.1. Method 3005A / Method 200.7 Section 9.4 - Preparation for Total Recoverable or Dissolved Metals Analysis by FLAA or ICP Spectroscopy

2.1.1. A representative aliquot of sample is heated with nitric and hydrochloric acids and substantially reduced in volume. The digestate is filtered (if necessary) and diluted to volume.

2.2. Method 3010A / Method 200.7 Section 9.3 - Preparation for Total Metals Analysis by FLAA or ICP Spectroscopy

2.2.1. A representative aliquot of sample is refluxed with nitric acid. After the digestate has been reduced to a low volume, it is refluxed with hydrochloric acid, filtered (if necessary) and brought up to volume.

2.3. Method 3020A / Method 200.0 Section 4.1.3 - Preparation for Total Metals for Analysis by GFAA Spectroscopy and ICP/MS.

2.3.1. A representative aliquot of sample is refluxed with nitric acid. After the digestate has been reduced to a low volume, it is cooled, filtered (if necessary) and brought up to volume.

2.4. Methods 7060A/206.2 and Methods 7740/270.2 - Preparation for Arsenic/Selenium Analysis by GFAA

2.4.1. A representative aliquot of sample is heated with nitric acid and peroxide until the digestate has been reduced to a low volume. The sample is cooled, filtered (if necessary) and brought up to volume.

2.5. Method 200.0 Section 4.1.4 - Total Recoverable GFAA Preparation (NPDES)

2.5.1. A representative aliquot of sample is heated with nitric acid and until the digestate has been reduced to a low volume. The sample is cooled, filtered (if necessary) and brought up to volume.

3. DEFINITIONS

Additional definitions of terms used in this SOP may be found in the glossary of the QAMP.

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following digestion.

3.4. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include: metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2. The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix D for additional contamination control guidelines.

4.3. Boron and silica from the glassware will migrate into the sample solution during and following sample processing. For critical low level determinations of boron and silica, only quartz and/or plastic labware should be used.

4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents and other matrices may not be digested using these methods if they are not soluble with acids. If physical interferences are present, they should be documented.

4.5. Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.6. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs the sample must be reprepared. Antimony is easily lost by volatilization from hydrochloric acid media.

4.7. Precipitation of silver chloride (AgCl) may occur when chloride ions and high concentrations of silver (i.e., greater than 1 mg/L) are present in the sample.

4.8. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. The following materials are known to be **corrosive:**

hydrochloric acid and nitric acid.

5.3.2. The following materials are known to be **oxidizing agents:**

nitric acid and hydrogen peroxide.

5.3.3. All sample digestions, including cooling of digestates, must be carried out in a fume hood.

5.4. The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood. The analyst should also be aware of the potential for a vigorous reaction.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor.

5.7. Always carry bulk concentrated acid bottles in appropriate impact proof containers.

5.8. Acid/peroxide spills must be neutralized immediately, flushed with water and cleaned up using appropriate spill kits.

5.9. Discard chipped or broken beakers to prevent injury. Chipped glassware may be fire polished as an alternative to disposal.

5.10. Any and all accidents and spills must be reported to the lab supervisor or EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

6.1. Hot plate, digestion block or other adjustable heating source capable of maintaining a temperature of 95°C (± 4).

6.2. Calibrated thermometer that covers a temperature range of 0-200°C.

6.3. Griffin beakers of assorted sizes or equivalent.

6.4. Watch glasses, ribbed or equivalent.

6.5. Whatman No. 4 filter paper or equivalent.

6.6. Funnels or equivalent filtration apparatus.

6.7. Centrifugation equipment (if desired method of removing particulates is centrifugation).

6.8. Graduated cylinder or equivalent capable of measuring 50 mL within 3% accuracy.

6.9. Analytical balance capable of accurately weighing to the nearest 0.01 grams.

6.10. Repipetors or suitable reagent dispensers.

6.11. Calibrated automatic pipettes with corresponding pipette tips or Class A glass volumetric pipettes.

6.12. Class A volumetric flasks.

6.13. pH indicator strips (pH range 0 - 6).

6.14. Plastic digestate storage bottles.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative SOPs.

7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom STL solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Working ICP LCS/MS spike solution: Prepare the ICP LCS/MS working spike solution from custom stock standards to the final concentration listed in Table III. The working

spike must be prepared in a matrix of 5% HNO_3. This acid (5 mL of concentrated HNO_3 per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working ICP LCS solution must be made fresh every three months.

7.4. Working GFAA LCS/MS spike solution: Prepare the GFAA working LCS spike solution by diluting the custom stock solution (7.2) 200x. The working spike solution must be prepared in a matrix of 5% HNO_3. This acid (5 mL of concentrated HNO_3 per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working GFAA LCS solution must be made fresh every three months.

7.5. The TCLP MS working spike solution is provided directly by the vendor, no further standard preparation is necessary.

7.6. The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom STL solution, the individual facility must purchase a solution from the designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.7. Aqueous laboratory control samples (LCSW) and matrix spike samples are prepared as described in Sections 9.5 and 9.6. Refer to Tables III and IV(Appendix A) for details regarding the stock, working standard and final digestate spike concentrations for ICP and GFAA LCS and matrix spike preparations.

7.8. Nitric acid (HNO_3), concentrated, trace metal grade or better.

7.9. Nitric acid, 1:1 - dilute concentrated HNO_3 with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

7.10. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.11. Hydrochloric acid, 1:1 - dilute concentrated HCl with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

7.12. 30% Hydrogen peroxide (H_2O_2), reagent grade.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. For dissolved metals analysis, the samples should be filtered through a 0.45 um filter prior to preservation. Filtration must be done in the field or within 24 hours of collection.

Note: If a sample being analyzed for dissolved metals is found to contain sediment the analyst should contact their supervisor or group leader. The client should be notified of the problem to decide how to treat the sample.

9. QUALITY CONTROL

Table VI (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using any method contained within this SOP the following requirements must be met:

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDL's must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in STL QA Policy QA-005. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the STL reporting limit.

9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance, which should contain all the analytes of interest. The results of the initial demonstration study must be acceptable before analysis of samples may begin. The results of the initial demonstration study may be used to

extend a method for the analysis of other elements provided all acceptance criteria are met.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.1.2.2. Calculations and acceptance criteria for QC check samples are given in the determinative SOPs (CORP-MT-0001, CORP-MT-0003).

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS) are not included in the sample count for determining the size of a preparation batch. MS/MSD are not included in the sample count unless there are multiple sets of MS/MSD per batch. In other words, the first MS/MSD are not counted; all additional MS and MSDs are counted as samples.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Criteria for the acceptance of blanks are contained within the individual analytical method SOP's. If the method blank does not meet the criteria contained within the analytical method SOPs; the blank and all associated samples in the batch must be redigested.

9.4.1. Aqueous method blanks are prepared by taking 50 mL or 50 g of reagent water through the appropriate procedure as described in Section 11.

9.4.2. TCLP method blanks are prepared by taking 50 mL or 50 g of leachate fluid through the appropriate procedure as described in Section 11.

9.5. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried

through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOP's. Corrective action when LCS results fail to meet control limits will be repreparation and reanalysis of the batch. Refer to Section 7.3 and 7.4 for instructions on preparation of the aqueous LCS spike solution.

9.5.1. The aqueous LCS is prepared by spiking a 50 mL aliquot of reagent water with 1.0 mL of the working LCS/MS spike solution (7.3 or 7.4). The LCS is then processed through the appropriate procedure as described in Section 11.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis. If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. Corrective action when MS results fail to meet control limits does not include repreparation of samples unless the results indicate that a spiking error may have occurred.

9.6.1. The aqueous matrix spike sample is prepared by spiking a 50 mL aliquot of a sample with 1.0 mL of the working LCS/MS spike solution (7.3 or 7.4). The matrix spike sample is then processed as described in Section 11.

9.6.2. The TCLP matrix spike sample is prepared by spiking a 50 mL aliquot of a leachate with 0.5 mL of the working TCLP spike solution (7.5). The matrix spike sample is then processed as described in Section 11.

NOTE: The TCLP matrix spike must be added prior to preservation of the leachate.

9.6.3. If insufficient sample is available to process a MS/MSD, then a second LCS must be processed. The LCS pair is then evaluated according to the MS/MSD criteria.

9.7. Quality Assurance Summaries - Certain clients may require specific project or program QC that may supersede the SOP requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

10. CALIBRATION AND STANDARDIZATION

10.1 Hotplate temperature must be verified daily for each hotplate used and must be recorded on either the metals preparation log or in a hotplate temperature logbook. The hotplate temperature should be verified by measuring the temperature of a beaker of reagent water placed on each hotplate.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. All digestion procedures must be carried out in a properly functioning hood.

11.4. All samples are to be checked out of sample control with the chain of custody documentation filled out completely.

11.5. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample.

11.6. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating prep, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge like, organic liquid, lots of sediment etc.) contact the lab supervisor or project manager for further instructions. In some cases it may be more appropriate to process these samples as solids.

11.7. If possible prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and reporting group.

11.8. In most cases, both AA and ICP digests are required on each sample. It is recommended that both aliquots be measured out and processed at the same time.

11.9. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

11.10. The following procedure must be followed for all aqueous sample preparations:

11.10.1. Measure sample pH with pH paper on a separate aliquot of sample.

Note: If the sample pH is > 2 pH units, the client must be notified of the anomaly.

Note: If sample pH has already been verified and documented in sample receipt this step may be omitted.

11.10.2. Mix sample by shaking the container.

11.10.3. Measure and transfer 50 mL or 50 g of the sample into a beaker.

Note: This SOP allows for samples to be weighed instead of measured volumetrically.

11.10.4. Measure two extra aliquots of sample selected for the MS/MSD analysis. Spike each aliquot with the appropriate spiking solutions (7.3-7.5,9.6).

11.10.5. Measure and transfer 50 mL of reagent water into a beaker for the method blank.

11.10.6. Measure and transfer 50 mL of reagent water into a beaker for the LCS and add the appropriate spiking solutions (7.3-7.5,9.6).

11.11. Proceed to the appropriate Section for the desired method as follows:

Method 3005A or Method 200.7 Section 9.4	11.12
Method 3010A or Method 200.7 Section 9.3	11.13
Method 3020A or Method 200.0 Section 4.1.3	11.14
Method 7060A/7740 or Method 206.2/270.2	11.15
Method 200.0 Section 4.1.4	11.16

11.12. **Method 3005A / Method 200.7 Section 9.4 - Preparation for Total Recoverable or Dissolved Metals Analysis by FLAA or ICP (See Figures 1, 6 and 7)**

11.12.1. To the sample beaker, add 1 mL of concentrated HNO_3 and 2.5 mL of concentrated HCl.

11.12.2. Cover with ribbed watch glass.

11.12.3. Heat at 95°C (± 4) until volume is reduced to between 15 and 20 mL.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.

11.12.4. Cool the beaker in a fume hood.

11.12.5. Wash down beaker walls and watch glass with reagent water.

11.12.6. Filter sample, if insoluble materials are present, through Whatman 4 filter paper that has been pre-rinsed with dilute nitric acid.

Note: If any samples in a preparation batch are filtered, the method blank and LCS associated with that batch must also be filtered.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.12.7. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

11.12.8. Adjust the final volume/mass to 50 mL or 50 g with reagent water. The sample is now ready for analysis

11.13. **Method 3010A / Method 200.7 Section 9.3 - Preparation for Total Metals Analysis by FLAA or ICP Spectroscopy (See Figures 2, 6 and 7)**

11.13.1. To the sample beaker, add 3.0mL of concentrated HNO_3.

11.13.2. Cover with ribbed watch glass.

11.13.3. Place beaker on hotplate 95°C (± 4) and evaporate for 4-5 hours or to low volume of 15-20 mL while ensuring that no portion of the bottom of the beaker is allowed to go dry.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.

11.13.4. If necessary, add another 1.5 ml portion of concentrated HNO_3 and re-cover the beaker. Reflux 15 minutes.

11.13.5. Add 5 mL of 1:1 HCl.

11.13.6. Cover and reflux for an additional 15 minutes to dissolve precipitate or residue. Cool in a fume hood.

11.13.7. Wash down beaker walls and watch glass with reagent water.

11.13.8. Filter sample, if insoluble materials are present, through Whatman 4 filter paper.

Note: If any samples in the QC batch are filtered the method blank and LCS associated with that batch must also be filtered.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.13.9. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

11.13.10. Adjust final volume/mass to 50 mL or 50 g with reagent water. The sample is now ready for analysis.

11.14. **Method 3020A / Method 200.0 Section 4.1.3 - Preparation for Total Metals Analysis by GFAA or Total Recoverable Metals by ICPMS (See Figures 3, 6 and 7)**

11.14.1. To the sample beaker, add 1.5 mL of concentrated HNO_3.

11.14.2. Cover with ribbed watch glass.

11.14.3. Place beaker on hotplate 95°C (± 4) and evaporate to low volume of 15-20 mL while ensuring that no portion of the bottom of the beaker is allowed to go dry.

NOTE: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.

11.14.4. If necessary, add another 1.5 mL portion of concentrated HNO_3. Recover, and reflux 15 minutes. Cool the beaker in a fume hood.

11.14.5. Filter sample, if insoluble materials are present, through Whatman 4 filter paper.

Note: If any samples in the QC batch are filtered the method blank and LCS associated with that batch must also be filtered.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.14.6. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

11.14.7. Adjust final volume to 50 mL with reagent water. The sample is now ready for analysis.

11.15. **Method 7060A/7740 and Method 206.2/270.2 - Preparation for Arsenic and Selenium Analysis by GFAA (See Figures 4, 6 and 7)**

11.15.1. To the sample beaker, add 1 mL of 30 % H_2O_2 and 1.0 mL of 1:1 HNO_3.

11.15.2. Heat, until the digestion is complete, at 95°C (± 4) or until the volume has been reduced to 15-20 mL.

11.15.3. Cool beaker.

11.15.4. Filter sample, if insoluble materials are present, through Whatman 4 filter paper that has been pre-rinsed with dilute nitric acid.

Note: If any samples in the QC batch are filtered the method blank and LCS associated with that batch must also be filtered.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.15.5. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

11.15.6. Adjust final volume to 50 mL with reagent water. The sample is now ready for analysis.

11.16. **Method 200.0 Section 4.1.4 - Preparation for Total Recoverable GFAA Analyses. (See Figures 5 and 7)**

11.16.1. To the sample beaker, add 1.0 mL of 1:1 HNO_3.

11.16.2. Heat, until the digestion is complete, at 95°C (± 4) or until the volume has been reduced to 15 - 20 mL.

11.16.3. Cool beaker.

11.16.4. Filter sample, if insoluble materials are present, though Whatman 4 filter paper.

Note: If any samples in the QC batch are filtered the method blank and LCS associated with that batch must also be filtered.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.16.5. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

11.16.6. Adjust final volume to 50 mL with reagent water. The sample is now ready for analysis.

12. DATA ANALYSIS AND CALCULATIONS

Not Applicable.

13. METHOD PERFORMANCE

13.1. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. In general, the matrix spike recovery should fall within +/- 20 % and the matrix spike duplicates should compare within 20% RPD. Method blanks must meet the criteria specified in determinative SOPs. The laboratory control samples should recover within 20% of the true value until in house control limits are established. Acceptance criteria are given in the determinative SOPs.

13.2. The initial demonstration study as detailed in Section 9.1.2 must be acceptable before the analysis of field samples under this SOP may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

13.3. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The facility EH & S coordinator should be contacted if additional information is required.

15.2. Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, Revision 1, July 1992. Methods 3005A, 3010A, 3020A, 7060A and 7740A.

16.2. Methods for the Chemical Analysis of Water and Waste (MCAWW), 1983.

16.3. CORP-MT-0001, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis of Water and Wastes, Method 6010A and Method 200.7.

16.4. CORP-MT-0003, Graphite Furnace Atomic Absorption Spectroscopy, SW846 Method 7000A and MCAWW 200 Series Methods.

16.5. QA-003, STL QC Program.

16.6. QA-004, Rounding and Significant Figures.

16.7. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference methods.

17.1.1. Modifications applicable to SW-846 reference methods.

17.1.1.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

17.1.1.2. The referenced methods as well as Table 3-1 of SW-846 refer to the use of a 100 mL aliquot for digestion. This SOP requires the use of a 50 mL sample size to reduce waste generation. The use of reduced sample volumes are supported in EPA's document "Response to Public

Comments Background Document, Promulgation of the Second Update to SW-846, Third Edition" dated November 3, 1994. This document stated "flexibility to alter digestion volumes is addressed and "allowed" by the table (3-1) and is also inherently allowed by specific digestion methods. Table 3-1 is only to be used as guidance when collecting samples..." EMSL-Ci has also taken the stance that "reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology." Additionally, in written correspondence from the Office of Solid Waste, Olliver Fordham stated " As a "representative sample" can be assured, scaling causes no loss of precision and accuracy in the analysis."

17.1.2. Modifications Specific to Method 3010A

17.1.2.1. Section 11.13.7 of this SOP requires the sample be reduced to a volume of 15 - 20 mL. Section 7.2 of Method 3010A states the volume should be reduced to 3 mL but also states that no portion of the bottom of the beaker should go dry. The SOP required volume is a closer approximation of the volume required to provide an adequate covering of the beaker so as to prevent the loss of critical analytes through volatilization.

17.1.2.2. The scope of 3010A has been expanded to include silver based on comparison studies with 7760A. Method 3010A consistently demonstrated improved accuracy and precision over Method 7760A in the matrices tested (reagent water, surface water and TCLP leachate) up to a concentration of 1 ppm silver.

17.1.3. Modifications Specific to Method 3020A

17.1.3.1. Section 11.14.8 of this SOP requires the sample be reduced to a volume of 15 - 20 mL. Section 7.2 of Method 3010A states the volume should be reduced to 3 mL but also states that no portion of the bottom of the beaker should go dry. The SOP required volume is a closer approximation of the volume required to provide an adequate covering of the beaker so as to prevent the loss of critical analytes through volatilization.

17.1.4. Modifications Specific to Method 7060A/7740

17.1.4.1. Methods 7060A and 7740A incorporate the use of a two step dilution to accommodate the addition of a nickel nitrate modifier. This SOP performs the dilution directly in one step and omits the addition of the modifier. The modifier is added automatically at the instrument by direct injection into the furnace.

17.1.5. Modifications Specific to MCAWW Methods

It was determined by technical review that several of the MCAWW methods were equivalent to the SW-846 methods and therefore were combined under the scope of this SOP as described in Section 11.0. The nature of the differences were deemed insignificant in regards to the amount of acid added and the evaporative volume based on the flexibility allowed by the methods (i.e., add additional acid as required) and the subjective wording of the methods (i.e., evaporate to near dryness vs. an exact volume).

17.2. Modifications from previous SOP

17.2.1. Added ICP/MS to the digestion procedures.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none. Refer to the Appendices for any facility specific information required to support this SOP.

17.4. Documentation and Record Management

The preparation benchsheet should, at a minimum, include the following information:

- Preparation date, analyst name, matrix, prep type (ICP or GFAA), SOP reference.
- Sample ID; initial weight/volume and final weight/volume.
- Standards Documentation (source, lot, prep date, volume added).
- Analyst Signature.

- Reviewer's Signature and date.

Figure 1. Method 3005A / Method 200.7 Section 9.4 (Section 11.12)

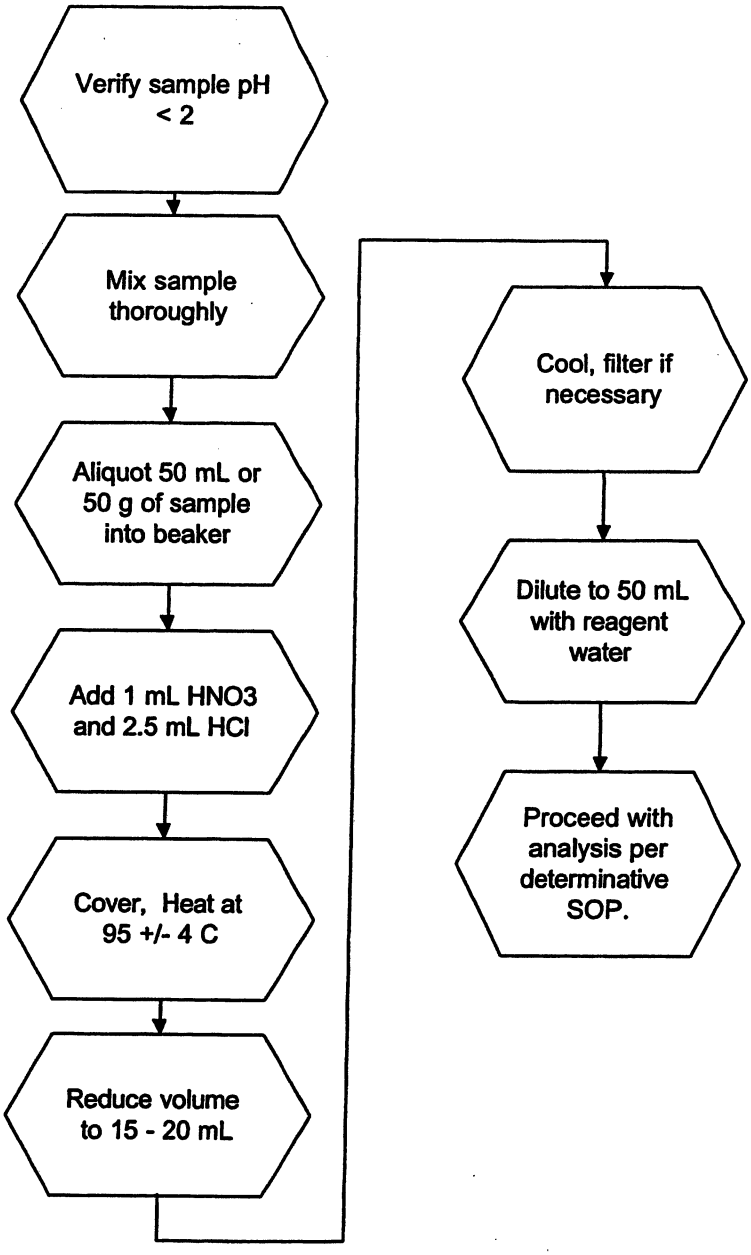


Figure 2. Method 3010A / Method 200.7 Section 9.3 (Section 11.13)

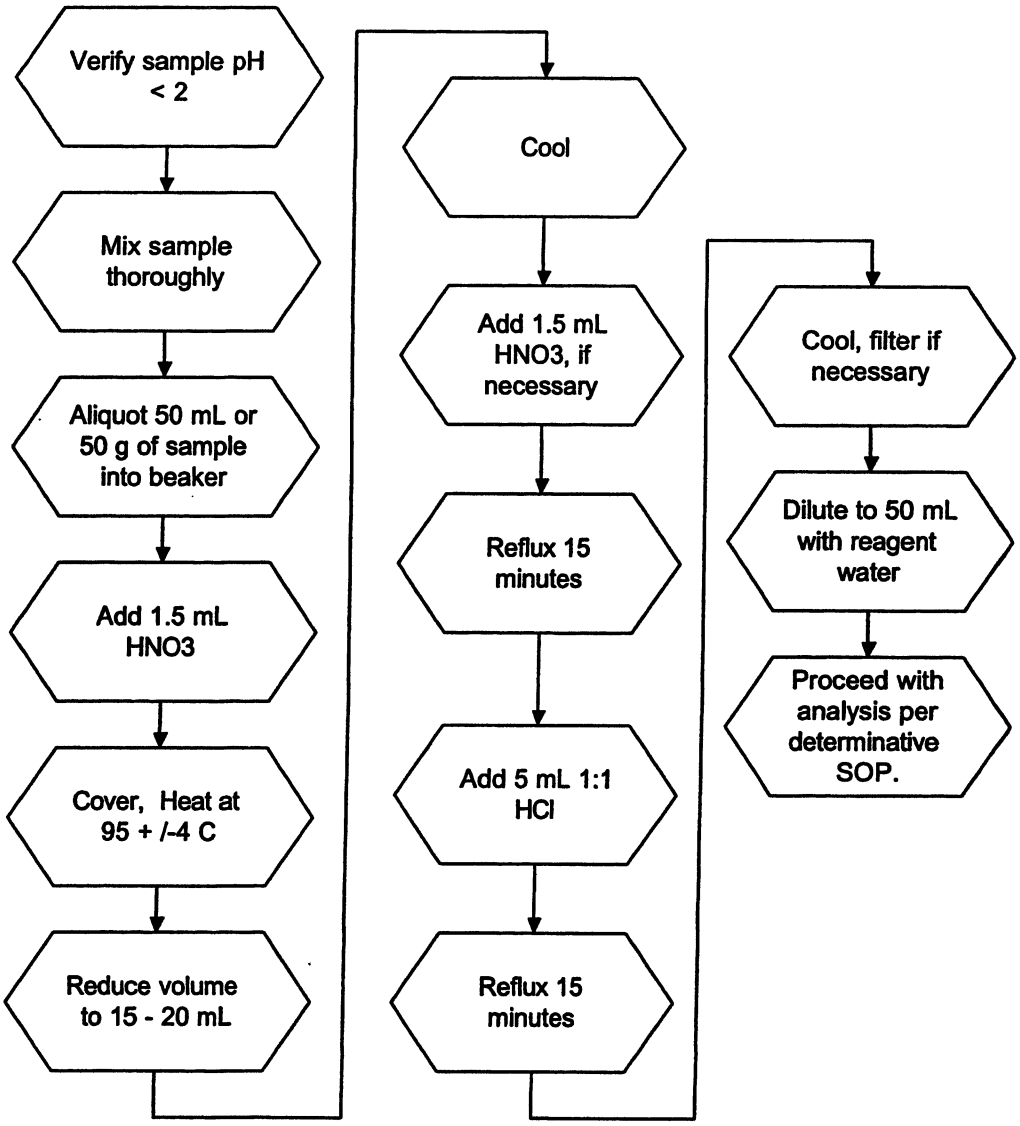


Figure 3. Method 3020A / Method 200.0 Section 4.1.3 (Section 11.14)

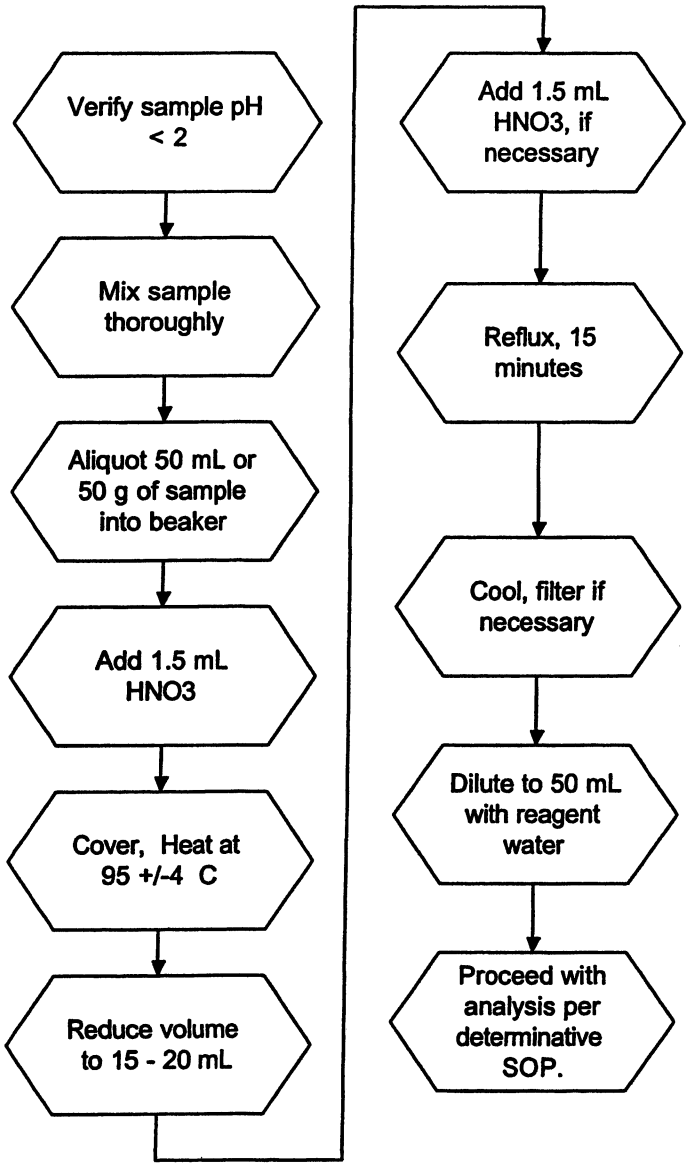


Figure 4. Method 7060A/7740A and Method 206.2/270.2 (Section 11.15)

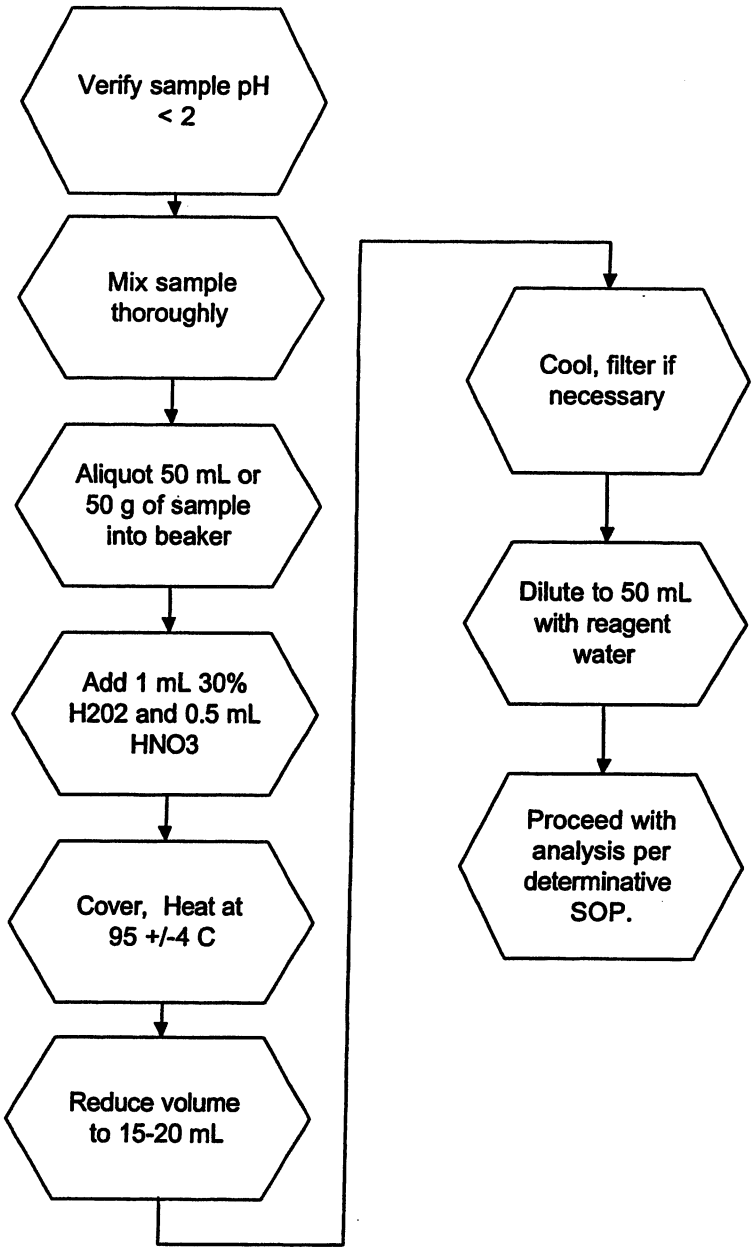


Figure 5. Method 200.0 Section 4.1.4 (Section 11.16)

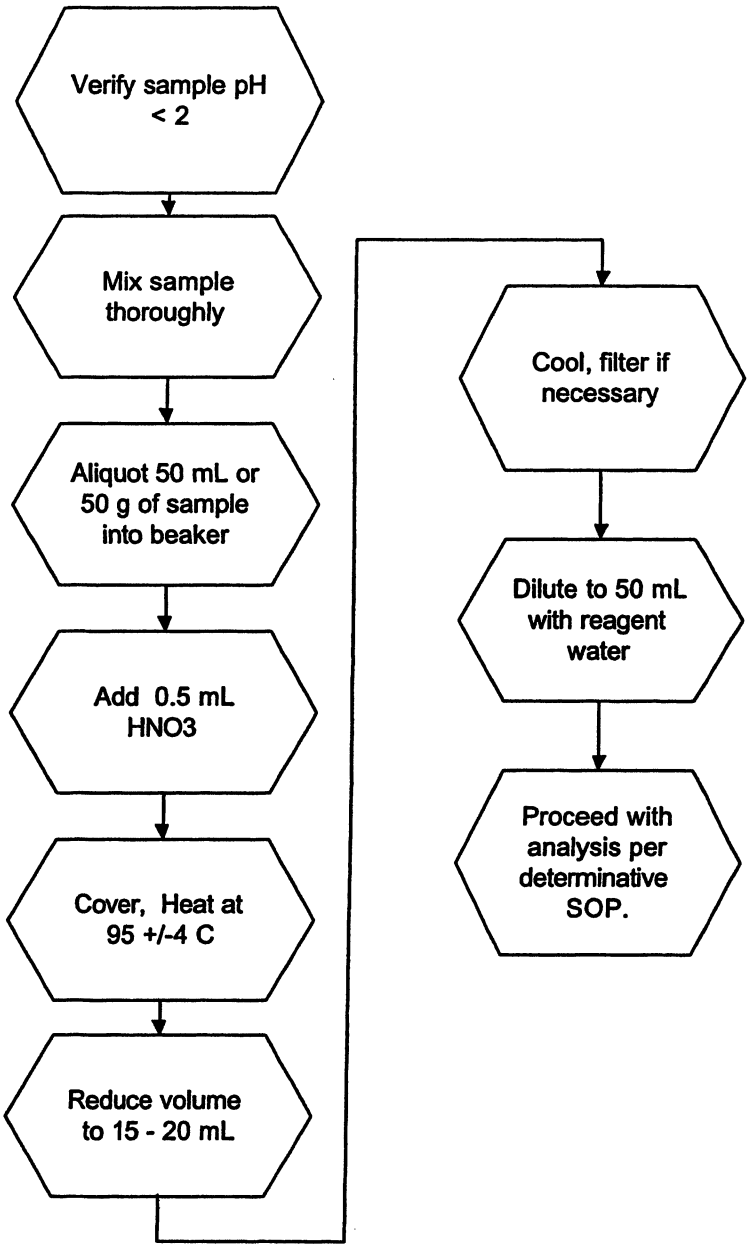
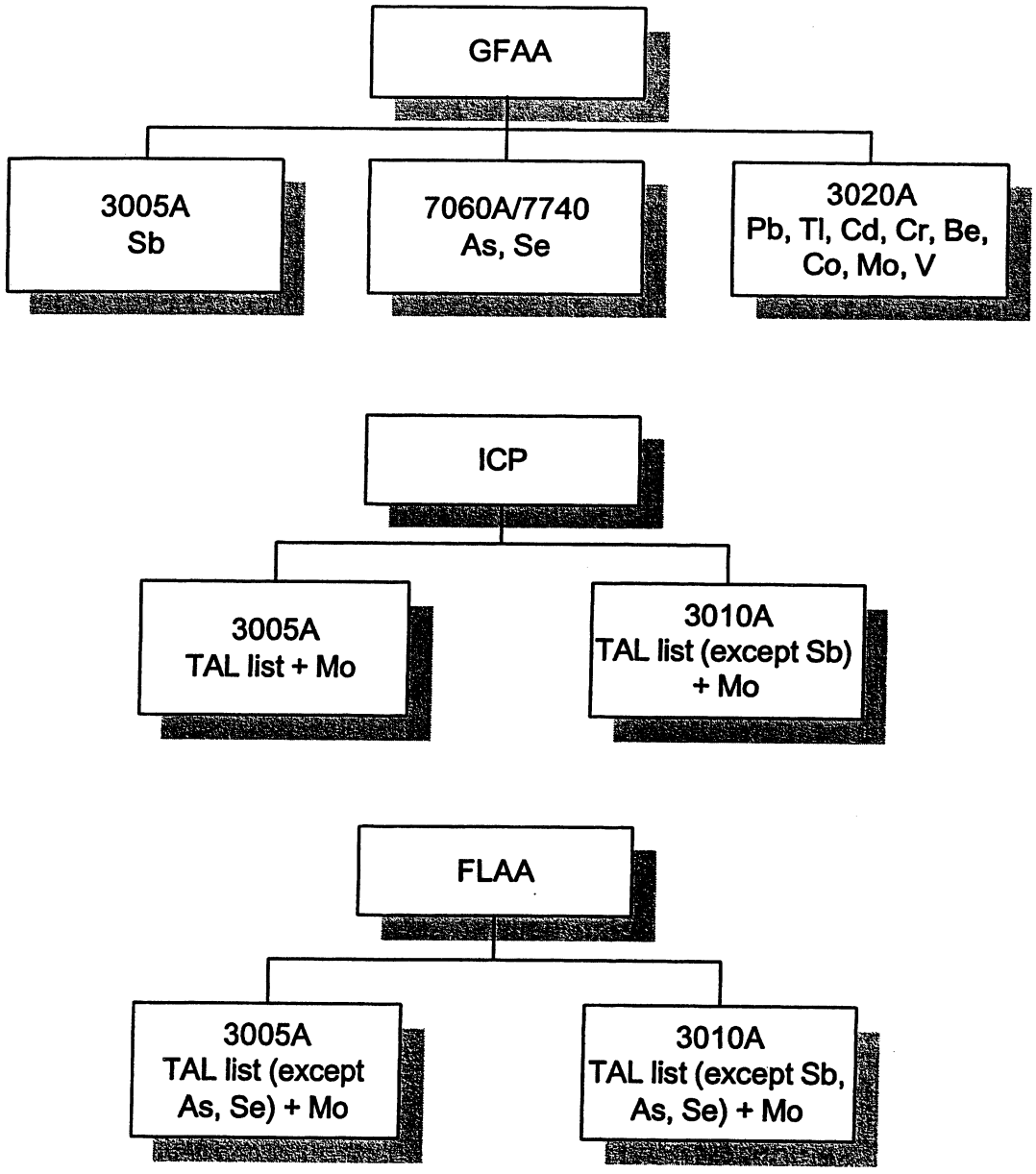
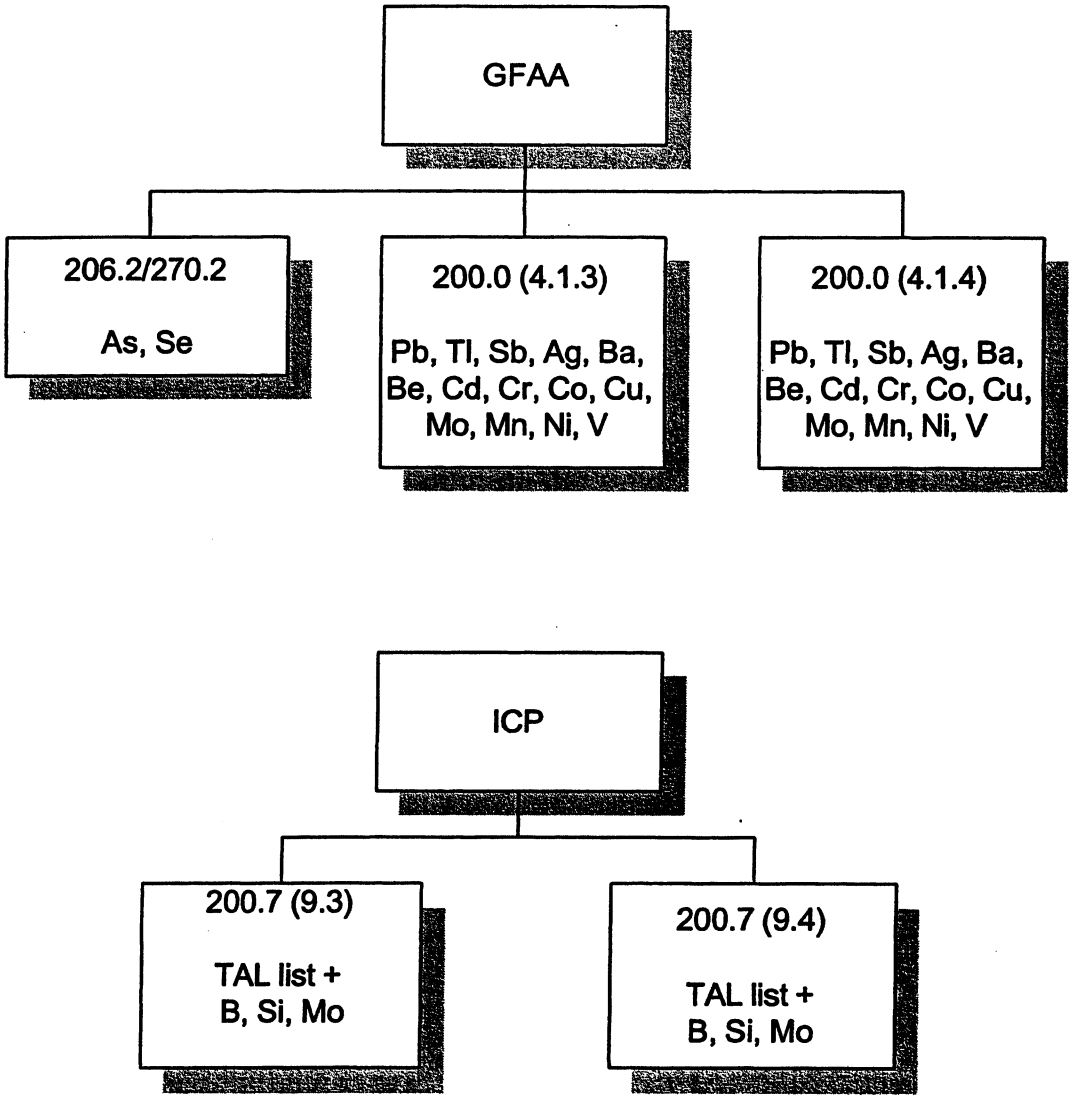


Figure 6. Overview of SW846 Aqueous Preparation Methods by Determinative Method



TAL list: Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn

Figure 7. Overview of MCAWW Aqueous Preparation Methods by Determinative Technique



TAL list: Al, Sb, As, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, Zn

APPENDIX A

TABLES

TABLE I. Approved Preparation Method Analytes - SW846

ELEMENT	Symbol	CAS Number	3005A	3010A	3020A	7060A 7740
Aluminum	Al	7429-90-5	X	X		
Antimony	Sb	7440-36-0	X			
Arsenic	As	7440-38-2	X	X		X
Barium	Ba	7440-39-3	X	X		
Beryllium	Be	7440-41-7	X	X	X	
Cadmium	Cd	7440-43-9	X	X	X	
Calcium	Ca	7440-70-2	X	X		
Chromium	Cr	7440-47-3	X	X	X	
Cobalt	Co	7440-48-4	X	X	X	
Copper	Cu	7440-50-8	X	X		
Iron	Fe	7439-89-6	X	X		
Lead	Pb	7439-92-1	X	X	X	
Magnesium	Mg	7439-95-4	X	X		
Manganese	Mn	7439-96-5	X	X		
Molybdenum	Mo	7439-98-7	X	X	X	
Nickel	Ni	7440-02-0	X	X		
Potassium	K	7440-09-7	X	X		
Selenium	Se	7782-49-2	X	X		X
Silver	Ag	7440-22-4	X	X		
Sodium	Na	7440-23-5	X	X		
Thallium	Tl	7440-28-0	X	X	X	
Vanadium	V	7440-62-2	X	X	X	
Zinc	Zn	7440-66-6	X	X		

X - Designates that the preparation method is approved for an element

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

TABLE II. Approved Preparation Method Analytes - NPDES

ELEMENT	Symbol	CAS Number	200.7 (9.4)	200.7 (9.3)	200.0 (4.1.4)	200.0 (4.1.3)	206.2 270.2
Aluminum	Al	7429-90-5	X	X			
Antimony	Sb	7440-36-0	X	X	X	X	
Arsenic	As	7440-38-2	X	X			X
Boron	B	7440-42-8	X	X			
Barium	Ba	7440-39-3	X	X	X	X	
Beryllium	Be	7440-41-7	X	X	X	X	
Cadmium	Cd	7440-43-9	X	X	X	X	
Calcium	Ca	7440-70-2	X	X			
Chromium	Cr	7440-47-3	X	X	X	X	
Cobalt	Co	7440-48-4	X	X	X	X	
Copper	Cu	7440-50-8	X	X	X	X	
Iron	Fe	7439-89-6	X	X	X	X	
Lead	Pb	7439-92-1	X	X	X	X	
Magnesium	Mg	7439-95-4	X	X			
Manganese	Mn	7439-96-5	X	X	X	X	
Molybdenum	Mo	7439-98-7	X	X	X	X	
Nickel	Ni	7440-02-0	X	X	X	X	
Potassium	K	7440-09-7	X	X			
Selenium	Se	7782-49-2	X	X			X
Silicon	Si	7631-86-9	X	X			
Silver	Ag	7440-22-4	X	X	X	X	
Sodium	Na	7440-23-5	X	X			
Thallium	Tl	7440-28-0	X	X	X	X	
Vanadium	V	7440-62-2	X	X	X	X	
Zinc	Zn	7440-66-6	X	X			

X - Designates that the preparation method is approved for an element

Note: Additional elements may be analyzed following digestion by these protocols provided the method performance criteria specified in Section 13.0 of the SOP are met.

TABLE III. ICP and FLAA Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	Working LCS/MS Standard (mg/L)	Aqueous LCS/ MS Level * (ug/l)
Aluminum	100	2000
Antimony	25	500
Arsenic	100	2000
Barium	100	2000
Beryllium	2.5	50
Cadmium	2.5	50
Calcium	2500	50000
Chromium	10	200
Cobalt	25	500
Copper	12.5	250
Iron	50	1000
Lead	50	500
Magnesium	2500	50000
Manganese	25	500
Molybdenum	50	1000
Nickel	25	500
Phosphorous	500	10000
Potassium	2500	50000
Selenium	100	2000
Silver	2.5	50
Sodium	2500	50000
Thallium	100	2000
Vanadium	25	500
Zinc	25	500
Boron	50	1000
Tin	100	2000
Titanium	50	1000

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 1.0 mL working spike (7.3) to 50 mL of sample.

TABLE IV. GFAA Matrix Spike and Aqueous LCS Spike Levels

ELEMENT	Stock LCS/MS Standard (mg/L)	Working LCS/MS Standard (ug/L)	Aqueous LCS/ MS Level * (ug/l)
Arsenic	400	2000	40
Selenium	400	2000	40
Lead	400	2000	40
Thallium	400	2000	40
Antimony	400	2000	40
Cadmium	40	200	4
Chromium	100	500	10
Silver	50	250	5

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 1.0 mL working spike (7.4) to 50 mL of sample.

TABLE V. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	RL (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)*
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 0.5 mL working spike (7.4) to 50 mL of sample.

TABLE VI. Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: CORP-MT-0001 CORP-MT-0003	Redigest and reanalyze samples associated with the method blank.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: CORP-MT-0001 CORP-MT-0003	Redigest and reanalyze all samples associated with the LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: CORP-MT-0001 CORP-MT-0003	Reprep not required unless preparation error suspected.
Matrix Spike Duplicate	See Matrix Spike	Refer to determinative SOPs: CORP-MT-0001 CORP-MT-0003	See Corrective Action for Matrix Spike.

APPENDIX B

CONTAMINATION CONTROL GUIDELINES

APPENDIX B. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

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Revision No. 3.2
Revision Date: 01/19/01

STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSES, SW-846 METHOD 6010B AND EPA METHOD 200.7

(SUPERSEDES: REVISION 3.1, REVISION DATE 10/04/00)

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1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of trace elements including metals in solution by Inductively Coupled Plasma -Atomic Emission Spectroscopy (ICP-AES) using SW-846 Method 6010B and EPA Method 200.7 . Table I of Appendix A lists the elements appropriate for analysis by Methods 6010B and 200.7. Additional elements may be analyzed under Methods 6010B and 200.7 provided that the method performance criteria presented in Section 13.0 are met.

1.2. ICP analysis provides for the determination of metal concentrations over several orders of magnitude. Detection limits, sensitivity and optimum concentration ranges of the metals will vary with the matrices and instrumentation used. For instance, in comparison to conventional ICP technique, ICP-Trace can achieve detection levels comparable to those determined using the graphite furnace atomic absorption spectroscopy (GFAAS) technique.

1.3. Method 6010B is applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, and TCLP, EP and other leachates/extracts. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators may require digestion of **dissolved samples** and this must be clarified and documented before project initiation. Silver concentrations must be below 2.0 mg/L in aqueous samples and 100 mg/kg in solid matrix samples. Precipitation may occur in samples where silver concentrations exceed these levels and lead to the generation of erroneous data.

1.4. Method 200.7 is applicable to the determination of dissolved, suspended, total recoverable, and total elements in water, waste water, and solid wastes. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples if the criteria in Section 11.1 are met. Silver concentrations must be below 0.1 mg/L in aqueous samples and 50 mg/kg in solid matrix samples.

1.5. State-specific requirements may take precedence over this SOP for drinking water sample analyses.

1.6. The applicable LIMS method codes are QO (6010B), QM (6010B Trace), AS (200.7), JI (200.7 Trace).

2. SUMMARY OF METHOD

2.1. This method describes a technique for the determination of multi elements in solution using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate actions taken. Alternatively, multivariate calibration methods may be chosen for which point selection for background correction is superfluous since whole spectral regions are processed.

2.2. Refer to the appropriate SOPs for details on sample preparation methods.

3. DEFINITIONS

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.

3.4. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

4. INTERFERENCES

4.1. Spectral, physical and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.
- Stray light from the line emission of high concentration elements.

4.1.1. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.1.2. Inter-element correction factors (IECs) are necessary to compensate for spectral overlap. Inter-element interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Inter-element corrections IECs must be applied to the analyte to remove the effects of these unwanted emissions.

4.1.3. Physical interferences are generally considered to be effects associated with sample transport, nebulization and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, dilution of the sample, use of a peristaltic pump, mass flow controller, use of an internal standard and/or use of a high solids nebulizer can reduce the effect.

4.1.4. Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not significant with the ICP technique but if observed can be minimized by buffering the sample, matrix matching or standard addition procedures.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL North Canton associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.3.1. The following materials are known to be **corrosive**:

sulfuric acid, hydrochloric acid, nitric acid and hydrofluoric acid. (NOTE: sulfuric and hydrofluoric acids are used in cleaning the ICP torch and hydrofluoric acid is also commonly used in air toxics preparations.)

5.3.2. The following materials are known to be **oxidizing agents**:

nitric acid and hydrogen peroxide.

5.3.3. The plasma emits strong UV light and is harmful to vision. **NOTE: AVOID looking directly at the plasma.**

5.3.4. The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Metals digestates can be processed outside of a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported **immediately** to a laboratory supervisor.

5.6. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S Manager and laboratory supervisor for guidance.

6. EQUIPMENT AND SUPPLIES

6.1. Inductively Coupled Plasma Atomic Emission Spectrometer equipped with autosampler and background correction.

6.2. Radio Frequency Generator.

6.3. Argon gas supply, welding grade or equivalent.

6.4. Coolflow or appropriate water cooling device.

6.5. Peristaltic Pump.

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.7. Class A volumetric flasks.

6.8. Autosampler tubes.

7. REAGENTS AND STANDARDS

7.1. Intermediate standards are purchased as custom STL North Canton multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles. Intermediate standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem. Expiration dates can be extended provided that the acceptance criteria described in laboratory-specific SOPs are met.

7.2. Working calibration and calibration verification solutions may be used for up to 3 months and must be replaced sooner if verification from an independent source indicates a problem. Standards should be prepared in a matrix of 5% hydrochloric and 5% nitric acids. An exception to this is in the event the Trace ICP is utilized without the internal standard. In this case, the standard acid matrix must be matched to the final preparation matrix as listed in Section 11.9.

7.3. Refer to Tables III, IV, IVA, V and VI (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification, interference correction and spiking solutions.

7.4. Concentrated nitric acid (HNO_3), trace metal grade or better.

7.5. Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.6. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding times for metals are six months from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. Soil samples do not require preservation but must be stored at 4°C ± 2° until the time of preparation .

9. QUALITY CONTROL

Table VII (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using either Method 200.7 or Method 6010B, the following requirements must be met.

Instrument Detection Limit (IDL) - The IDL for each analyte must be determined for each analyte wavelength used least each instrument. The IDL must be determined annually. If the instrument is adjusted in anyway that may affect the IDL, the IDL for that instrument must be redetermined. The IDL shall be determined by multiplying by 3, the standard deviation obtained from the analysis of a standard solution (each analyte in reagent water) at a concentration 3x - 5x the previously determined IDL, with seven consecutive measurements. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure performed between the analysis of separate samples). The result of the IDL determination must be below the STL North Canton reporting limit. The CLP IDL procedure can be used for this method.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte prior to the analysis of any client samples. Refer to STL North Canton SOP NC-QA-0021 for details on MDL analysis and criteria.

9.1.2. Linear Range Verification (LR) - The linear range must be determined on at least an annual basis for each analyte wavelength used on each instrument. The linear range is the concentration above which results cannot be reported without dilution of the sample. The standards used to verify the linear range limit must be analyzed during a routine analytical run and must read within 5% of the expected value.

For the **initial** determination of the upper limit of the linear dynamic range (LDR) for each wavelength, determine the signal responses from a minimum of three to five different concentration standards across the estimated range. One standard should be near the upper limit of the estimated range. The concentration measured at the LDR must be no more than 10% less than the expected level extrapolated from lower standards. If the instrument is adjusted in any way that may affect the LRs, new dynamic ranges must be determined. The LR data must be documented and kept on file.

9.1.3. Background Correction Points - To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on

either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Background correction points must be set prior to determining IECs. Refer to the facility-specific instrument operation SOP and ICP instrument manual for specific procedures to be used in setting background correction points.

9.1.4. Inter-element Corrections (IECs) - ICP interelement correction factors must be determined prior to the analysis of samples and every six months thereafter. If the instrument is adjusted in any way that may affect the IECs, the IECs must be redetermined. When initially determining IECs for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC then the possibility of contamination should be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., GFAA or ICP-MS). Published wavelength tables (e.g. MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IECs. Refer to the facility specific instrument operation SOP and instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which results in a false analyte signal greater than $\pm$ the RL as defined in Tables I, IA or II. To determine IECs, run a single element standard at the established linear range. To calculate an IEC, divide the observed concentration of the analyte by the actual concentration of the "interfering element."

Note: Trace ICP IECs are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IECs will be required on a more frequent basis for the Trace as reflected by the ICSA response. Additional spectral interference is present from easily ionizable elements such as potassium and sodium in axial viewing instruments.

9.1.5. Rinse Time Determination - Rinse times must be determined upon initial set-up of an ICP instrument. To determine the appropriate rinse time for a particular ICP system, the linear range verification standard (see 9.1.3) should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in

environmental samples at that level and an excessive rinse time would be required at the linear range level). Until the required rinse time is established, the method recommends a rinse period of at least 60 seconds between samples and standards. If a memory effect is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented and kept on file, if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

9.2. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20x higher than the blank contamination level).

- If the analyte is a common laboratory contaminant (copper, iron, lead (Trace only) or zinc) the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

- For dissolved metals samples which have not been digested, a CCB result is reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.

9.3. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. Aqueous LCS spike levels are provided in Table III (Appendix A). The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

- If any analyte is outside established control limits the system is out of control and corrective action must occur. Unless in-house control limits are established, a control limit of 80 - 120% recovery must be applied.
- In the event that an MS/MSD analysis is not possible a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.
- In the instance where the LCS recovery is greater than 120% and the sample results are < RL, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the report narrative.**
- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.
- For dissolved metals samples which have not been digested, a CCV result is reported as the LCS. The CCV run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCV.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSDs. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Tables III and VI (Appendix A).

- If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. For both methods 200.7 and 6010B, control limits of 75-125% recovery and 20% RPD or historical acceptance criteria must be

applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported as NC, MSB (i.e., not calculated). If the reporting software does not have the ability to report NC, MSB then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level." Two other narrative notes for metals analyses: Matrix spike/spike duplicate spike recovery/recoveries was/were outside the acceptance limits of some analytes. The acceptable LCS analysis data indicated that the analytical system was operating within control and this condition is most likely due to matrix interference. See the Matrix Spike Report for the affected analytes which will be flagged with N. Matrix spike/spike duplicate relative percent difference (RPD) exceeded the acceptance limits for some analytes. The imprecision may be attributed to sample heterogeneity. See the Matrix Spike Report for the affected analytes, which will be flagged with *.

- If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- For dissolved metals samples which have not been digested, a MS/MSD must be performed per batch of up to 20 samples by spiking two aliquots of the sample at the levels specified in Table III (Appendix A).

9.5. Dilution test – A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. One sample per preparation batch must be processed as a dilution test. The test is performed by running a sample at a 5x (1:4) dilution. Samples identified as field blanks cannot be used for dilution tests. The results of the diluted sample, after correction for dilution, should agree within 10% of the original sample determination when the original sample concentration is greater than 50x the IDL. If the results are not within 10%, the possibility of chemical or physical interference exists.

9.6. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). For analyses conducted under Method 200.7, the ICV result must fall within 5% of the true value for that solution with relative standard deviation <3% from replicate (minimum of two) exposures. For Method 6010B, the ICV must fall

within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the RL from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (See Section 11.10 or 11.13 for required run sequence).

9.7. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV is be a mid-range standard made from a dilution of the calibration standard. The CCV for both methods must fall within 10% of the true value for that solution with relative standard deviation <5% from replicate (minimum of two) exposures. A CCB is analyzed immediately following each CCV. (See Section 11.10 or 11.13 for required run sequence.) The CCB result must fall within +/- RL from zero. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within 10% of the action limit, reanalysis and recalibration are not required before continuation of the run. Sample results may only be reported when bracketed by valid CCV/CCB pairs. If a mid-run CCV or CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. (Refer to Section 11.13 for an illustration of the appropriate rerun sequence).

9.8. Interference Check Analysis (ICSA/ICSAB) - The validity of the interelement correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Table V (Appendix A) for the details of ICSA and ICSAB composition. Custom STL North Canton multielement ICS solutions must be used. All analytes should be spiked into the ICSAB solution, therefore, if a non-routine analyte is required then it should be manually spiked into the ICSAB using a certified ultra high purity single element solution or custom lab-specific mix. If the ICP will display overcorrection as a negative number then the non-routine elements can be controlled from the ICSA as described in section 9.8.3. Elements known to be interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICP runs.

9.8.1. The ICSA and ICSAB solutions must be run at the beginning of the run. (See Section 11.10 or 11.13 for required run sequence.)

9.8.2. The ICSAB results for the interferents must fall within 80 - 120% of the true value. If any ICSAB interferent result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the samples rerun.

9.8.3. ICSA results for the non-interfering elements with reporting limits ≤ 10 ug/L must fall within the STL North Canton guidelines of ± 2x RL from zero. ICSA results for the non-interfering elements with RLs> 10 µg/L must fall within the STL North Canton guidelines of ± 1x RL from zero. If the ICSA results for the non-interfering elements do not fall within +/- 2x RL (RL ≤10) or ± 1xRL (RL>10) from zero the field sample data must be evaluated as follows:

- If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.
- If the affected element was not required then the sample data can be accepted.
- If the interfering elements are not present in the field sample at a concentration which would result in a false positive or negative result greater than +/- 2x RL from zero then the field sample data can be accepted.
- If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than ± 2x RL from zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10x the analyte signal in the ICSA.
- If the data does not meet the above conditions then the IECs must be re-evaluated and corrected if necessary and the affected samples reanalyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually the calculations must be clearly documented on the raw data.

9.9. CRI - To verify linearity near the CRDL for ICP analysis, a CRI standard is run at the beginning of each sample analysis run but not before the ICV. (See Section 11.10 or 11.13 for required run sequence).

Note: The custom STL North Canton CRI mix contains most analytes at a level two times the standard lab reporting limit.

9.10. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Section 11.17 for additional information on when MSA is required as well as Appendix D for specific MSA requirements.

9.11. Quality Assurance/Project Summaries - Certain clients may require project- or program-specific QC which may supersede this SOP requirements. Quality Assurance Summaries (QASs) or equivalent documents providing project-specific requirements should be developed so that project staff clearly understand the special project requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.2. Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures. Flush the system with the calibration blank between each standard or as the manufacturer recommends. The calibration curve must consist of a minimum of a blank and a standard. Refer to the facility-specific instrument SOP or ICP instrument manual for a detailed set up and operation protocols.

10.3. Calibration must be performed daily and each time the instrument is set up. Instrument runs may be continued over periods exceeding 24 hours as long as all calibration verification (CCV) and interference check QC criteria are met. The instrument standardization date and time must be included in the raw data.

10.4. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corresponding corrective actions.

11. PROCEDURE

11.1. For 200.7 analyses, dissolved (preserved) samples must be digested unless it can be documented that the sample meets all of the following criteria:

A. Visibly transparent with a turbidity measurement of 1 NTU or less.

B. Is of one liquid phase and free of particulate or suspended matter following acidification.

C. Is NOT being analyzed for silver.

11.2. A minimum of two exposures for each standard, field sample and QC sample is required. The average of the exposures is reported. For Trace ICP analyses, the results of the sum channel must be used for reporting.

11.3. Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in 9.1.6 it can be demonstrated that a shorter rinse time may be used. Triton-X can be added to the rinse solution to facilitate the rinse process.

11.4. The use of an autosampler for all runs is strongly recommended.

11.5. The use of automated QC checks through the instrument software is highly recommended for all calibration verification samples (ICV,CCV), blanks (ICB,CCB,PB), interference checks (ICSA,ICSAB) and field samples (linear range) to improve the data review process.

11.6. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.7. To facilitate the data review and reporting processes it is strongly recommended that all necessary dilutions be performed before closing out the instrument run.

11.8. The use of an internal standard is recommended on the conventional, non-Trace ICPs as an alternative to using the method of standard additions. This technique is useful in overcoming matrix interferences especially in high solids matrices. However, for conventional ICP techniques, internal standards may not be necessary provided that one of the following is performed to minimize physical interferences: (1) peristaltic pump is used, (2) high solids nebulizer is used, or (3) high solids samples are diluted and reanalyzed.

11.9. The use of an internal standard is **required** on the Trace ICP unless the calibration and QC standards are matrix matched to each digestion procedure used as follows:

Preparation Method	% HNO_3	% HCl
CLP Aqueous	1	5
CLP Soil	5	2.5
SW846 3050	10	10
SW846 3005	2	5
SW846 3010	6	5

The following procedural guidelines must be followed when using an internal standard:

11.9.1. Typically used internal standards are: yttrium or scandium. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)

11.9.2. The internal standard (IS) must be added to every sample and standard at the same concentration. It is recommended that the IS be added to each analytical sample automatically through use of a third pump channel and mixing coil. Internal standards should be added to blanks, samples and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

11.9.3. The concentration of the internal standard should be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.

11.9.4. The internal standard raw intensity counts must be printed on the raw data.

11.9.5. The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte). The instrument automatically adjusts sample results based on comparison of the internal standard intensity in the sample to the internal standard intensity at calibration.

11.9.5.1. If the internal standard counts fall within ±30% of the counts observed in the ICB then the data is acceptable.

11.9.5.2. If the internal standard counts in the field samples are more than ±30% higher than the expected level, the field samples must then be:

(1) Diluted and reanalyzed;

(2) The IS concentrations must be raised; or

(3) A different internal standard must be used.

11.10. The following analytical sequence must be used for Methods 6010B and 200.7:

Instrument Calibration
ICV
ICB
CRI
ICSA
ICSAB
7 samples
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table VII (Appendix A) for Method 6010B and 200.7 quality control criteria.

11.11. Additional quality control analyses are necessary for analysis under the Contract Laboratory Program (CLP). If these are included then CLP, 6010 and 200.7 samples can be included in the same sequence. Refer to CORP-MT-0002NC for details.

11.12. Full method required QC must be available for each wavelength used in determining reported analyte results.

11.13. The following run sequence provides an illustration of a mid-run CCV or CCB failure and the appropriate corrective action run sequence as described in Section 9.7:

Original Run: Instrument Calibration

ICV

ICB

CRI

ICSA

ICSAB

7 samples

CCV1

CCB1

10 samples

CCV2

CCB2

10 samples **

CCV3 *

CCB3 *

10 samples **

CCV4

CCB4

10 samples

CCV5

CCB5

* Failure occurs at CCV3/CCB3

**Samples requiring rerun for affected analytes

Reanalysis:	Recalibrate
	ICV
	ICB
	CRI
	ICSA
	ICSAB
	CCV2
	CCB2
	10 samples
	CCV3
	CCB3
	10 samples
	CCV4
	CCB4

Notes:

Samples between CCV4 and CCV5 do not require reanalysis as they were bracketed by compliant QC samples.

See CORP-MT-0002NC for the appropriate reanalysis sequence if CLP requirements must also be met.

11.14. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.15. All measurements must fall within the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that

exceed the linear range or use an alternate wavelength for which QC data are established. If an interelement correction exists for an analyte which exceeds the linear range, the IEC may be inaccurately applied. Therefore, even if an overrange analyte may not be required to be reported for a sample, if that analyte is a interferent for any requested analyte in that sample, the sample must be diluted. Acid strength must be maintained in the dilution of samples.

11.16. For TCLP samples, full four-point MSA will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

The reporting and regulatory limits for TCLP analyses as well as matrix spike levels are detailed in Table VI (Appendix A). Appendix E provides guidance on performing MSA analyses.

11.17. Any variation in procedure shall be completely documented using instrument run logs, maintenance logs, report narratives, a Nonconformance Memo, or an anomaly report and is approved by a Supervisor/Group Leader and QA Manager. If contractually required, the client shall be notified by the Project Manager.

11.18. Nonconformance documentation shall be filed in the project file.

11.19. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%\,R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.6. The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg/Kg, dry\ weight = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. The dilution test percent difference for each component is calculated as follows:

$$\%Difference = \frac{|I - S|}{I} \times 100$$

Where:
I = Sample result (Instrument reading)
S = Dilution test result (Instrument reading × 5)

12.9. Appropriate factors must be applied to sample values if dilutions are performed.

12.10. Sample results should be reported with up to three significant figures in accordance with the STL North Canton significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Refer to Tables I, IA & II in Appendix A for the list of Method 6010B and 200.7 analytes as well as additional analytes that may be analyzed using this SOP.

13.3. Method performance is determined by the analysis of MS and MSD samples as well as method blanks and laboratory control samples. The MS or MSD recovery should fall within +/- 25 % and the MS/MSD should compare within 20% RPD or within the laboratory's historical acceptance limits. These criteria apply to analyte concentrations

greater than or equal to 10xIDL. Method blanks must meet the criteria specified in Section 9.2. The laboratory control samples should recover within 20% of the true value or within the laboratory's historical acceptance limits.

13.4. Training Qualification:

The group/team leader or the supervisor has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed of according to the facility hazardous waste procedures and per the local, state, and federal regulations. The Environmental Health and Safety Director should be contacted, if additional information is required.

15.2. Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

16. REFERENCES

16.1. 40 CFR Part 136, Appendix B, 7-5-95, Determination of Method Detection Limits.

16.2. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, Revision 2, December 1996. Method 6010B.

16.3. Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry, Revision 4.4, May 1994. Method 200.7.

16.4. CORP-MT-0002NC, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Method 200.7 & CLP-M, SOW ILMO3.0 and ILMO4.0).

16.5. QA-003, STL North Canton QC Program.

16.6. QA-004, Rounding and Significant Figures.

16.7. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference method

17.1.1. Modifications/interpretations from both Methods 6010B and 200.7.

17.1.1.1. STL North Canton laboratories use mixed calibration standard solutions purchased from approved vendors instead of using individual mixes prepared in house as recommended by the subject methods.

17.1.1.2. Methods 200.7 and 6010B state that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. In determining IECs, because of lack of definition clarification for "concentration range around the calibration blank," STL North Canton has adopted the procedure in EPA CLP ILMO4.0.

17.1.1.3. Section 8.5 of Method 6010B and Section 9.5 of Method 200.7 recommend that whenever a new or unusual matrix is encountered, a series of tests be performed prior to reporting concentration data for that analyte. The dilution test helps determine if a chemical or physical interference exists. Because STL North Canton laboratories receive no prior information from clients regarding when to expect a new or unusual matrix, STL North Canton may select to perform a dilution test on one sample in each prep batch. According to the method, the post digestion spike (PDS) determines any potential matrix interferences. At STL North Canton labs, matrix interference is determined by evaluating data for the LCS and MS/MSD. STL North Canton requires documented, clear guidance when a new or unusual matrix will be received for a project and a request to perform the dilution test or PDS on a client-identified sample.

17.1.2. Modifications from Method 200.7.

17.1.2.1. Method 200.7 defines the IDL as the concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of the calibration blank signal at the

same wavelength. STL North Canton labs utilize the CLP IDL definition as defined in Section 9.1.1 of this SOP.

17.1.2.2. The calibration blank is prepared in an acid matrix of 5% HNO_3/5% HCl instead of the specified 2% HNO_3/10% HCl matrix as the former matrix provides for improved performance relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied.

17.1.2.3. Method section 9.3.4 specifies that "Analysis of the IPC (ICSA/AB) solution immediately following calibration must verify that the instrument is within ± 5% of calibration with a relative standard deviation <3% from replicate integrations ≥ 4." STL North Canton uses a minimum of two exposures.

17.1.2.4. Section 7.12 of 200.7 indicates that the QCS (ICV) should be prepared at a concentration near 1 ppm. The ICV specified in this SOP accommodates the 1 ppm criteria for the majority of analytes. For the remaining analytes, this SOP specifies ICV concentrations which are appropriate to the range of calibration. The intent of the ICV, verification of calibration standard accuracy, is independent of the ICV concentration used.

17.1.2.5. The ICS criteria applied by this SOP differ from those stated in the method. Method 200.7 section 10.4 states that results should fall within the established control limits of 3 times the standard deviation of the calibration blank for that analyte. The control limits listed in this SOP are those applicable to the EPA designed solution.

17.1.2.6. Method 200.7 section 9.3.4 states the CCB should be less than the IDL, but > the lower 3-sigma control limit of the calibration blank. The intent of this requirement is to ensure that the calibration is not drifting at the low end. STL North Canton has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. SOP section 9.7 provides the detailed corrective action criteria that must be followed.

17.1.3. Modifications from Method 6010B.

17.1.3.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the

reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

17.1.3.2. Method 6010B section 8.6.1.3 states that the results of the calibration blank are to agree within 3x the IDL. If not, repeat the analysis two or more times and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. The intent of this requirement is to ensure that the calibration is not drifting at the low end. STL North Canton has adopted an absolute control limit of +/- RL from zero for calibration blank criteria. See SOP Section 9.7 for a detailed description of the required corrective action procedures.

17.2. Modifications from previous SOP

Refer to revision 2 of this SOP.

17.3. Facility-Specific SOPs

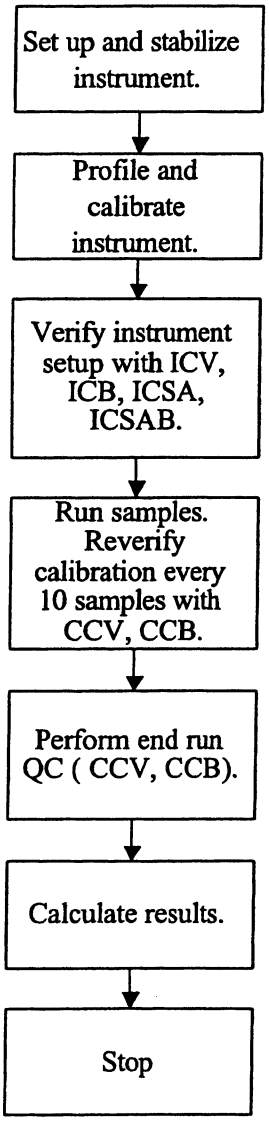
Each facility shall review and revise as appropriate this SOP to reflect any facility-specific requirements. If no facility-specific amendments are required, the SOP can be adopted as is.

17.4. Documentation and Record Management

The following documentation comprises a complete ICP raw data package:

- Raw data (direct instrument printout).
- Relevant sample preparation benchsheets.
- Run log printout from instrument software where this option is available (TJA) or manually generated run log (i.e., Ward WSL printout).
- Data review checklist - See Appendix B.
- Standards documentation (including prep and expiration dates, source, and lot #).
- Nonconformance/anomaly documentation (if applicable).

17.5. Flow Diagram



APPENDIX A

TABLES

TABLE I. Method 200.7 and 6010B Target Analyte List

ELEMENT	Symbol	CAS #	6010B analyte	200.7 analyte	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Aluminum	Al	7429-90-5	X	X	200	20
Antimony	Sb	7440-36-0	X	X	60	6
Arsenic	As	7440-38-2	X	X	300	30
Barium	Ba	7440-39-3	X	X	200	20
Beryllium	Be	7440-41-7	X	X	5.0	0.5
Boron	B	7440-42-8		X	200	20
Cadmium	Cd	7440-43-9	X	X	5.0	0.5
Calcium	Ca	7440-70-2	X	X	5000	500
Chromium	Cr	7440-47-3	X	X	10	1
Cobalt	Co	7440-48-4	X	X	50	5
Copper	Cu	7440-50-8	X	X	25	2.5
Iron	Fe	7439-89-6	X	X	100	10
Lead	Pb	7439-92-1	X	X	100	10
Magnesium	Mg	7439-95-4	X	X	5000	500
Manganese	Mn	7439-96-5	X	X	15	1.5
Molybdenum	Mo	7439-98-7	X	X	40	4
Nickel	Ni	7440-02-0	X	X	40	4
Potassium	K	7440-09-7	X	X	5000	500
Selenium	Se	7782-49-2	X	X	250	25
Silver	Ag	7440-22-4	X	X	10	1
Sodium	Na	7440-23-5	X	X	5000	500
Thallium	Tl	7440-28-0	X	X	2000	200
Vanadium	V	7440-62-2	X	X	50	5
Zinc	Zn	7440-66-6	X	X	20	2

TABLE IA. Method 200.7 and 6010B Trace ICP Target Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Arsenic	As	7440-38-2	10	1.0
Lead	Pb	7439-92-1	3.0	0.3
Selenium	Se	7782-49-2	5.0	0.5
Thallium	Tl	7440-28-0	10	1.0
Antimony	Sb	7440-36-0	10	1.0
Cadmium	Cd	7440-43-9	2.0	0.2
Silver	Ag	7440-22-4	5.0	0.5
Chromium	Cr	7440-47-3	5.0	0.5

TABLE II. Non-Routine Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Tin	Sn	7440-31-5	100	10
Titanium	Ti	7440-32-6	50	5

TABLE III. Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	LCS Level (ug/L)	Matrix Spike Level (ug/L)
Aluminum	2000	2000
Antimony	500	500
Arsenic	2000	2000
Barium	2000	2000
Beryllium	50	50
Cadmium	50	50
Calcium	50000	50000
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1000	1000
Lead	500	500
Magnesium	50000	50000
Manganese	500	500
Molybdenum	1000	1000
Nickel	500	500
Potassium	50000	50000
Selenium	2000	2000
Silver	50	50
Sodium	50000	50000
Thallium	2000	2000
Vanadium	500	500
Zinc	500	500
Boron	1000	1000
Tin	2000	2000
Titanium	1000	1000

TABLE IV. ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	100000	200	25000	50000
Antimony	10000	60	1000	5000
Arsenic	10000	300	1000	5000
Barium	10000	200	1000	5000
Beryllium	10000	5	1000	5000
Cadmium	10000	5	1000	5000
Calcium	100000	5000	25000	50000
Chromium	10000	10	1000	5000
Cobalt	10000	50	1000	5000
Copper	10000	25	1000	5000
Iron	100000	100	25000	50000
Lead	10000	100	1000	5000
Magnesium	100000	5000	25000	50000
Manganese	10000	15	1000	5000
Molybdenum	10000	40	1000	5000
Nickel	10000	40	1000	5000
Potassium	100000	5000	25000	50000
Selenium	10000	250	1000	5000
Silver	2000	10	500	1000
Sodium	100000	5000	25000	50000
Thallium	20000	2000	5000	10000
Vanadium	10000	50	1000	5000
Zinc	10000	20	1000	5000
Boron	10000	200	1000	5000
Tin	10000	100	1000	5000
Titanium	10000	50	1000	5000

TABLE IVA. Trace ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	50000	200	12500	25000
Antimony	1000	10	250	500
Arsenic	1000	10	250	500
Barium	4000	10	1000	2000
Beryllium	4000	5	1000	2000
Cadmium	1000	2	250	500
Calcium	100000	5000	25000	50000
Chromium	4000	5	1000	2000
Cobalt	4000	50	1000	2000
Copper	4000	25	1000	2000
Iron	50000	100	12500	25000
Lead	1000	3	250	500
Magnesium	100000	5000	25000	50000
Manganese	4000	15	1000	2000
Molybdenum	4000	40	1000	2000
Nickel	4000	40	1000	2000
Potassium	100000	5000	25000	50000
Selenium	1000	5	250	500
Silver	2000	5	500	1000
Sodium	100000	5000	25000	50000
Thallium	2000	10	500	1000
Vanadium	4000	50	1000	2000
Zinc	4000	20	1000	2000

TABLE V. Interference Check Sample Concentrations*

Element	ICSA (ug/L)	ICSAB (ug/L)
Aluminum	500000	500000
Antimony	-	1000
Arsenic	-	1000
Barium	-	500
Beryllium	-	500
Cadmium	-	1000
Calcium	500000	500000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200000	200000
Lead	-	1000
Magnesium	500000	500000
Manganese	-	500
Molybdenum	-	1000
Nickel	-	1000
Potassium	-	10000
Selenium	-	1000
Silver	-	1000
Sodium	-	10000
Thallium	-	10000**
Vanadium	-	500
Zinc	-	1000
Tin	-	1000
Boron		1000
Titanium		1000

* Custom STL North Canton solutions contain analytes common to all STL North Canton facilities. Non-routine elements not listed above should be spiked into the ICSAB at 1000 ug/L.

** Thallium level for Trace ICP should be at 1000 ug/L.

TABLE VI. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	Reporting Level (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

TABLE VII. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Two-point Initial Calibration	Beginning of every analytical run, every 24 hours, whenever instrument is modified, or CCV criterion is not met		Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
ICV	Beginning of every analytical run.	Method 200.7: 95 - 105 % recovery. RSD dupl. exp < 3% Method 6010B: 90 - 110 % recovery. RSD dupl. exp < 5%	Terminate analysis; Correct the problem; Recalibrate.
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate.
CCV	Every 10 samples and at the end of the run.	Method 200.7 & 6010B: 90 - 110 % recovery. RSD dupl. exp < 5%	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
ICSA	Beginning of every run	See Section 9.8.3	See Section 9.8.3
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.8.2.

* See Sections 11.10 and 11.13 for exact run sequence to be followed.

TABLE VII. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Dilution Test	One per prep batch.	For samples > 50x IDL, dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference per client request.
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Common lab contaminants may be accepted up to 2x the RL after consultation with the client (See 9.2). Sample results greater than 10x the blank concentration are acceptable. Samples for which the contaminant is < RL may not require redigestion or reanalysis (see Section 9.2).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.2 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits. Samples for which the contaminant is < RL and the LCS results are > 120% may not require redigestion or reanalysis (see Section 9.3)	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.

TABLE VII. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Matrix Spike	One per sample preparation batch of up to 20 samples.	75 - 125 % recovery or in-house control limits. For TCLP See Section 11.17.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. For TCLP see Section 11.17.
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery; RPD ≤ 20% .	See Corrective Action for Matrix Spike.

APPENDIX B

STL NORTH CANTON ICP DATA REVIEW CHECKLIST

(EXAMPLE)

SCAL STD_____ ICSA STD_____ SCAL2 STD_____ ICSAB STD_____

CRI STD_____ ICV STD_____ CLP CRI STD_____ CCV STD_____

(Example)

STL North Canton ICP Data Review Checklist

Run/Project Information:

Run Date: _____ **Analyst:**_____ **Instrument:**_____

Prep Batches Run:_____

Cu/MnRatio:_____

Circle Methods used: **6010B / 200.7: CORP-MT-0001 Rev 2 CLP ILM03.0: CORP-MT-0002 Rev 1**

6010A/200.7: CORP-MT-0001NC, Rev 2

Review Items

A. Calibration/Instrument Run QC	**Yes**	**No**	**N/A**	**2nd Level**
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits ? (*ICV:* 6010B, CLP = 90 - 110%, 200.7 = 95 -105%) *(CCV: 90 - 110%)*				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP) ?				
4. High Std. (HCAL) reanalyzed before samples and recovered within QC limits ? (6010A/200.7 95-105%, CLP- N/A)				
5. CRI run and recovered within QC limits ? (+/- 50% for non-CLP - 6010A only)				
6. ICSA/ICSAB run at required frequency and within SOP limits ?				
B. Sample Results				
1. Were samples with concentrations > the linear range for any parameter diluted and reanalyzed ?				
2. All reported results bracketed by in control QC ?				
3. Sample analyses done within holding time ?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				

3. MS/MSD run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits ?				
5. Serial dilution done per prep batch (or per SDG for CLP) ?				
6. Post digest spike analyzed if required (CLP only) ?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/LR/IEC data on file ?				
3. Calculations checked for error ?				
4. Transcriptions checked for error ?				
5. All client/project specific requirements met ?				
6. Date/time of analysis verified as correct ?				

Level I Analyst:_____ **Date:**_____ **Time:**_____

Comments:_____

Level II Reviewer:_____ **Date:**_____ **Time:**_____

APPENDIX C

CROSS REFERENCE OF TERMS USED IN METHODS 6010B, 200.7, AND BY STL NORTH CANTON

CROSS REFERENCE OF TERMS COMMONLY USED IN METHODS EPA 200.7, SW6010B, AND STL NORTH CANTON INC. SOP

EPA 200.7	SW6010B	STL North Canton Inc. SOP
Calibration blank (CB)	Calibration blank	Initial and continuing calibration blanks (ICB/CCB)
Dilution test	Dilution test	Dilution Test
Instrument detection limit (IDL)	Instrument detection limit (IDL)	Instrument detection limit (IDL)
Instrument performance check (IPC)	Continuing calibration verification (CCV)	Continuing calibration verification (CCV)
Internal standard	Internal standard	Internal standard (IS)
Laboratory duplicates	n/a	n/a
Laboratory fortified blank (LFB)	n/a	Laboratory control sample (LCS)
Laboratory fortified sample matrix (LFM)	Matrix spike and matrix spike duplicate (MS/MSD)	Matrix spike and matrix spike duplicate (MS/MSD)
Laboratory reagent blank (LRB)	Method blank	Method or Prep blank (MB)
Linear dynamic range (LDR)	Linear dynamic range (LDR)	Linear dynamic range (LDR)
Method detection limit (MDL)	Method detection limit (MDL)	Method detection limit (MDL)
Quality control sample (QCS)	Check standard or Initial calibration verification (ICV)	Initial calibration verification (ICV)
Spectral interference check solution (SIC)	Interference check solution (ICS)	Interference check solution (ICSA/ICSAB)

APPENDIX D

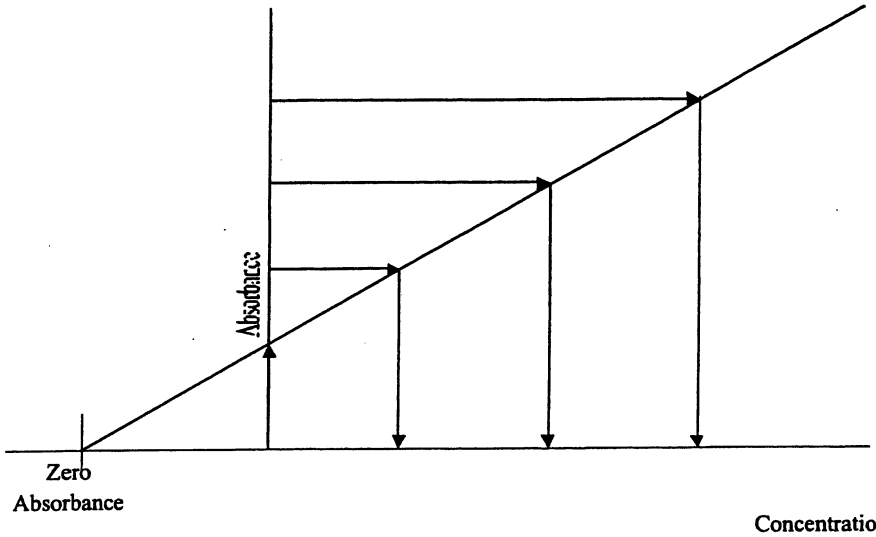
MSA GUIDANCE

Appendix D. MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown.



Conc. of Sample	Addn 0 No Addn	Addn 1 Addn of 50% of Expected Amount	Addn 2 Addn of 100% of Expected Amount	Addn 3 Addn of 150% of Expected Amount

For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.

- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX E

TROUBLESHOOTING GUIDE

APPENDIX E. TROUBLESHOOTING GUIDE

Problem	Possible Cause/ Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer Clean or replace mixing chamber Lower Torch
Instrument Drift	RF not cooling properly Vacuum level is too low Replace torch (Crack) Clean or replace nebulizer (blockage) Check room temperature (changing) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Adjust sample carrier gas Reprofile Horizontal Mirror Replace PA tube
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage(back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Cu/Mn Ratio Outside Limits or Low Sensitivity	Plasma conditions changed Clean nebulizer, torch or spray chamber Replace tubing (clogged) Realign torch Check IECs
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

APPENDIX F

CONTAMINATION CONTROL GUIDELINES

APPENDIX F. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipet tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Latex gloves contain over 500 ppb of zinc.

APPENDIX G

PREVENTIVE MAINTENANCE

APPENDIX G. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Daily	Change sample pump tubing and pump windings Check argon gas supply level Check rinse solution and fill if needed Check waste containers and empty if needed Check sample capillary tubing is clean and in good condition Check droplet size to verify nebulizer is not clogged. Check sample flow for cross flow nebulizer Check Cu/Mn ratio-should be 30% of value at date that IECs were performed Check pressure for vacuum systems
As Needed	Clean plasma torch assembly to remove accumulated deposits Clean nebulizer and drain chamber; keep free-flowing to maintain optimum performance Replace peristaltic pump tubing, sample capillary tubing and autosampler sipper probe
Weekly	Apply silicon spray on autosampler tracks Check water level in coolflow
Monthly	Clean air filters on back of power unit to remove dust Check D mirror for air instruments
Bi-yearly	Change oil for vacuum systems Replace coolant water filter (may require more or less frequently depending on quality of cooling water)

APPENDIX H

ICP OPERATING INSTRUCTIONS

ICP Analysis (TJA 61E)

(Example)

1. SETUP

a. Plasma Control Panel (enter)

b. (F1)-Startup

c. (F9)-Continue

d. (F2)-Levels

1. Change auxiliary gas to low – use space bar to toggle
2. Change nebulizer gas flow to 0.5 L/min.
3. Change pump rate to 130
4. Esc
5. Allow instrument to warm up approximately 30 minutes.

2. DEVELOPMENT

a. Methods (enter)

b. Enter method name

c. (F3)-Method Info.

d. Change file name

e. (F9)-Done

f. (F9)-Done/Keep

3. OPERATION

a. Analysis (enter)

b. (F5)-Profile

1. (F3)-Automatic

2. (F1)-Run

3. If peak position is greater than +/- .05 units from the center peak position, you must adjust the profile. If it is within +/- .05 units, press (F9)-done.

4. To adjust select (F1)-CalcSS and enter current vernier position. (enter)

5. Adjust to new vernier position(F9)-done

6. Rerun profile until peak position is +/- .05 units.

7. (F9)-Done

c. Autosampler (F9)

1. Enter method name (enter)

2. Enter autosampler table name (enter)

3. (F1)-Run

STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS SAMPLES BY COLD VAPOR ATOMIC ABSORPTION, SW846 7470A AND MCAWW 245.1

(SUPERSEDES: REVISION 2.2, DATED 02/05/01)

Prepared by:		5/21/01 Date
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Approved by:	Corporate Technology	5/17/01 Date

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7470A and MCAWW Method 245.1.

1.2. The associated LIMs method codes are BL (Method 245.1) and O8 (Method 7470A).

1.3. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants, potassium permanganate and potassium persulfate, has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation and volume of sample used.

1.4. Method 7470A is applicable to the preparation and analysis of mercury in ground water, aqueous samples, TCLP, and other leachates/extracts. Certain solid and sludge type wastes may also be analyzed, however Method 7471A (see CORP-MT-0007NC) is usually the method of choice. All matrices require sample preparation prior to analysis.

1.5. Method 245.1 is applicable to the determination of mercury in drinking, surface and saline waters and domestic and industrial wastes. All matrices require sample preparation prior to analysis.

1.6. The STL North Canton reporting limit for mercury in aqueous matrices is 0.0002 mg/L except for TCLP or SPLP leachates for which the reporting limit is 0.002 mg/L.

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in sulfuric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the

sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3. DEFINITIONS

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following digestion.

4. INTERFERENCES

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1. Potassium permanganate, which is used to breakdown organic mercury compounds also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.2. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.3. Chlorides can cause a positive interference. Sea waters, brines and industrial effluents high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample headspace before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.4. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This

is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.5. Samples containing high concentrations of oxidizing organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

4.6. The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL North Canton associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. The following materials are known to be **corrosive:**

hydrochloric acid, nitric acid and sulfuric acid.

5.3.2. The following materials are known to be **oxidizing agents**:

nitric acid, potassium permanganate, potassium persulfate and magnesium perchlorate.

5.3.3. Mercury is a highly toxic element that must be handled with care. The analyst must be aware of the handling and clean up techniques before working with mercury.

Since mercury vapor is toxic, precaution must be taken to avoid its inhalation, ingestion or absorption through skin. All lines should be checked for leakage and the mercury vapor must be vented into a hood or passed through a mercury absorbing media such as:

5.3.3.1. Equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4, or

5.3.3.2. Iodine, 0.25%, in a 3% KI solution.

5.3.4. Magnesium sulfate is known to be a reproductive toxin (mutagen).

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported **immediately** to a laboratory supervisor.

5.6. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.

5.7. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders are located outside the laboratory and the gas led to the instrument through approved lines.

5.8. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

6. EQUIPMENT AND SUPPLIES

6.1. Temperature controlled water bath (capable of maintaining a temperature of 90-95 °C) or autoclave that is able to obtain conditions of 15 lbs., 120 °C for 15 minutes.

6.2. Atomic Absorption Spectrophotometer equipped with:

6.2.1. Absorption Cell with quartz end windows perpendicular to the longitudinal axis. Dimensions of the cell must result in sufficient sensitivity to meet the SOP defined reporting limit. The quartz windows must be maintained to provide accurate

measurements. Any scratches or fingerprints can alter the absorption of UV radiation.

6.2.2. Mercury specific hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL).

6.2.3. Peristaltic pump, which can deliver 1 L/min, air.

6.2.4. Flowmeter capable of measuring an airflow of 1 L/min.

6.2.5. Recorder or Printer.

6.2.6. Aeration Tubing: A straight glass frit having a course porosity and Tygon tubing is used for the transfer of mercury vapor from the sample bottle to the absorption cell and return.

6.2.7. Drying device (a drying tube containing magnesium perchlorate or magnesium sulfate and/or a lamp with a 60 W bulb) to prevent condensation in cell. The lamp is positioned to shine on the absorption cell maintaining the air temperature in the cell about 10 °C above room temperature. Other drying devices that achieve the same purpose are also acceptable (i.e., Gortex filter).

NOTE: Instruments designed specifically for the measurement of mercury using the cold vapor technique may be substituted for the atomic absorption spectrophotometer.

6.3. BOD bottles or equivalent.

6.4. Nitrogen or argon gas supply, welding grade, or equivalent.

6.5. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.6. Class A volumetric flasks.

6.7. Thermometer (capable of accurate readings at 95 °C).

6.8. Disposable cups or tubes.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Stock (10 ppm) mercury standards (in 10% HNO_3) are purchased as custom STL North Canton solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Working mercury standard (0.1 ppm): Take 1 mL of the stock mercury standard (7.2) and dilute to 100 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO_3. This acid (150 uL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.4. The calibration standards listed in Table I must be prepared fresh daily from the working standard (7.3) by transferring 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working mercury standard into 100 mL flasks and diluting to volume with reagent water.

Note: Alternate approaches to standard preparation may be taken and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained. For example, automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

7.5. The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.

7.6. Refer to Table I (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All standards must be processed through the entire analytical procedure including sample preparation.

7.7. Nitric acid (HNO_3), concentrated, trace metal grade or better.

Note: If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

7.8. Sulfuric acid (H_2SO_4), concentrated, trace metal grade or better.

7.9. Stannous sulfate solution: Add 25 g of stannous sulfate to 250 mL of 0.5 N sulfuric acid. This mixture is a suspension and should appear cloudy. This solution should be made daily and should be stirred continuously during use.

Note: Stannous chloride may be used in place of stannous sulfate. Prepare the stannous chloride solution according to the recommendations provided by the instrument manufacturer.

7.10. Sodium chloride-hydroxylamine hydrochloride solution: Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride to every 100 mL of reagent water.

Note: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.11. Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate for every 100 mL of reagent water.

7.12. Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate for every 100 mL of reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for mercury is 28 days from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. Refrigeration is not required. Preservation must be verified prior to analysis.

9. QUALITY CONTROL

Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to the analysis of any analyte using 7470A or the 245.1, the following requirements must be met.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined

on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the STL North Canton reporting limit.

9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (Method Blanks, LCS, and MS/MSDs) are not included in the sample count for determining the size of a preparation batch.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit or at or above 10% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20 times higher than the blank contamination level).

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

9.5. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. On going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The LCS must be carried through the entire analytical procedure. If the LCS is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, a control limit of 80 - 120% recovery must be applied.

- In the instance where the LCS recovery is > 120% and the sample results are < RL, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the case narrative.

- In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table I (Appendix A).

- If analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 75 - 125 % recovery and 20% RPD must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation

and reanalysis of the batch. MS/MSD results, which fall outside the control limits, must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.7. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV result must fall within 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. (See Section 11.2.11 and Section 11.2.12 for required run sequence). If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include repreparation of the ICV, ICB, CRA, CCV, and CCB with the calibration curve.

9.8. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV must be a mid-range standard at a concentration other than that of the ICV. The CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. (See Section 11.2.11 and 11.2.12 for required run sequence.) The CCB result must fall within +/- RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs.

9.9. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the sample prior to preparation. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences, which cause a baseline shift. Refer to Section 11.2.13 for additional information on when full 4 point MSA is required as well as Appendix C for specific MSA requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Calibration standards must be processed through the preparation procedure as described in Section 11.1.

10.2. Due to the differences in preparation protocols separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.

10.3. Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.4. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility specific instrument SOP and CVAA instrument manual for detailed setup and operation protocols.

10.5. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at the STL North Canton reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7.5 and Table I for additional information on preparing calibration standards and calibration levels.

10.6. The calibration curve must have a correlation coefficient of ≥ 0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results can not be reported from a curve with an unacceptable correlation coefficient.

10.7. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corrective actions.

11. PROCEDURE

11.1. Sample Preparation:

11.1.1. All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed through the digestion procedure as well as the field samples. Transfer 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working standard (7.3) into a series of 100 ml class A volumetrics, then dilute to volume . For the ICV, use a 2.5 ml aliquot of the working standard. The ICV working standard must be made from a source other than that used for the calibration standards.

11.1.2. Transfer 100 mL of well-mixed sample or standard to a clean sample digestion bottle.

Note: Reduced sample volumes can be used as long as a representative sample can be obtained and the reagent levels are adjusted to maintain the same sample to reagent ratio. All samples and standards must be processed similarly.

Note: Spiking is done before the addition of acids or reagents.

11.1.3. Add 5 mL of concentrated H_2SO_4 and 2.5 mL of concentrated HNO_3 mixing after each addition.

11.1.4. Add 15 mL of potassium permanganate solution. For samples high in organic materials or chlorides, additional permanganate may be added. Shake and add additional portions of permanganate solution until a purple color persists for at least 15 minutes. If after the addition of up to 25-mL additional permanganate the color does not persist, sample dilution prior to reanalysis may be required.

Note: When performing analyses using automated vs. manual techniques the sample dilution resultant from the addition of more than the original aliquot of permanganate solution must be compensated for by the addition of the same volume of permanganate to all other associated samples and standards in the run. In instances, where this is not feasible, the addition of excess reagent can be addressed through mathematical correction of the results to account for the resultant dilution effect.

11.1.5. Add 8 mL of potassium persulfate solution and heat for two hours in a water bath at 90 - 95 °C.

NOTE: Alternatively, for RCRA analyses using 7470A, samples may be digested using an autoclave for 15 minutes at 120 °C and 15 lbs.

11.1.6. Cool samples.

11.2. Sample Analysis:

11.2.1. Because of differences between various makes and models of CVAA instrumentation, no detailed operating instructions can be provided. Refer to the facility specific instrument operating SOP and the CVAA instrument manual for detailed setup and operation protocols.

11.2.2. All labs are required to detail the conditions/programs utilized for each instrument within the facility specific instrument operation SOP.

11.2.3. When ready to begin analysis, add 6 mL of sodium chloride-hydroxylamine hydrochloride solution to the samples to reduce the excess permanganate (the permanganate has been reduced when no purple color remains). Add this solution in 6-mL increments until the permanganate is completely reduced.

11.2.4. Manual determination:

11.2.4.1. Treating each sample individually, purge the headspace of the sample bottle for at least one minute.

11.2.4.2. Add 5 mL of stannous chloride solution and immediately attach the bottle to the aeration apparatus.

11.2.4.3. Allow the sample to stand quietly without manual agitation while the sample is aerated (1 L/min flow). Monitor the sample absorbance during aeration. When the absorbance reaches a maximum and the signal levels off, open the bypass valve and continue aeration until the absorbance returns to its baseline level. Close the bypass valve and remove the aeration device.

11.2.4.4. Place the aeration device into 100 mL of 1% HNO_3 and allow to bubble rinse until the next sample is analyzed.

11.2.5. Automated determination: Follow instructions provided by instrument manufacturer.

11.2.6. Perform a linear regression analysis of the calibration standards by plotting maximum response of the standards vs. concentration of mercury. Determine the mercury concentration in the samples from the linear regression fit of the calibration curve. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.

11.2.7. All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard.

11.2.8. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.

11.2.9. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

11.2.10. Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

11.2.11. The following analytical sequence must be used with 7470A and 245.1:

Instrument Calibration
ICV
ICB
Maximum 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table II (Appendix A) for quality control criteria to apply to Methods 7470A and 245.1.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions.

11.2.12. The following run sequence is consistent with 7470A, CLP and 245.1 and may be used as an alternate to the sequence in 11.2.11. This run sequence is recommended if multiple method requirements must be accommodated in one analytical run:

Instrument Calibration
ICV
ICB
CRA*
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run.
CCV

CCB

Refer to the appropriate CLP SOP (CORP-MT-0006) for quality control requirements for QC samples.

* Refer to the CLP SOP for information on the CRA.

11.2.13. For TCLP samples, full four point MSA will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

The reporting and matrix spike levels for TCLP analyses are detailed in Table I (Appendix A). Appendix E provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sample preparation.

11.3. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data are reviewed periodically throughout the run.

11.4. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.5. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.6. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix spike recoveries are calculated according to the following equation:

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:

DU1 = Sample result
DU2 = Sample duplicate result

12.5. The final concentration for an aqueous sample is calculated as follows:

$$mg/L = C \times D$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor

12.6. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.7. Appropriate factors must be applied to sample values if dilutions are performed.

12.8. Sample results should be reported with up to three significant figures in accordance with the STL North Canton significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Method performance is determined by the analysis of method blanks, laboratory control samples, matrix spike and matrix spike duplicate samples. The matrix spike recovery should fall within +/- 25 % and the matrix spike duplicates should compare within 20% RPD. The method blanks must meet the criteria in Section 9.3. The laboratory control sample should recover within 20% of the true value until in house limits are established.

13.3. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7470A (Mercury).

16.2. "Methods for the Chemical Analysis of Water and Wastes", EPA-600/4-79-020, U.S.EPA, August 1983, Method 245.1.

16.3. U.S.EPA Statement of Work for Inorganics Analysis, ILMO3.0 and ILMO4.0.

16.4. QA-003, STL North Canton QC Program.

16.5. QA-004, Rounding and Significant Figures.

16.6. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference method.

17.1.1. Modifications from both 7470A and 245.1.

17.1.1.1. The 200 series methods and Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.1.2. This SOP allows for the use of reduced sample volumes to decrease waste generation. Reagent levels are adjusted to maintain the same ratios as stated in the source methods. According to a letter from Robert Booth of EPA EMSL-Cinn to David Payne of EPA Region V, "Reduction in sample size and appropriate corresponding reduction in

sample volume is not considered a significant change in the methodology."

17.1.1.3. The alternate run sequence presented in Section 11.2.12 is consistent with method requirements. An additional QC analysis (CRA) was added to accommodate the CLP protocol requirements.

17.1.2. Modifications from Method 7470A

17.1.2.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.1.2.2. Documentation is on file from EPA's Office of Solid Waste (Olliver Fordham 11/28/95) regarding the acceptance of the autoclave as an equivalent heating device to the water bath. In his letter, Mr. Fordham cited the CLP water protocol 245.1 CLP-M and therefore the operating parameters from that method were adopted for 7470A (15 minutes at 120 °C and 15 lbs.).

17.1.3. Modifications from 245.1

17.1.3.1. Method 245.1 Section 9.3 states concentrations should be reported as follows: Between 1 and 10 ug/L, one decimal; above 10 ug/L, to the nearest whole number. STL North Canton reports all Hg results under this SOP to two significant figures.

17.2. Modifications from previous SOP

17.2.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.2.2. Documentation is on file from EPA's Office of Solid Waste (Olliver Fordham 11/28/95) regarding the acceptance of the autoclave as an equivalent heating device to the water bath. In his letter, Mr. Fordham cited the CLP water protocol 245.1 CLP-M and therefore the operating parameters from the method were adopted for 7470A (15 minutes at 120° C and 15 lbs.).

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none. Refer to the Appendices for any facility specific information required to support this SOP.

17.4. Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

- Raw data (direct instrument printout)
- Run log printout from instrument software where this option is available or manually generated run log. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence).
- Data review checklist - See Appendix B
- Standards Documentation (source, lot, date).
- Copy of digestion log.
- Non-conformance summary (if applicable).

Figure 1. Aqueous Sample Preparation - Mercury

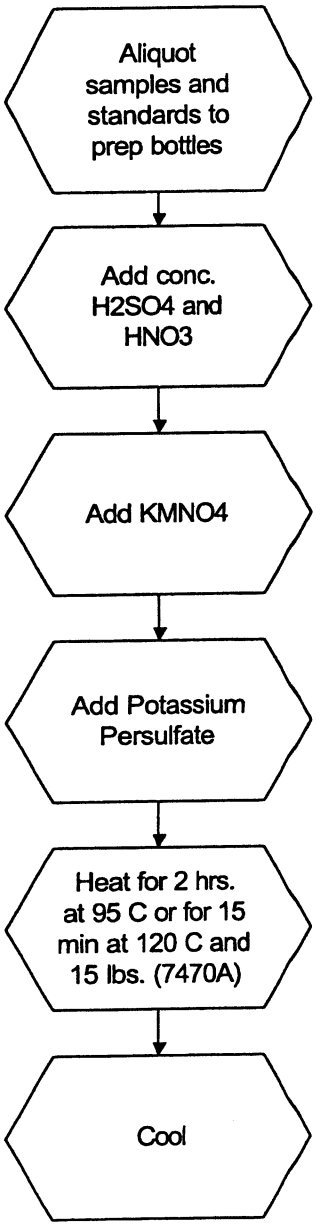
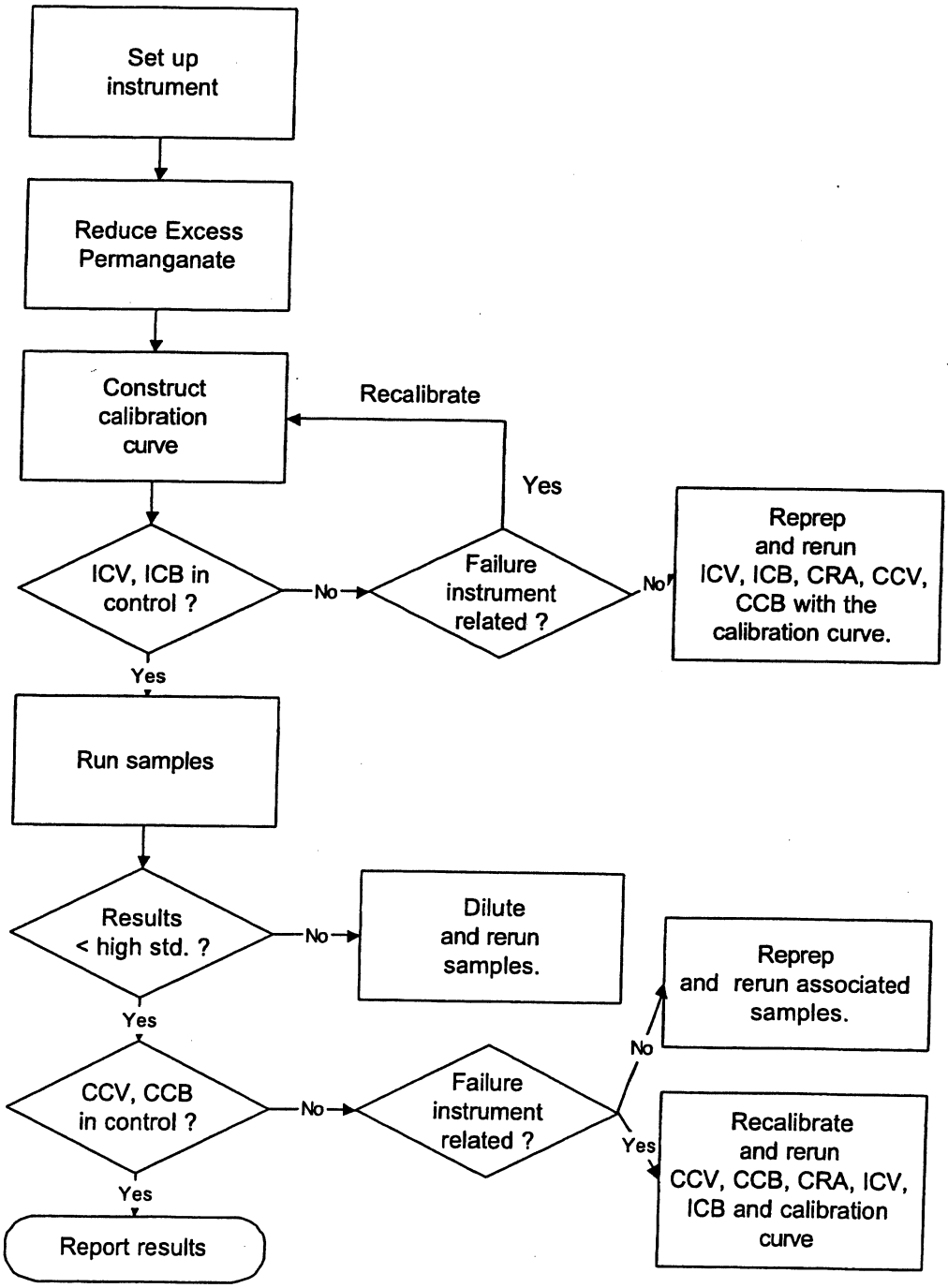


Figure 2. CVAA Mercury Analysis



APPENDIX A

TABLES

TABLE I. MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC STANDARD AND SPIKING LEVELS (MG/L)

Standard Aqueous RL	0.0002
TCLP RL	0.002
Std 0	0
Std 1	0.0002
Std 2	0.0005
Std 3	0.001
Std 4	0.005
Std 5 **	0.010
ICV	0.001 or 0.0025 ***
LCS/CCV	0.0025 or 0.005 ***
Aqueous MS	0.001
TCLP MS	0.005

* **SOP specified calibration levels must be used unless prevented by the instrument configuration or client specific requirements. Deviations from specified calibration levels must be documented in the facility specific instrument operation SOP and must be approved by the facility technical manager and Quality Assurance Manager.**

** **Optional standard which may be used to extend the calibration range as allowed by the instrument configuration. If the instrument configuration prevents the use of 6 standards, the 2-ppb standard may be eliminated in favor of the 10 ppb standard.**

*** **Concentration level dependent on high calibration standard used. CCV must be 50% of high standard concentration and ICV must be 20-25% of high standard concentration.**

TABLE II. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run.	90-110 % recovery.	Terminate analysis; Correct the problem; Recalibrate or reprep with calibration curve. (See Section 9.7).
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate or reprep with calibration curve. (See Section 9.7).
CCV	Every 10 samples and at the end of the run.	80 - 120 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep with calibration curve. (See Section 9.8).
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB or reprep with calibration curve. (See Section 9.8).
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL do not require redigestion (See Section 9.4).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.4 for additional requirements.

*See Sections 11.2.11 and 11.2.12 for exact run sequence to be followed.

TABLE II. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits.	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS (see Section 9.5).
Matrix Spike	One per sample preparation batch of up to 20 samples.	75 - 125 % recovery or in-house control limits. If the MS/MSD is out for an analyte, it must be in control in the LCS.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. (see Section 9.6) For TCLP see Section 11.2.13
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery or in-house control limits; RPD ≤ 20%. (See MS)	See Corrective Action for Matrix Spike.

APPENDIX B
EXAMPLE
STL NORTH CANTON Hg DATA REVIEW CHECKLIST

Example
STL North Canton Hg Data Review Checklist

Run/Project Information

Run Date: _____ **Analyst:** _____ **Instrument:** _____
Prep Batches Run: _____

Circle Methods used: **7470A / 245.1 :** CORP-MT-0005 Rev 1 **7471 / 245.5 :** CORP-MT-0007 Rev 1
CLP - AQ : CORP-MT-0006 Rev 0 **CLP - SOL :** CORP-MT-0008 Rev 0

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2ndLevel
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels?				
2. ICV/CCV analyzed at appropriate frequency and within control limits?				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?				
4. CRA run (CLP only)?				
B. Sample Results				
1. Were samples with concentrations > the high calibration standard diluted and reanalyzed?				
2. All reported results bracketed by in control QC?				
3. Sample analyses done within holding time?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits?				
2. Method blank done per prep batch and < RL or CRDL (CLP)?				
3. MS run at required frequency and within limits?				
4. MSD or DU run at required frequency and RPD within SOP limits?				
D. Other				
1. Are all nonconformances documented appropriately?				
2. Current IDL/MDL data on file?				
3. Calculations and Transcriptions checked for error?				
4. All client/ project specific requirements met?				
5. Date of analysis verified as correct?				

Analyst: _____ **Date:** _____
Comments:
_____
_____
_____

2nd Level Reviewer : _____ **Date:** _____
_____
_____
_____

APPENDIX C

MSA GUIDANCE

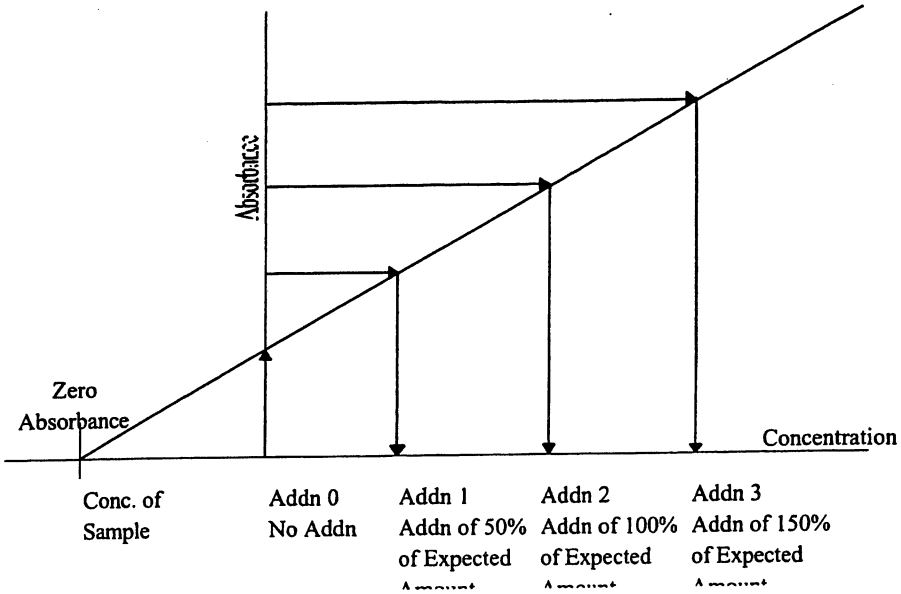
APPENDIX C. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.
- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX D

TROUBLESHOOTING GUIDE

APPENDIX D. TROUBLESHOOTING GUIDE

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Wrong lamp Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak EDL power supply set on "Continuous"
Erratic Readings	Source lamp not aligned properly Lamp not prewarmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
EDL Won't Light	Lamp cable not plugged in Lamp power set at 0 Lamp is dead Power supply fuse is blown Short in cord
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell
Background Correction Light Blinking	Background screen or attenuator faulty

APPENDIX E

CONTAMINATION CONTROL GUIDELINES

APPENDIX E. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

APPENDIX F

PREVENTIVE MAINTENANCE

APPENDIX F. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption (Leeman PS 200) [1]

Daily	Semi-annually	Annually
Clean lens.	Check Hg lamp intensity.	Change Hg lamp.
Check aperture.		Check liquid/gas separator.
Check argon flow.		
Check tubing.		
Check drain.		
Replace drying tube.		

Cold Vapor Atomic Absorption (PE 5000) [1]

Daily	Monthly
Clean aspirator by flushing with DI water.	Clean cell in aqua regia.
Check tubing and replace if needed.	Clean aspirator in aqua regia.
Clean windows with methanol.	
Change silica gel in drying tube.	
Check argon gas supply.	
Adjust lamp.	

APPENDIX G

INSTRUMENT SET UP

Hg Analysis (Leeman PS200II)

SYSTEM INITIALIZATION AND WARM UP

1. F1 Menu
2. Instrument

a. TASKMASTER

b. #4 Wake System Up Enter

The warming up period takes approximately 10 minutes.

TO SET UP INSTRUMENT FOR ANALYSIS

1. F1 Menu
2. Autosampler

   A. Rack Entry

   B. Edit (ex. Rack 1), Enter

   C. Cup ID - Enter (clears sample #'s)

   D. Extended ID- type in matrix of sample (water or solid), Enter.

   E. Press Insert Key and move cursor with arrows to cup ID and begin typing labels.

   F. F3 Print Screen

3. Press F2 Macro key and type in analyst's first name - Enter

   A. Enter folder name - ex. HG0306, Enter. If folder does not exist, type Y - Enter.

B. Type in - "Rack 1", "Rack 2" etc. , Enter.

C. Type in : FROM CUP TO CUP

Ex. = 1 30

Do the same for position 2 if needed. If not needed, you must press Enter 3 times to begin analysis.

STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: ACID DIGESTION OF SOILS, SW846 METHOD 3050B

(SUPERSEDES: REVISION 2.1 DATE 02/11/00)

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation of soil samples for the analysis of certain metals by Graphite Furnace Atomic Absorption (GFAA), Flame Atomic Absorption (FLAA), Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP), and Inductively Coupled Plasma-Mass Spectrometry (ICP/MS) as specified in SW846 Method 3050B.

1.2. Samples prepared by the protocols detailed in this SOP may be analyzed by ICP, ICP/MS, FLAA or GFAA for the elements listed in Table I (Appendix A). Other elements and matrices may be analyzed following digestion by these protocols provided that the method performance criteria specified in Section 13.0 of this SOP are met.

1.3. This method is not a total digestion, but will dissolve almost all metals that could become "environmentally available". By design, metals bound in silicate structures are not dissolved by this procedure, as they are not usually mobile in the environment. This SOP can be applied to metals in solids, sludges, wastes and sediments.

1.4. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

A representative 1 gram (wet weight) portion of sample is digested in nitric acid and hydrogen peroxide. The digestate is refluxed with hydrochloric acid for ICP, FLAA or antimony by GFAA analysis. The digestates are then filtered and diluted to 100 mL/100 g.

3. DEFINITIONS

3.1. Refer to the glossary in the Laboratory Quality Manual (LQM), latest version.

3.2. Total Metals: The concentration determined on an unfiltered sample following digestion. Note that this method is designed to determine the total *environmentally available* metals.

4. INTERFERENCES

4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include: metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2. The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination.

4.3. Boron and silica from the glassware will grow into the sample solution during and following sample processing. For critical low level determinations of boron and silica, only quartz and/or plastic labware should be used.

4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents and other matrices may not be digested using these methods if they are not soluble with acids. If physical interferences are present, they should be documented.

4.5. Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.6. Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs the sample must be reprepared. Antimony is easily lost by volatilization from hydrochloric media.

4.7. Specific analytical interferences are discussed in each of the determinative methods.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL North Canton associates.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. The following materials are known to be **corrosive:**

Hydrochloric acid and nitric acid.

5.3.2. The following materials are known to be **oxidizing agents**:

Nitric acid and hydrogen peroxide.

5.3.3. All heating of samples must be carried out in a fume hood.

5.4. The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood. The analyst should also be aware of the potential for a vigorous reaction.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit or under other means of mechanical ventilation.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported **immediately** to a laboratory supervisor.

5.8. Always carry bulk concentrated acid bottles in appropriate impact proof containers.

5.9. Acid/peroxide spills must be neutralized immediately, flushed with water and cleaned up using appropriate spill kits.

5.10. Discard chipped or broken beakers to prevent injury. Chipped glassware may be fire polished as an alternative to disposal.

5.11. Any and all accidents and spills must be reported to the lab supervisor or EH&S coordinator.

6. EQUIPMENT AND SUPPLIES

6.1. Hot plate, digestion block, steam bath or other heating source capable of maintaining a temperature of 90-99°C.

6.2. Calibrated thermometer that covers a temperature range of 0-200°C.

6.3. Griffin beakers of assorted sizes or equivalent.

6.4. Vapor recovery device (Watch glasses, ribbed or other device).

6.5. Whatman No. 41 filter paper or equivalent.

6.6. Funnels or equivalent filtration apparatus.

6.7. Centrifugation equipment (if desired method of removing particulates is centrifugation).

6.8. Graduated cylinder or equivalent capable of measuring 100 mL within 3% accuracy.

6.9. Analytical balance capable of accurately weighing to the nearest 0.01 grams.

6.10. Repippetors or suitable reagent dispensers.

6.11. Calibrated automatic pipettes with corresponding pipette tips or Class A glass volumetric pipettes.

6.12. Class A volumetric flasks.

6.13. pH indicator strips (pH range 0 - 6).

6.14. Plastic bottles.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative SOPs.

7.2. Laboratory Control Sample (LCS) and matrix spike (MS) solutions are purchased as custom STL North Canton solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Working ICP LCS/MS spike solution: Prepare the ICP LCS/MS working spike solutions from custom stock standards to the final concentration listed in Table II. The working spike must be prepared in a matrix of 5% HNO_3. This acid (5 mL of concentrated HNO_3 per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working ICP LCS solution must be made fresh every three months.

7.4. Working GFAA LCS/MS spike solution: Prepare the GFAA working LCS/MS spike solutions by diluting the custom stock solution (7.2) 200x. The working spike solution must be prepared in a matrix of 5% HNO_3. This acid (5 mL of concentrated HNO_3 per 100 mL) must be added to the volumetric flask before the addition of the stock standard aliquot. The working GFAA LCS/MS solution must be made fresh every three months.

7.5. The LCS and MS samples must contain all the elements designated for analysis in each batch of samples. If a non-routine element is required that is not contained in the custom STL North Canton solution, the individual facility must purchase a solution from the designated vendor that will cover the additional analyte(s) of interest and provide for a final spike concentration that is appropriate to the determinative method.

7.6. Aqueous laboratory control samples (LCSW) and matrix spike samples are prepared as described in Sections 9.5 and 9.6. Refer to Tables II and III (Appendix A) for details regarding the stock, working standard and final digestate spike concentrations for ICP and GFAA LCS and matrix spike preparations.

7.7. Nitric acid (HNO_3), concentrated, trace metal grade or better.

7.8. Nitric acid, 1:1 - dilute concentrated HNO_3 with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

7.9. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.10. Hydrochloric acid, 1:1 - dilute concentrated HCl with an equal volume of reagent water.

Note: When preparing diluted acids always add acid to water. If the water is added to the acid a violent reaction may occur.

7.11. 30% Hydrogen peroxide (H_2O_2), reagent grade.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2. Soil samples do not require preservation but must be stored at 4 °C ± 2 °C until the time of analysis.

9. QUALITY CONTROL

Table IV (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using Method 3050B the following requirements must be met.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements or verified as detailed in STL North Canton QA Policy QA-005. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the STL North Canton reporting limit.

9.1.2. Initial Demonstration Study- this requires the analysis of four QC check samples. The QC check sample is a well characterized laboratory generated sample used to monitor method performance, which should contain all the analytes of interest. The results of the initial demonstration study must be acceptable before analysis of samples may begin. The results of the initial demonstration study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.1.2.2. Calculations and acceptance criteria for QC check samples are given in the determinative SOPs (CORP-MT-0001, CORP-MT-0003).

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not counted towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. Criteria for the acceptance of blanks are contained within the individual analytical method SOP's. If the method blank does not meet the criteria contained within the analytical method SOPs, the blank and all associated samples in the batch must be redigested.

9.5. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Criteria for the acceptance of LCS results are contained within the individual analytical method SOP's. Corrective action when LCS results fail to meet control limits will be repreparation and reanalysis of the batch. Tables II and III provide the details regarding the stock, working standards and final spike concentrations for ICP and GFAA. Refer to Section 7.3 or 7.4 for instructions on preparation of the aqueous LCS.

9.5.1. The LCS is prepared by spiking a 1mL or 1 g aliquot of reagent water with 2 mL of the working LCS/MS spike solution (7.3 or 7.4). The LCS is then processed as described in either Section 11.10.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis. If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. Corrective action when MS results fail to meet control limits

does not include repreparation of samples unless the results indicate that a spiking error may have occurred. Tables II and III provide the details regarding the stock, working standards and final matrix spike concentrations for ICP and GFAA. Refer to Sections 7.3 and 7.4 for instructions on preparation of the working matrix spike solutions.

9.6.1. The soil matrix spike sample is prepared by spiking a 1 g aliquot of a sample with 2mL of the working LCS/MS spike solution (7.3 or 7.4). The matrix spike sample is then processed as described in either Section 11.10.

9.7. Quality Assurance Summaries - Certain clients may require specific project or program QC which may supersede the SOP requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Hotplate or block temperature must be verified daily for each unit used and must be recorded on either the metals preparation log or in a hotplate temperature logbook. The hotplate temperature should be verified by measuring the temperature of a beaker of reagent water placed on each hotplate. For block digestors, use a tube containing water.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. The heating procedures are carried out in a properly functioning hood.

11.4. All samples are to be checked out of sample control with the chain of custody documentation filled out completely.

11.5. Proper sample identification is extremely important in any preparation procedure. Labeling of beakers and bottles must be done in a manner to ensure connection with the proper sample. The use of automatic label printing programs is recommended to reduce transcription errors (QuantIMS option).

11.6. Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating prep, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous but it appears more like a waste (biphasic, sludge like, organic liquid, lots of sediment etc.) contact the lab supervisor or project administrator for further instructions. In some cases it may be more appropriate to process these samples as solids.

11.7. If possible prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and reporting group.

11.8. In some cases, both AA and ICP digests are required on each sample. It is recommended that both aliquots be weighed out and processed at the same time.

11.9. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

11.10. Preparation of Soils, Sediments and Sludges for Analysis by GFAA, ICP, ICP/MS and FLAA.

11.10.1. Mix sample thoroughly by stirring with a clean plastic or wooden spoon or spatula.

11.10.2. For each digestion procedure required (i.e., ICP or GFAA), weigh a 1.0 portion of solid and record the exact weight to the nearest 0.01 g. A 2 g sample size may also be used if needed to meet the reporting limits.

11.10.3. Measure additional aliquots of the designated samples for the MS and MSD analyses.

11.10.4. Spike each of the MS and MSD aliquots with 2 mL of the working LCS/MS spiking solution (7.3 or 7.4).

11.10.5. Prepare a beaker for the method blank.

11.10.6. Prepare a beaker for the LCS. Add 2 mL of the working LCS/MS spiking solution (7.3 or 7.5).

11.10.7. Add 10 mL of 1:1 HNO_3 and mix the sample.

11.10.8. Heat sample to 95°±4° C and reflux for 10 minutes without boiling, using a vapor recovery device.

Note: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY during any part of the digestion. Doing so will result in the loss of analyte and the sample must be reprepared.

11.10.9. Allow sample to cool, if necessary.

11.10.10. Add 5 mL of concentrated HNO_3 and replace vapor recovery device.

11.10.11. Reflux at 95°±4° C for 30 minutes. (Add reagent water as needed to ensure that the volume of solution is not reduced to less than 5 mL.)

11.10.12. If brown fumes are observed, repeat step 11.10.10 until no more fumes are evolved.

11.10.13. Allow the samples to cool, if necessary.

11.10.14. Add approximately 2 mL of reagent water and 1 mL of 30 % H_2O_2. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

11.10.15. Replace the vapor recovery device and heat sample until effervescence subsides.

11.10.16. Allow the sample to cool, if necessary.

11.10.17. Continue adding 30% H_2O_2 in 1 mL aliquots with warming until effervescence is minimal or sample appearance is unchanged.

Note: Do not add more than a total of 10 mL of 30 % H_2O_2.

11.10.18. Continue heating at 95°±4° C for 75 minutes until the volume is reduced to approximately 5-10 mL. Alternatively the sample may be heated for 2 hours.

11.10.19. If the sample is being prepared for ICP or FLAA analyses add 10 mL of concentrated HCl and reflux for an additional 10 minutes without boiling. This step is omitted for analysis by ICP-MS and GFAA

Note: Antimony and silver have poor solubility in dilute nitric acid solution. Therefore it is strongly recommended that these elements are determined by the ICP procedure that includes HCl as the final digestion acid.

11.10.20. Allow the sample to cool.

11.10.21. Wash down beaker walls and vapor recovery device with reagent water.

11.10.22. Filter sample through Whatman 41 filter paper or equivalent into a graduated cylinder or equivalent or a pre-weighed bottle. Other measuring bottles (for example, Corning Snap Seals™) may be used if their accuracy is documented and is better than $\pm$ 2%. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

Note: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material

11.10.23. Dilute sample to 100 mL or 100g with reagent water. The sample is now ready for analysis.

Note: This SOP allows for samples to be weighed instead of measured volumetrically. This assumes the density of the diluted sample is close to 1.0 g/mL (See Section 17.1.2).

12. DATA ANALYSIS AND CALCULATIONS

Not Applicable.

13. METHOD PERFORMANCE

13.1. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. In general, the matrix spike recovery should fall within +/- 20 % and the matrix spike duplicates should compare within 20% RPD. Method blanks must meet the criteria specified in the determinative SOPs. The laboratory control samples should recover within 20% of the true value until in house control limits are established. Acceptance criteria are given in the determinative SOPs.

13.2. The initial demonstration study as detailed in Section 9.1.2 must be acceptable before the analysis of field samples under this SOP may begin. The results of the initial demonstration

study may be used to extend a method for the analysis of other elements provided all acceptance criteria are met.

13.3. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

15.2. Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996. Method 3050B.

16.2. CORP-MT-0001, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analysis of Water and Wastes, Method 6010B and Method 200.7.

16.3. CORP-MT-0003, Graphite Furnace Atomic Absorption Spectroscopy, SW846 Method 7000A and MCAWW 200 Series Methods.

16.4. NC-MT-0002, Inductively Coupled Plasma-Mass Spectrometry, EPA Methods 6020 and 200.8.

16.5. QA-003, STL North Canton QC Program.

16.6. QA-004, Rounding and Significant Figures.

16.7. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference method.

17.1.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants, as defined in the determinative SOPs, are allowed up to two times the reporting limit in the blank following consultation with the client.

17.1.2. This SOP allows for aqueous samples to be weighed instead of measured volumetrically. This assumes the density of the sample is close to 1.0 g/mL. Samples with large amounts of sediment or suspended solids, sludges, non-aqueous liquids must be processed volumetrically. Weighing samples directly into the digestion vessel minimizes the potential for cross contamination, offers improved accuracy over the use of graduated cylinders (comparable to volumetric flask accuracy), uses less glassware and is more efficient.

17.2. Modifications from previous SOP

17.2.1. ICP/MS has been added as an appropriate determinative technique.

17.2.2. The approved analyte list may be modified provided that additional elements meet the requirements in section 13.

17.2.3. Directions for digestion for set time periods rather than reduction to set volumes have been added.

17.2.4. The order of two steps in the digestion has been changed. (See section 11.10.20)

17.2.5. Definition of the method as determining total environmentally available metals has been added.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none. Refer to the Appendices for any facility specific information required supporting this SOP.

17.4. Documentation and Record Management

The preparation benchsheet should, at a minimum, include the following information:

- Preparation date, analyst initials, matrix, prep type (ICP or GFAA), SOP reference.
- Sample ID; initial weight/volume and final weight/volume.
- Standards Documentation (source, lot, prep date, volume added).
- Analyst Signature.
- Reviewer's Signature and date.

Figure 1. Soil Sample Preparation (Section 11.10)

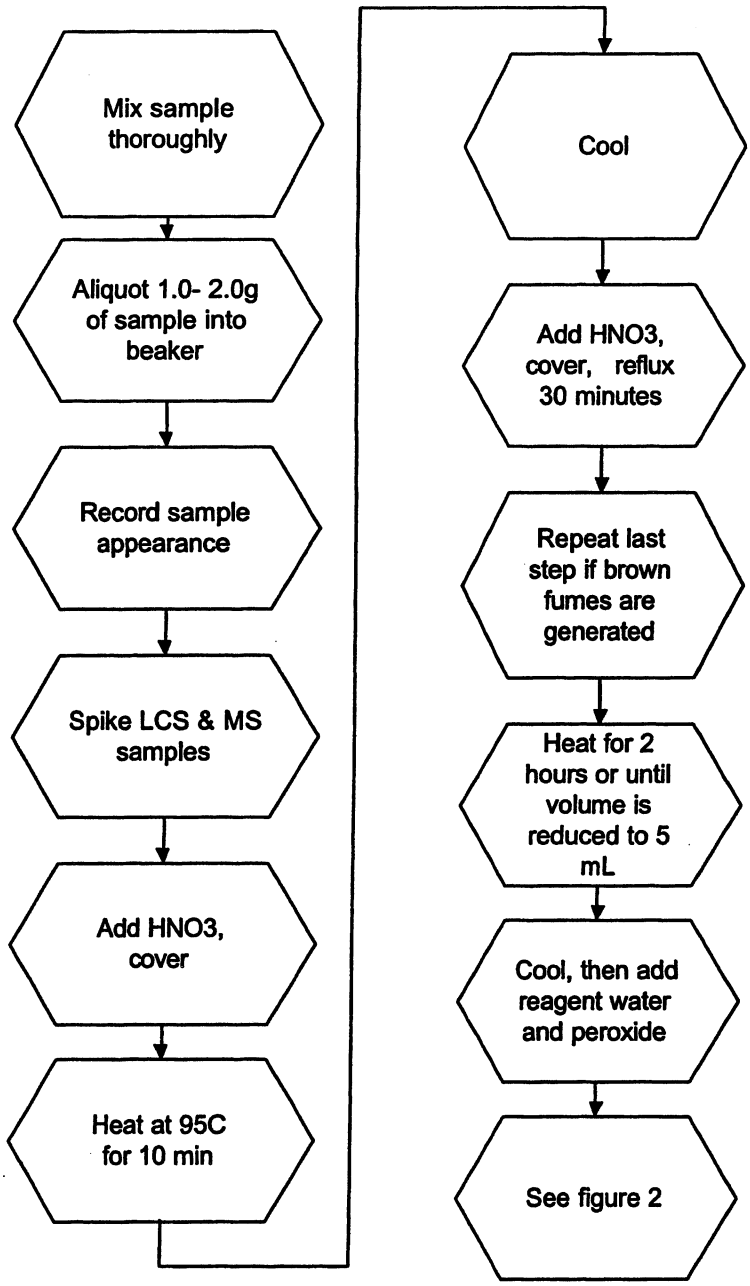
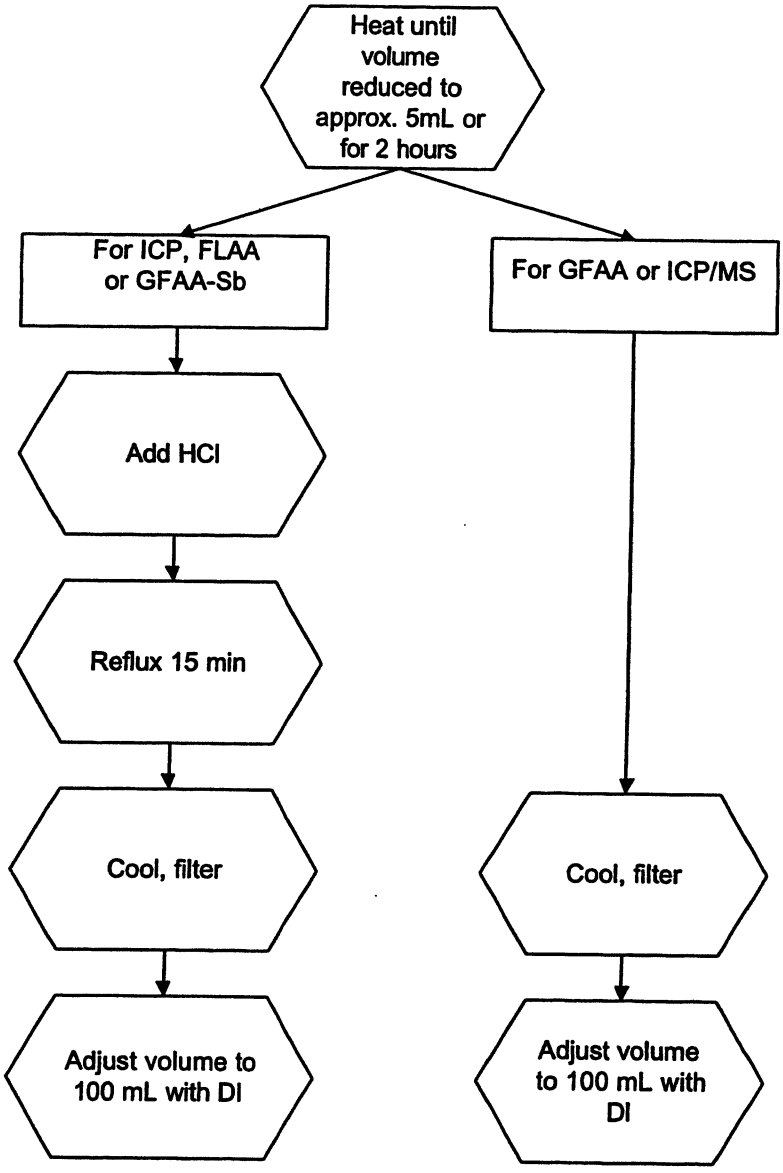


Figure 2. Soil Sample Preparation (continued)



APPENDIX A

TABLES

TABLE I. Method 3050A Approved Analyte List

ELEMENT	Symbol	CAS Number
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Thallium	Tl	7440-28-0
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

TABLE II. ICP and FLAA Soil Matrix Spike and Aqueous LCS Levels

ELEMENT	Working LCS/MS Standard (mg/L)	Aqueous LCS/MS Level* (ug/L)	Soil MS Level ** (mg/Kg)
Aluminum	100	2000	200
Antimony	25	500	50
Arsenic	100	2000	200
Barium	100	2000	200
Beryllium	2.5	50	5
Cadmium	2.5	50	5
Calcium	2500	50000	5000
Chromium	10	200	20
Cobalt	25	500	50
Copper	12.5	250	25
Iron	50	1000	100
Lead	25	500	50
Lithium	50	1000	100
Magnesium	2500	50000	5000
Manganese	25	500	50
Molybdenum	50	1000	100
Nickel	25	500	50
Potassium	2500	50000	5000
Selenium	100	2000	200
Silver	2.5	50	5
Sodium	2500	50000	5000
Strontium	50	1000	100
Thallium	100	2000	200
Vanadium	25	500	50
Zinc	25	500	50
Boron	50	1000	100
Tin	100	2000	200
Titanium	50	1000	100

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 2.0 mL working spike (7.3) to 100 mL of sample.

** Final soil spike concentration based on the addition of 2.0 mL working spike (7.3) to 1.0 g of sample/100 mL final volume (assumes 100% solids).

TABLE III. ICPMS Soil Matrix Spike and Aqueous LCS Levels

ELEMENT	Working LCS/MS Standard (mg/L)	Aqueous LCS/MS Level* (ug/L)	Soil MS Level ** (mg/Kg)
Aluminum	10	1000	100
Antimony	10	100	10
Arsenic	10	100	10
Barium	10	100	10
Beryllium	10	100	10
Cadmium	10	100	10
Calcium	100	1000	100
Chromium	10	100	10
Cobalt	10	100	10
Copper	10	100	10
Iron	100	1000	100
Lead	10	100	10
Magnesium	100	1000	100
Manganese	10	100	10
Molybdenum	10	100	10
Nickel	10	100	10
Potassium	100	1000	100
Selenium	10	100	10
Silver	10	100	10
Sodium	100	1000	100
Strontium	10	100	10
Thallium	10	100	10
Vanadium	10	100	10
Zinc	10	100	10
Boron	10	100	10
Tin	10	100	10
Titanium	10	100	10
Zirconium	10	100	10

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 1.0 mL working spike (7.4) to 100 mL of sample.

** Final soil spike concentration based on the addition of 1.0 mL working spike (7.4) to 1.0 g of sample/100 mL final volume (assumes 100% solids).

TABLE IV. GFAA Soil Matrix Spike and Aqueous LCS Spike Levels

ELEMENT	Stock LCS/MS Standard (mg/L)	Working LCS/MS Standard (ug/L)	Aqueous LCS/ MS Level * (ug/L)	Soil MS Level** (mg/Kg)
Arsenic	400	2000	40	4
Selenium	400	2000	40	4
Lead	400	2000	40	4
Thallium	400	2000	40	4
Antimony	400	2000	40	4
Cadmium	40	200	4	0.4
Chromium	100	500	10	1
Silver	50	250	5	0.5

* Levels shown indicate the spike concentration in the final digestate of the aqueous LCS or matrix spike based on the addition of 1.0 mL working spike (7.5) to 50 mL of sample.

** Final soil spike concentration based on the addition of 2.0 mL working spike (7.5) to 1.0 g of sample/100 mL final volume (assumes 100% solids).

TABLE V. Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Method Blank	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: CORP-MT-0001 CORP-MT-0003	Redigest and reanalyze samples.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: CORP-MT-0001 CORP-MT-0003	Redigest and reanalyze all samples associated with the LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	Refer to determinative SOPs: CORP-MT-0001 CORP-MT-0003	Reprep not required unless preparation error suspected.
Matrix Spike Duplicate	See Matrix Spike	Refer to determinative SOPs: CORP-MT-0001 CORP-MT-0003	See Corrective Action for Matrix Spike.

APPENDIX B

CONTAMINATION CONTROL GUIDELINES

APPENDIX B. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or latex gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

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Implementation Date: 06/05/01

SOP No. CORP-MT-0007NC
Revision No. 2.3
Revision Date: 05/15/01

STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: PREPARATION AND ANALYSIS OF MERCURY IN SOLID SAMPLES BY COLD VAPOR ATOMIC ABSORPTION SPECTROSCOPY, SW846 7471A AND MCAWW 245.5

(SUPERSEDES: REVISION 2.2, DATED 02/06/01)

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1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of mercury (Hg, CAS # 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7471A and MCAWW Method 245.5.

1.2. The associated LIMs method code is O9.

1.3. CVAA analysis provides for the determination of total mercury (organic and inorganic). The combination of the oxidants, potassium permanganate and potassium persulfate, has been found to give 100% recovery with both types of compounds. Detection limits, sensitivity and optimum concentration ranges for mercury analysis will vary with the matrices, instrumentation and volume of sample used.

1.4. Methods 7471A and 245.5 are applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits, wastes, wipes and sludge-type materials. All matrices require sample preparation prior to analysis.

1.5. The STL North Canton reporting limit for mercury in solid matrices is 0.033 mg/kg based on a 0.6 g sample aliquot (wet weight).

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of mercury in solution. The procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. A representative portion of the sample is digested in hydrochloric and nitric acids. Organic mercury compounds are oxidized with potassium permanganate and potassium persulfate and the mercury reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. Concentration of the analyte in the sample is determined by comparison of the sample absorbance to the calibration curve (absorbance vs. concentration).

3. DEFINITIONS

3.1. Total Metals: The concentration determined on an unfiltered sample following digestion.

4. INTERFERENCES

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1. Potassium permanganate which is used to breakdown organic mercury compounds also eliminates possible interferences from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.2. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on the recovery of mercury from spiked samples.

4.3. Chlorides can cause a positive interference. Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) and purging the sample headspace before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

Note: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.4. Interference from certain volatile organic materials that absorb at this wavelength may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.5. Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs the recovery of mercury will be low. The problem can be eliminated by reducing the volume of original sample used.

4.6. The most common interference is laboratory contamination which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL North Canton associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. The following materials are known to be **corrosive:**

hydrochloric acid, nitric acid and sulfuric acid.

5.3.2. The following materials are known to be **oxidizing agents**:

nitric acid, potassium permanganate, potassium persulfate and magnesium perchlorate.

5.3.3. Mercury is a highly toxic element that must be handled with care. The analyst must be aware of the handling and clean-up techniques before working with mercury. Since mercury vapor is toxic, precaution must be taken to avoid its inhalation, ingestion or absorption through skin. All lines should be checked for leakage and the mercury vapor must be vented into a hood or passed through a mercury absorbing media such as:

5.3.3.1. Equal volumes of 0.1 M $KMnO_4$ and 10% H_2SO_4, or

5.3.3.2. Iodine, 0.25%, in a 3% KI solution.

5.3.4. Magnesium sulfate is known to be a reproductive toxin (mutagen).

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported **immediately** to a laboratory supervisor.

5.6. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.

5.7. Cylinders of compressed gas must be handled with caution, in accordance with local regulations. It is recommended that, wherever possible, cylinders be located outside the laboratory and the gas led to the instrument through approved lines.

5.8. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

6. EQUIPMENT AND SUPPLIES

6.1. Temperature controlled water bath (capable of maintaining temperature of 90- 95 °C).

6.2. Atomic Absorption Spectrophotometer equipped with:

6.2.1. Absorption Cell with quartz end windows perpendicular to the longitudinal axis. Dimensions of the cell must result in sufficient sensitivity to meet the SOP defined reporting limit. The quartz windows must be maintained to provide accurate measurements. Any scratches or fingerprints can alter the absorption of UV radiation.

6.2.2. Mercury specific hollow cathode lamp (HCL) or electrodeless discharge lamp (EDL).

6.2.3. Peristaltic pump which can deliver 1 L/min air.

6.2.4. Flowmeter capable of measuring an airflow of 1 L/min.

6.2.5. Recorder or Printer.

6.2.6. Aeration Tubing: A straight glass frit having a course porosity and Tygon tubing is used for the transfer of mercury vapor from the sample bottle to the absorption cell and return.

6.2.7. Drying device (a drying tube containing magnesium perchlorate or magnesium sulfate and/or a lamp with a 60 W bulb) to prevent condensation in cell. The lamp is positioned to shine on the absorption cell maintaining the air temperature in the cell about 10 °C above room temperature. Other drying devices that acheive the same purpose are also acceptable (i.e., Gortex filter).

Note: Instruments designed specifically for the measurement of mercury using the cold vapor technique may be substituted for the atomic absorption spectrophotometer.

6.3. BOD bottles or equivalent.

6.4. Nitrogen or argon gas supply, welding grade or equivalent.

6.5. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.6. Class A volumetric flasks.

6.7. Top-loading balance, capable of reading up to two decimal places.

6.8. Thermometer (capable of accurate readings at 95 °C).

6.9. Disposable cups or tubes.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2. Stock (10 ppm) mercury standards (in 10% HNO_3) are purchased as custom STL North Canton solutions. All standards must be stored in FEP fluorocarbon or previously unused

polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Working mercury standard (0.1 ppm): Take 1 mL of the stock mercury standard (7.2) and dilute to 100 mL with reagent water. The working mercury standard must be made daily and must be prepared in a matrix of 0.15% HNO_3. This acid (150 uL of concentrated HNO_3) must be added to the flask/bottle before the addition of the stock standard aliquot.

7.4. The calibration standards must be prepared fresh daily from the working standard (7.3) by transferring 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working mercury standard into sample preparation bottles and proceeding as specified in Section 11.1

Note: Alternate approaches to standard preparation may be taken and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained. For example, automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

7.5. The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.

7.6. Refer to Table I (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification and spiking solutions. All standards must be processed through the entire analytical procedure including sample preparation.

7.7. Nitric acid (HNO_3), concentrated, trace metal grade or better.

Note: If a high reagent blank is obtained, it may be necessary to distill the nitric acid.

7.8. Sulfuric acid (H_2SO_4), concentrated, trace metal grade or better.

7.9. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.10. Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3.

7.11. Stannous sulfate solution: Add 25 g of stannous sulfate to 250 mL of 0.5 N sulfuric acid. This mixture is a suspension and should appear cloudy. This solution should be made daily and should be stirred continuously during use.

Note: Stannous chloride may be used in place of stannous sulfate. Prepare the stannous chloride solution according to the recommendations provided by the instrument manufacturer.

7.12. Sodium chloride-hydroxylamine hydrochloride solution: Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride to every 100 mL of reagent water.

Note: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.13. Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate for every 100 mL of reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for mercury is 28 days from time of collection to the time of sample analysis.

8.2. Soil samples do not require preservation but must be stored at 4° C ± 2° C until the time of analysis.

9. QUALITY CONTROL

Table II (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to the analysis of any analyte using 7471A or the 245.5, the following requirements must be met.

9.1.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the STL North Canton reporting limit.

9.1.2. Initial Demonstration Study - This requires the analysis of four QC check samples. The QC check sample is a well characterized laboratory generated sample used to

monitor method performance. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

9.1.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.2. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.3. Sample Count - Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not included in the sample count for determining the size of a preparation batch.

9.4. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20 times higher than the blank contamination level).

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

9.5. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the

method within acceptable accuracy and precision guidelines. The LCS must be carried through the entire analytical procedure. If the LCS is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, a control limit of 70-130% recovery must be applied.

- In the instance where the LCS recovery is > 130% and the sample results are < RL, the data may be reported with qualifiers. Such action must be taken in consultation with the client and must be addressed in the case narrative."

- In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.6. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table I (Appendix A).

- If analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 70 - 130 % recovery and 20% RPD must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not

necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.7. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV result must fall within 10% of the true value for that solution . An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. (See Section 11.2.10 and Section 11.2.11 for required run sequence). If the cause of the ICV or ICB failure was not directly instrument related the corrective action will include repreparation of the ICV, ICB, CRA, CCV and CCB with the calibration curve.

9.8. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV must be a mid-range standard at a concentration other than that of the ICV. The CCV result must fall within 20% of the true value for that solution. A CCB is analyzed immediately following each CCV. (See Section 11.2.10 and 11.2.11 for required run sequence.) The CCB result must fall within +/- RL from zero. Each CCV and CCB analyzed must reflect the conditions of analysis of all associated samples. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs.

9.9. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the sample prior to preparation. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Section 11.2.12 for additional information on when full 4 point MSA is required as well as Appendix C for specific MSA requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Calibration standards must be processed through the preparation procedure as described in Section 11.1.

10.2. Due to the differences in preparation protocols separate calibration and calibration verification standards must be prepared for aqueous and solid matrices.

10.3. Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.4. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required). Refer to the facility specific instrument SOP and CVAA instrument manual for detailed setup and operation protocols.

10.5. Calibrate the instrument according to instrument manufacturer's instructions, using a minimum of five standards and a blank. One standard must be at the STL North Canton reporting limit. Analyze standards in ascending order beginning with the blank. Refer to Section 7.5 and Table I for additional information on preparing calibration standards and calibration levels.

10.6. The calibration curve must have a correlation coefficient of ≥0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results can not be reported from a curve with an unacceptable correlation coefficient.

10.7. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corrective actions.

11. PROCEDURE

11.1. Standard and Sample Preparation:

11.1.1. All calibration and calibration verification standards (ICV, ICB, CCV, CCB) are processed through the digestion procedure as well as the field samples.

11.1.2. Transfer 0, 0.2, 0.5, 1.0, 5.0 and 10.0 mL aliquots of the working standard (7.3) into a series of sample digestion bottles. For the ICV, transfer a 2.5 ml aliquot of the working standard. The ICV working standard must be made from a source other than that used for the calibration standards.

Note: Alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations as detailed in Table I are maintained.

11.1.3. Add reagent water to each standard bottle to make a total volume of 10 mL. Continue preparation as described under 11.1.5 below.

11.1.4. Transfer triplicate 0.2 g portions of a well mixed sample into a clean sample digestion bottle. Add 10mL of reagent water to each standard bottle. Continue

preparation as described under 11.1.5.

11.1.5. Water Bath protocol:

11.1.5.1. To each **standard** bottle: Add 5 mL of aqua regia.
To each **sample** bottle: Add *10* mL of reagent water and 5 mL of aqua regia.

11.1.5.2. Heat for 2 minutes in a water bath at 90 - 95 ° C.

11.1.5.3. Add *40* mL of distilled water.

11.1.5.4. Add 15 mL of potassium permanganate solution.

11.1.5.5. Heat for 30 minutes in the water bath at 90 - 95 °C.

11.1.5.6. Cool.

11.1.5.7. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.

11.1.5.8. To each **standard** bottle: Add 50 mL of reagent water.
To each **sample** bottle: Add *50* mL of reagent water.

11.1.5.9. Continue as described under Section 11.2.

11.2. Sample Analysis:

11.2.1. Because of differences between various makes and models of CVAA instrumentation, no detailed operating instructions can be provided. Refer to the facility specific instrument operating SOP and the CVAA instrument manual for detailed setup and operation protocols.

11.2.2. All labs are required to detail the conditions/programs utilized for each instrument within the facility specific instrument operation SOP.

11.2.3. Manual determination:

11.2.3.1. Treating each sample individually, purge the head space of the sample bottle for at least one minute.

11.2.3.2. Add 5 mL of stannous chloride solution and immediately attach the bottle to the aeration apparatus.

11.2.3.3. Allow the sample to stand quietly without manual agitation while the sample is aerated (1 L/min flow). Monitor the sample absorbance during aeration. When the absorbance reaches a maximum and the signal levels off, open the bypass valve and continue aeration until the absorbance returns to its baseline level. Close the bypass valve and remove the aeration device.

11.2.3.4. Place the aeration device into 100 mLs of 1% HNO_3 and allow to bubble rinse until the next sample is analyzed.

11.2.4. Automated determination: Follow instructions provided by instrument manufacturer.

11.2.5. Perform a linear regression analysis of the calibration standards by plotting maximum response of the standards vs. ug of mercury. Determine the mercury concentration in the samples from the linear regression fit of the calibration curve. Calibration using computer or calculation based regression curve fitting techniques on concentration/response data is acceptable.

11.2.6. All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples for analytes that exceed the highest calibration standard.

11.2.7. If the sample results are negative and the absolute value of the negative result is greater than the reporting limit, the sample must be diluted and reanalyzed.

11.2.8. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

11.2.9. Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB; resloping is acceptable as long as it is immediately preceded and followed by a compliant CCV and CCB.

11.2.10. The following analytical sequence must be used with 7471A and 245.5:

Instrument Calibration
ICV
ICB

Maximum 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

Refer to Quality Control Section 9.0 and Table II (Appendix A) for quality control criteria to apply to Methods 7471A and 245.5.

Note: Samples include the method blank, LCS, MS, MSD, duplicate, field samples and sample dilutions.

11.2.11. The following run sequence is consistent with 7471A, CLP and 245.5 and may be used as an alternate to the sequence in 11.2.10. This run sequence is recommended if multiple method requirements must be accommodated in one analytical run:

Instrument Calibration
ICV
ICB
CRA*
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run.
CCV
CCB

Refer to the appropriate CLP SOP (CORP-MT-0008) for quality control requirements for QC samples.

* Refer to the CLP SOP for information on the CRA.

11.2.12. For TCLP samples, full four point MSA will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

Appendix E provides guidance on performing MSA analyses. For TCLP mercury determinations, MSA spikes must be added prior to sample preparation.

11.3. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.4. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.5. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.6. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix spike recoveries are calculated according to the following equation:

$$\%\,R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:

DU1 = Sample result
DU2 = Sample duplicate result

12.5. For automated determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$mg/kg,\ dry\ weight = (C \times V \times D)/(W \times S)$$

Where:

C = Concentration (ug/L) from instrument readout
V = Volume of digestate (L)
D = Instrument dilution factor
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation.

12.6. For manual (total) determinations, the final concentration determined in solid samples when reported on a dry weight basis is calculated as follows:

$$mg/kg,\ dry\ weight = (C)/(W \times S)$$

Where:
C = Concentration (ug) from instrument readout
W = Weight in g of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on a wet weight basis, the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. Sample results should be reported with up to three significant figures in accordance with the STL North Canton significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.1.

13.2. Method performance is determined by the analysis of method blank, laboratory control sample, matrix spike and matrix spike duplicate samples. The matrix spike recovery should fall within +/- 30 % and the matrix spike duplicates should compare within 20% RPD. The method blanks must meet the criteria in Section 9.4. The laboratory control sample should recover within 20% of the true value until in house limits are established.

13.3. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A (Mercury).

16.2. "Methods for the Chemical Analysis of Water and Wastes", EPA-600/4-79-020, U.S.EPA, August 1983, Method 245.5.

16.3. U.S.EPA Statement of Work for Inorganics Analysis, ILMO3.0.

16.4. QA-003, STL North Canton QC Program.

16.5. QA-004, Rounding and Significant Figures.

16.6. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference method.

17.1.1. Modifications from both 7471A and 245.5.

17.1.1.1. A potassium persulfate oxidation step has been included to facilitate the breakdown of organic mercurials which are not completely oxidized by potassium permanganate. Use of potassium persulfate in combination with the permanganate improves the recovery of mercury from organo-mercury compounds. The use of persulfate has been incorporated in several recent EPA mercury protocols.

17.1.1.2. The alternate run sequence presented in Section 11.2.11 is consistent with method requirements. An additional QC analysis (CRA) was added to accommodate the CLP protocol requirements.

17.1.2. Modifications from Method 7471A

17.1.2.1. Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.2.2. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit.

17.1.3. Modifications from 245.5

17.1.3.1. Method 245.5 Section 9.3 states concentrations should be reported as follows: Between 0.1 and 1 ug/g, to the nearest 0.01 ug; between 1 and 10 ug/g, to the nearest 0.1ug; above 10 ug/g, to the nearest ug. STL North Canton reports all Hg results under this SOP to two significant figures.

17.2. Modifications from previous SOP

17.2.1. Section 1.4 reporting limit changed form 0.1 mg/kg based on a 0.2 g to 0.3 mg/kg based on a 0.6 g sample aliquot

17.2.2. Section 9.3 added MS and MSDs as not counted in determination of preparation batches.

17.3. Facility Specific SOPs

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none. Refer to the Appendices for any facility specific information required to support this SOP.

17.4. Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

- Raw data (direct instrument printout)
- Run log printout from instrument software where this option is available or manually generated run log. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence).
- Data review checklist - See Appendix B
- Standards Documentation (source, lot, date).
- Copy of digestion log.

Non-conformance summary (if applicable).

Figure 1. Solid Sample Preparation for Mercury - Water Bath Procedure

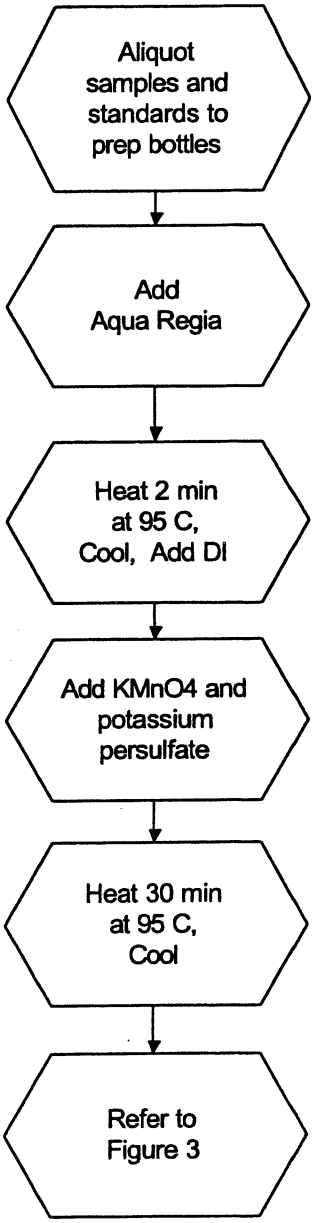
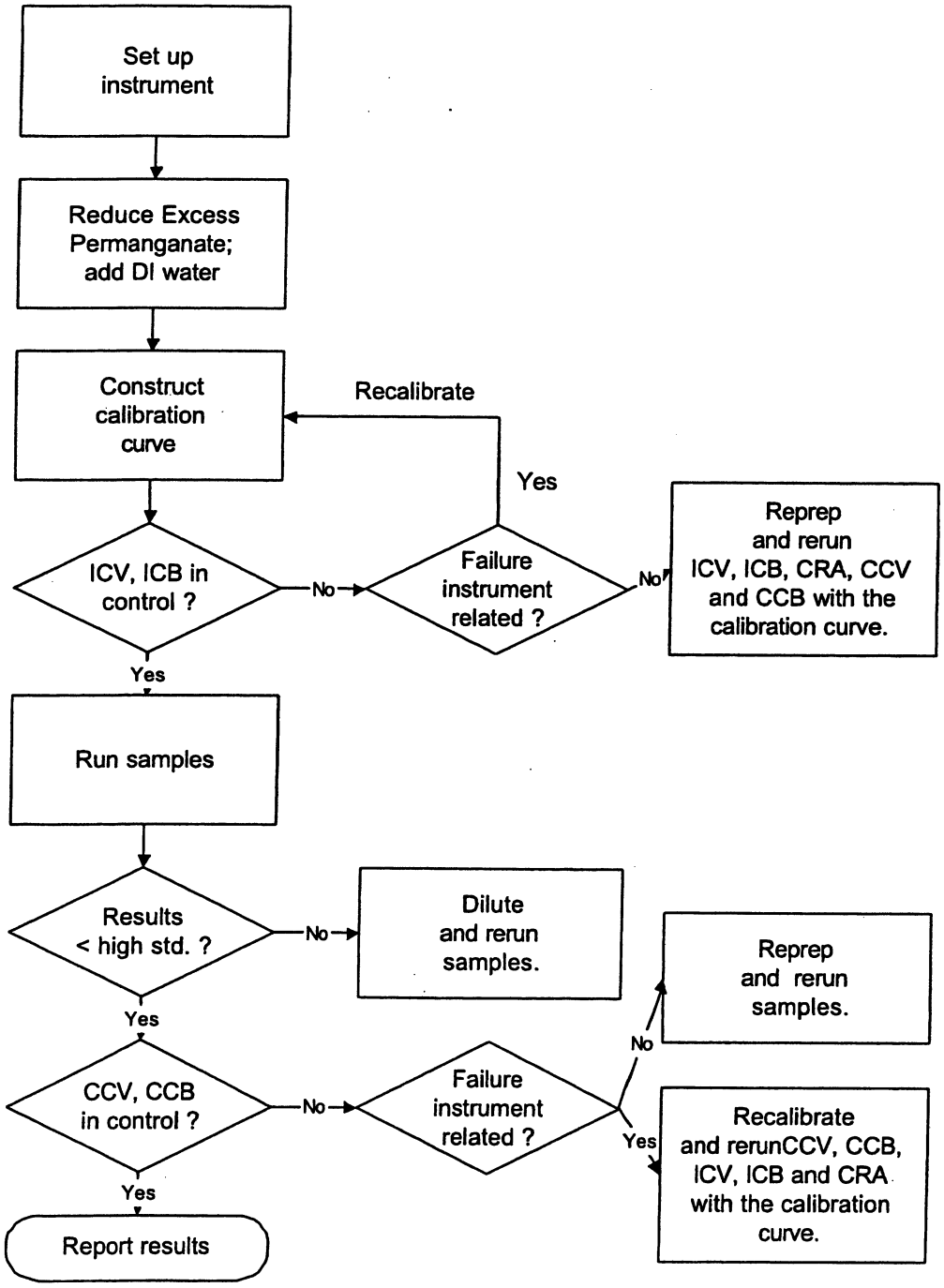


Figure 2. CVAA Mercury Analysis



APPENDIX A

TABLES

TABLE I. MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC STANDARD AND SPIKING LEVELS

Soil RL (mg/kg)	0.1
Std 0 (mg/L)	0
Std 1 (mg/L)	0.0002
Std 2 (mg/L)	0.0005
Std 3 (mg/L)	0.001
Std 4 (mg/L)	0.005
Std 5 (mg/L) **	0.010
ICV (mg/L)	0.001 or 0.0025 ***
CCV/LCS (mg/L)	0.0025 or 0.005 ***
MS (mg/L)	0.001

* **SOP specified calibration levels must be used unless prevented by the instrument configuration or client specific requirements. Deviations from specified calibration levels must be documented in the facility specific instrument operation SOP and must be approved by the facility technical manager and Quality Assurance Manager.**

** **Optional standard which may be used to extend the calibration range as allowed by the instrument configuration. If the instrument configuration prevents the use of 6 standards, the 2 ppb standard may be eliminated in favor of the 10 ppb standard.**

*** **Concentration level dependent on high calibration standard used. CCV must be 50% of the high standard concentration and the ICV must be 20-25% of the high standard concentration.**

TABLE II. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run.	90-110 % recovery.	Terminate analysis; Correct the problem; Recalibrate or reprep with calibration curve (see Section 9.7).
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate or reprep with calibration curve (see Section 9.7).
CCV	Every 10 samples and at the end of the run.	80 - 120 % recovery.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep with calibration curve (see Section 9.8).
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB or reprep with calibration curve (see Section 9.8).
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL do not require redigestion (See Section 9.4)	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.4 for additional requirements.

*See Sections 11.2.10 and 11.2.11 for exact run sequence to be followed.

TABLE II. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits.	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS (see Section 9.5).
Matrix Spike	One per sample preparation batch of up to 20 samples.	75 - 125 % recovery or in-house control limits. If the MS/MSD is out for an analyte, it must be in control in the LCS.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. (see Section 9.6) For TCLP see Section 11.3.12
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery or in-house control limits; RPD ≤ 20%. (See MS)	See Corrective Action for Matrix Spike.

APPENDIX B

EXAMPLE
STL NORTH CANTON Hg DATA REVIEW CHECKLIST

Example
STL North Canton Hg Data Review Checklist

Run/Project Information

Run Date: _____ **Analyst:**_____ **Instrument:**_____
Prep Batches Run:_____

Circle Methods used: **7470A / 245.1 :** CORP-MT-0005 Rev 1 **7471 / 245.5 :** CORP-MT-0007 Rev 1
CLP - AQ : CORP-MT-0006 Rev 0 **CLP - SOL :** CORP-MT-0008 Rev 0

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2ndLevel
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits?				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP)?				
4. CRA run (CLP only)?				
B. Sample Results				
1. Were samples with concentrations > the high calibration standard diluted and reanalyzed?				
2. All reported results bracketed by in control QC ?				
3. Sample analyses done within holding time?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/MDL data on file?				
3. Calculations and Transcriptions checked for error ?				
4. All client/ project specific requirements met?				
5. Date of analysis verified as correct ?				

Analyst: _____ **Date:**_____
Comments:
_____
_____
_____

2nd Level Reviewer : _____ **Date:**_____
_____
_____
_____

APPENDIX C

MSA GUIDANCE

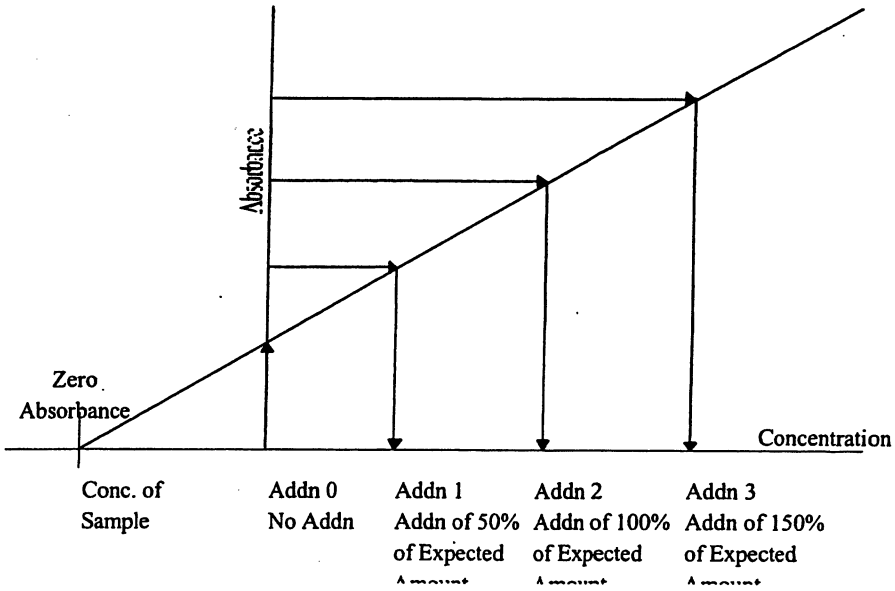
APPENDIX C. MSA GUIDANCE

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



- For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.
- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX D

TROUBLESHOOTING GUIDE

APPENDIX D. TROUBLESHOOTING GUIDE

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Wrong lamp Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak EDL power supply set on "Continuous"
Erratic Readings	Source lamp not aligned properly Lamp not prewarmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
EDL Won't Light	Lamp cable not plugged in Lamp power set at 0 Lamp is dead Power supply fuse is blown Short in cord
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell
Background Correction Light Blinking	Background screen or attenuator faulty

APPENDIX E

CONTAMINATION CONTROL GUIDELINES

APPENDIX E. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

APPENDIX F

PREVENTIVE MAINTENANCE

APPENDIX F. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption (Leeman PS 200) [1]

Daily	Semi-annually	Annually
Clean lens.	Check Hg lamp intensity.	Change Hg lamp.
Check aperture.		Check liquid/gas separator.
Check argon flow.		
Check tubing.		
Check drain.		
Replace drying tube.		

Cold Vapor Atomic Absorption (PE 5000) [1]

Daily	Monthly
Clean aspirator by flushing with DI water.	Clean cell in aqua regia.
Check tubing and replace if needed.	Clean aspirator in aqua regia.
Clean windows with methanol.	
Change silica gel in drying tube.	
Check argon gas supply.	
Adjust lamp.	

APPENDIX G

INSTRUMENT SET UP

Hg Analysis (Leeman PS200II)

SYSTEM INITIALIZATION AND WARM UP

1. F1 Menu
2. Instrument

a. Taskmaster

b. #4 Wake System Up Enter

The warming up period takes approximately 10 minutes.

TO SET UP INSTRUMENT FOR ANALYSIS

1. F1 Menu
2. Autosampler
   A. Rack Entry
   B. Edit (ex. Rack 1), Enter
   C. Cup ID - Enter (clears sample #'s)
   D. Extended ID- type in matrix of sample (water or solid), Enter.
   E. Press Insert Key and move cursor with arrows to cup ID and begin typing labels.
   F. F3 Print Screen
3. Press F2 Macro key and type in analyst's first name - Enter
   A. Enter folder name - ex. HG0306, Enter. If folder does not exist, type Y - Enter.
   B. Type in - "Rack 1", "Rack 2" etc. , Enter.

D. Type in : FROM CUP TO CUP

Ex. = 1 30

Do the same for position 2 if needed. If not needed, you must press Enter 3 times to begin analysis.

LABORATORY-SPECIFIC

QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: TOTAL ORGANIC CARBON (TOC) AND TOTAL INORGANIC CARBON (TIC)

(SUPERSEDES: REVISION 1, DATED 11/20/96)

Prepared by: Laurel E. Wood 6/1/99
Date

Reviewed by: Susan Macnaughton 6/1/99
Technology Specialist Date

Approved by: Gail Ann Pahr 6/7/99
Quality Assurance Manager Date

Approved by: [signature] 6-18-99
Environmental Health and Safety Coordinator Date

Approved by: [signature] 6-22-99
Laboratory Director Date

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Total Organic Carbon (TOC) or Total Inorganic Carbon (TIC) in waters and similar matrices. It is based on SW846 Method 9060 and EPA Method 415.1. The working linear range is instrument dependent at 1 mg/L to 50 mg/L with a reporting limit of 1 mg/L.

1.2. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

1.3. QuantIMS method codes are I8 (415.1 TIC), AY (9060 TIC), DA (415.1 – TOC) FM (9060 – TOC).

2. SUMMARY OF METHOD

2.1. Organic carbon is converted to carbon dioxide (CO_2) using chemical oxidation. The CO_2 is then measured by an infrared detector.

3. DEFINITIONS

3.1. Refer to the glossary in the Quality Assurance Management Plan (QAMP), current version.

4. INTERFERENCES

4.1. Contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts may cause Method interferences. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Carbonate and bicarbonate interfere but are eliminated by the acidification and purging step of the instrument.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfies ANSI Z87.1 (per the Chemical Hygiene Plan), Laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazard is known:

5.3.1. The following materials are known to be **corrosive:** Phosphoric **Acid, Sulfuric Acid.**

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable,** therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in/a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. O•I Corporation Model 1010 TOC Analyzer with 1051 vial multisampler and Printer

6.2. Nitrogen Gas and Regulator

6.3. Volumetric flasks: Various sizes

6.4. Volumetric pipettes: Various sizes

6.5. Vials: 40 mL glass

6.6. Graduated cylinders: Various sizes

6.7. pH Strips

6.8. Whatman filter #4

6.9. Top loading balance: capable of accurately weighing ± 0.01 g

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Sodium Persulfate: Reagent Grade

7.1.2. Sodium Persulfate Solution: Add 200 g sodium persulfate ($Na_2S_2O_8$) to a 1 liter volumetric flask and dilute to volume with reagent water.

7.1.3. Phosphoric Acid, concentrated: Reagent Grade

7.1.4. Phosphoric Acid Solution: Carefully add 59 mL concentrated phosphoric acid (H_2PO_4) to 900 mL of reagent water in a 1 liter volumetric flask. Dilute to volume with reagent water.

7.1.5. Sulfuric Acid, concentrated: Reagent Grade

7.2. Standards

7.2.1. All standards should be prepared in volumetric flasks, using volumetric pipettes, and diluted to volume with reagent water.

7.2.2. TOC Stock Standard

7.2.2.1. Primary and secondary sources are needed.

7.2.2.2. TOC 1000 mg/L

7.2.2.2.1. Dilute 1.06 g KHP (potassium acid phthalate) to volume in a 500 mL volumetric flask.

7.2.2.3. Prepare every six months.

7.2.3. TOC Calibration Standards

7.2.3.1. Prepare the following standards from the primary stock standard described in Section 7.2.2.2.1.

Concentration (mg/L)	Volume (mL)	Stock Concentration (mg/L)	Final Volume (mL)
50	5	1000	100
25	2.5	1000	100
10	1	1000	100
1	0.1	1000	100

7.2.4. TOC Verification Standard (LCS, MS/MSD and CCV)

7.2.4.1. Prepare the following standard from the secondary stock standard described in Section 7.2.2.2.1.

Concentration (mg/L)	Volume (mL)	Stock Concentration (mg/L)	Final Volume (mL)
25	12.5	1000	500

7.2.4.2. Prepare when setting up run.

7.2.5. TIC Stock Standard 1000 mg/L

7.2.5.1. Add 3.5 g Na_2HCO_3 (sodium bicarbonate) and 4.418 g Na_2CO_3 (sodium carbonate) to a 1000 mL flask. Dilute to volume with reagent water.

7.2.5.2. Prepare every 3 months.

7.2.6. TIC Calibration Standard

7.2.6.1. Prepare in the same manner as the TOC calibration standard (Section 7.2.3.1), using the TIC stock standard described in Section 7.2.5.1.

7.2.6.2. Prepare weekly.

7.2.7. TIC Verification Standard (LCS, MS/MSD and CCV)

7.2.7.1. Prepare in the same manner as the TOC verification standard (Section 7.2.4.1), using the TIC stock standard described in Section 7.2.5.1.

7.2.7.2. Prepare daily.

8. SAMPLE PRESERVATION AND STORAGE

8.1. Samples are preserved to a pH < 2 with sulfuric acid (H_2SO_4) or hydrochloric acid (HCl) and stored in plastic or glass containers at 4°C ± 2° C.

8.2. The holding time is twenty-eight days from sampling to analysis.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples, excluding QC samples (LCS, MS, MSD, and Method Blanks), which are processed similarly with respect to the procedure. All samples within the batch must be treated with the same lots of reagents and the same processes. All samples within the batch must be treated with the same processes.

9.2. Method Blank (MB)

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination

of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.2. A method blank consisting of 40 mL of reagent water and all reagents added to the samples must be prepared and analyzed with each batch of samples. The method blank is used to identify any background interference or contamination of the analytical system, which may lead to the reporting of elevated concentration levels or false positive data.

9.2.3. Corrective Action for Blanks

9.2.3.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be taken in consultation with the client and must be addressed in the project narrative.**

9.2.3.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

9.3. Laboratory Control Sample

9.3.1. Laboratory Control Samples are well characterized; laboratory generated samples used to monitor the laboratory's day to day performance of routine analytical methods. The LCS is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

9.3.2. A 25 mg/L LCS (TOC or TIC) must be analyzed with each batch of samples.

9.3.3. Corrective Action for LCS

9.3.3.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.3.1.1. The only exception to the requirement established in section 9.3.3.1 occurs if the LCS recovery is biased high and the associated sample(s) are ND (non detect). Under this condition, the batch is acceptable.

9.3.3.2. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. An MS/MSD consisting of 20 mL of sample and 20 mL of the 25 mg/L standard will be analyzed with each analytical batch of samples.

9.4.3. Corrective action for MS/MSDs

9.4.3.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

9.4.3.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported as DIL (diluted out).

9.4.3.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.5. QC Acceptance Criteria

9.5.1. Control limits are established by the laboratory as described in NC-QA-0018.

10. CALIBRATION AND BLANKING

10.1. Recommended Initial Setup

Contant Settings		
STD Mass	=	6.76 ug C
Sample Vol	=	2.0 mL
Acid Vol	=	4 x 100 uL
Oxidant Vol	=	10 x 100 uL

10.1.1. Adjust the nitrogen to 30 psi using the flow valve on the tank. The gauge should always be set at 30 psi when the instrument is not in use.

10.1.1.1. If the main valve reads < 200 psi, the tank should be changed. **Do not** allow the tank to go empty.

10.1.2. Remove the reagent bottles and fill with appropriate reagents (phosphoric acid solution and sodium persulfate). Do not fill bottles completely full; leave a small amount of air space. Loosely reconnect caps (and tubing), and replace the bottles into the instrument.

10.1.3. Blank and calibrate the instrument when CCVs and/or CCBs fail to meet acceptance criteria or when other problems are encountered.

10.1.3.1. Choose "sequences" from the "databases" menu option, open up the calibration template.

10.1.3.2. Confirm all information. If blanking is required, it is best if done at this step. Enter the desired number of blanks (no less than five) in the "reagent blanks before" field.

10.1.3.3. Analyze an ICV/ICB

10.1.3.4. Save the file using the current date as the filename.

10.1.3.5. Update data file information:

10.1.3.5.1. Choose the setup menu option, go into win TOC output, change the log fill name and prefix counter.

10.1.3.6. When analysis is complete, print the run from "/utilities/view run log"

10.1.3.7. Evaluate the data. The correlation coefficient of the original curve must be ≥0.995 or recalibration is required.

10.2. Continuing Calibration

10.2.1. The run is checked at the beginning, after every ten samples, and at the end of the run of the same species (TOC or TIC) using a midrange CCV made from a secondary source (Section 7.2.4 or 7.2.7) to verify continued linearity. A CCV cannot vary from the original curve by more than ± 10%, or recalibration is required.

10.2.2. System cleanliness is checked every ten samples and at the end of the run using a Continuing Calibration Blank (CCB). A CCB cannot contain the analyte of interest above the reporting limit, or recalibration is required.

10.3. Analytical Documentation

10.3.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

10.3.2. All standards are logged into a department standard logbook. All standards are assigned a unique number for identification. The supervisor or designee reviews logbooks.

10.3.3. Documentation, such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs, is available for each data file.

10.3.4. Sample results and associated QC are entered into the Laboratory Information Management System (LIMS) after final technical review.

11. PROCEDURE

11.1. Sample Preparation Procedure

11.1.1. Check the pH of the sample. If the pH > 2, add concentrated H_2SO_4 drop by drop to the sample (in the TOC vial) until the pH is < 2.

11.1.2. If excess particulate matter exists, filter an aliquot of sample through a Whatman #4 filter into a TOC vial.

11.2. Preparation Documentation

11.2.1. Record any sample preparation on the analytical logsheet.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Sample Analysis Procedure

12.1.1. Type a run protocol sequence into the computer using the run template, if desired. Update the data file information in the setup/winTOC output.

12.1.1.1. For method 9060, quadruplicate analysis is required. If quadruplicate reporting is requested each of four results are reported. Replicate analysis should be taken from separate vials, if available. If only one reportable result is requested per sample, the four results should be taken from one vial, and the average of the four results are reported.

12.1.1.2. For method 415.1 only one analysis is required. The single analysis result is reported directly from the instrument printout.

12.1.2. All samples and standards should be poured into 40 mL vials. Samples received in vials can be run in those containers, provided there is not an excess of solids.

12.1.3. Be sure the samples are loaded on the sampler, the first one positioned under the needle.

12.1.4. Click on the "Start" button.

12.1.5. Samples that fall outside the linear range (> 100 mg/L) of the instrument must be diluted and reanalyzed.

12.1.5.1. Samples following a high sample should be re-analyzed if carryover is a concern.

12.1.6. Print the run from "utilities/view run log."

12.1.7. When analysis is complete, properly dispose of or put away samples and standards.

12.2. Calculations

12.2.1. ***Total Organic Carbon, mg/L = Average Instrument Value x Dilution***

Where:

TOC, mg/L = average of the 4 instrument values x dilution, calculated (without dilution) by the instrument.*

12.2.2. ***LCS % Recovery =*** $\dfrac{\dfrac{\textit{Average of 4 instrument values*}}{25} \textit{ x 100}}{}$

12.2.3. ***MS/MSD % recovery***

$$\frac{\dfrac{\textit{(Avg of 4 instrument values* MS or MSD) - (Avg sample instrument value* ÷ 2)}}{\textit{12.5}}}{100} \text{ x}$$

**One of the values may be judged erroneous and disregarded if three of the four are consistent. If no consistency can be found in the four values, the sample must be rerun.*

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. The group/team leader has the responsibility to ensure that an associate who has been properly trained in its use and has the required experience performs this procedure.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Acid waste must be collected in clearly labeled acid water containers.

15.2. Solid materials (gloves, soiled paper products) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.3. Refer to the Laboratory Sample and Waste Disposal plan.

15.4. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of Quanterra. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by annual refresher training.

16. REFERENCES

16.1. References

16.1.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Total Organic Carbon, Method 9060

16.1.2. EPA 600, Methods for Chemical Analysis of Water and Wastes, Organic Carbon, Method 415.1

16.1.3. Quanterra® Quality Assurance Management Plan (QAMP), current version.

16.2. Associated SOP

16.2.1. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Reporting limits

17.1.1. The lower reporting limit is 1 mg/L.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Troubleshooting guide

17.2.1. See the manufacturer's instructions for an instrument troubleshooting guide and maintenance requirements.

17.3. Deviations from the Method

17.3.1. A blender is not used to homogenize samples.

STL STANDARD OPERATING PROCEDURE

TITLE: CYANIDE PREPARATION METHOD

(SUPERSEDES: REVISION 8.1, DATED 03/20/00)

Prepared by: _____ 6/5/01 Date

Reviewed by: _____ 6/5/01 Date
Technology Specialist

Approved by: _____ 6/5/01 Date
Quality Assurance Manager

Approved by: _____ 6/5/01 Date
Environmental Health and Safety Coordinator

Approved by: _____ 6-5-01 Date
Laboratory Director

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Total, Amenable, and Free Cyanide in solids, liquids, and waters. It is based on CLP ILM03.3, CLP ILM04.0, SW 846 Method 9012A, EPA 335.1, 335.2, 335.4, Standard Method 4500-CN-I and ASTM D 4282-83. The working linear range is 0.005 - 0.1 mg/L for waters and 0.25 to 5 mg/kg for solids.

1.2. The associated method codes are CLP ILM03.0 (DV), CLP ILM04.0 (OU), SW846 Method 9012A (QP), ASTM D4282-83, EPA 335.1 and 335.2 (CG), EPA 335.4 (O2), and Standard Method 4500-CN-E (A4).

1.3. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

2. SUMMARY OF METHOD

2.1. The Cyanide, as HCN, is released by distilling/refluxing the sample with strong acid and is trapped in a sodium hydroxide solution.

3. DEFINITIONS

3.1. Refer to the glossary in the Laboratory Quality Manual (LQM), latest version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Oxyidizing agents such as chlorine will decompose most cyanides. Sulfide will distill over with the cyanide and could affect colorimetric, titrimetirc, and electrode procedures. Refer to the preparation section on how to screen and treat samples appropriately.

4.3. Aldehydes convert cyanide to cyanohydrin which could result in the lost of cyanide. If the presence of aldehydes are suspected, stabilize the sample with NaOH at the time of collection and add 2 mL 3.5% ethylenediamine solution per 100 mL of sample.

4.4. Refer to Standard Methods for further information on possible interferences and associated treatments.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

5.2. Eye protection that satisfies ANSI Z87.1 (per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. The following are known to be **corrosive: sulfamic acid, sodium hydroxide, sulfuric acid, acetic acid.**

5.3.2. Cyanide is a highly toxic compound that must be handled with care. The analyst must be aware of the handling and clean-up techniques before using this material.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable,** therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Cyanide Distillation Apparatus

6.2. Analytical balance, capable of accurately weighing ± 0.0001 g

6.3. Vacuum pump

6.4. Graduated cylinders: various

6.5. Volumetric flasks: various

6.6. Volumetric pipets: various

6.7. Balance: Top loading, capable of accurately weighing ± 0.01 g

6.8. Lead Acetate Indicator Paper

6.9. Potassium Iodide (KI) Indicator Paper

6.10. Erlenmeyer flasks: various

6.11. Buret: Class A 10 mL

6.12. pH strips

6.13. Boiling stones or chips

6.14. Beakers: various

6.15. Snap seal containers: 120 mL

6.16. Plastic bottles with lids: 250mL or 500mL

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Sulfamic Acid: reagent grade (not used for CLP ILM03.0)

7.1.2. Sulfamic Acid Solution: Add 100 g of sulfamic acid to 800 mL reagent water and dilute to 1 liter with reagent water (not used for CLP ILM03.0)

7.1.3. Ascorbic Acid: reagent grade

7.1.4. Sodium Hydroxide: (NaOH), high purity grade.

7.1.5. Sodium Hydroxide, 1.25 N: Add 50 g of NaOH to 900 mL and dilute to 1 liter with reagent water.

7.1.6. Sodium Hydroxide, 0.25 N: Add 10 g of to 900 mL reagent water and dilute to 1 liter with reagent water.

7.1.7. Sulfuric Acid: (H_2SO_4), concentrated

7.1.8. Acetic Acid Solution: Add 10 mL of glacial acetic acid to 90 mL of reagent water.

7.1.9. Magnesium Chloride: ($MgCl_2 \bullet 6H_2O$), reagent grade

7.1.10. Magnesium Chloride Solution: Add 510 g $MgCl_2 \bullet 6H_2O$ to 500 mL reagent water and dilute to 1 liter with reagent water.

7.1.11. Calcium Hypochlorite: [Ca $(OC1)_2$], reagent grade

7.1.12. Calcium Hypochlorite Solution: Add 5 g of $Ca(OC1)_2$ to 100 mL of reagent water.

7.1.13. Methyl Red Indicator: Add 0.05 g of methyl red to 50 mL of glacial acetic acid and dilute to 100 mL with reagent water.

7.1.14. Methyl red reagent grade glacial

7.1.15. Acetic Acid: (CH_3COOH), glacial reagent grade

7.1.16. Zinc Acetate: [$Zn(C_2H_3O_2)$], reagent grade

7.1.17. Zinc Acetate Solution: Add 100 g zinc acetate to 800 mL reagent water and dilute to 1 liter with reagent water.

7.1.18. Sodium Acetate: [$NaC_2H_3O_2 \bullet 3H_2O$] reagent grade

7.1.19. Sodium Acetate Buffer: Add 410 g of sodium acetate to 500 mL of reagent water. Adjust the pH to 4.5 using glacial acetic acid and dilute to 1 liter with reagent water.

7.1.20. Rhodanine: reagent grade

7.1.21. 0.0192 N Silver Nitrate: reagent grade

7.1.22. Bismuth nitrate [$Bi(NO_3)_3$]: Dissolve 30 g of $Bi(NO_3)_3$ in 100 mL of reagent water. While stirring, add 250 mL of glacial acetic acid. Stir until dissolved. Dilute to 1 L with reagent water.

7.1.23. Cadmium chloride [$CdCl_2$] – anhydrous : Dissolve 1.0 g $CdCl_2$ in 90 mL of reagent water. Dilute to 100 mL with reagent water to produce a 10 g/L $CdCl_2$ solution.

7.2. Standards

7.2.1. Secondary Source Cyanide Stock Standard, 1000 mg/L: Add 2.51 g of potassium ferricyanide (KCN) and 2.0 g of potassium hydroxide (KOH) to a 1000 mL volumetric flask and dilute to volume with reagent water. Mix well. Prepare fresh every 1 - 3 months.

7.2.2. Note: Cyanide stock standard must be standardized prior to use (See Section 7.3) Working Standards: Add the required amounts of 1.25 N NaOH and cyanide standards to volumetric flasks according to the suggested chart. Dilute to volume using reagent water. See chart below:

Working Standards					
Volume 1.25 N NaOH (mL)	Std Volume (mL)	Std Conc. (mg/L)	Final Volume (mL)	Final Conc. (mg/L)	Expiration Time (Days)
20	10	1000	100	100	14
20	10	100	100	10	7
20	10	10	100	1	1

7.3. Cyanide Standardization

7.3.2. Using a volumetric pipet, aliquot 10.0 mL of the 1000 ppm stock cyanide standard into an Erlenmeyer flask and add 90 mL of reagent water.

7.3.3. Add 10 drops of Rhodanine indicator.

7.3.4. Titrate with 0.0192 N Silver nitrate until the color changes from yellow to pink/orange.

7.3.5. Calculation:

$$\text{Cyanide, mg/L} = \frac{(A)\ (1000)}{\text{mL cyanide solution (10)}}$$

Where: A = *mL Titrant for standard*

7.3.6. If the cyanide concentration is not 1000 ppm, adjust concentration accordingly.

8. SAMPLE PRESERVATION AND STORAGE

8.1. Solid and liquid samples are not chemically preserved. Water samples are preserved with NaOH to a pH>12. All samples are stored at 4° C ± 2°C in plastic or glass containers.

8.2. The holding time is fourteen days from sampling to analysis.

NOTE: CLP samples holding time is twelve days from receipt to analysis.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same process.

9.2. Method Blank (MB)

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.2. A blank consisting of 50 mL reagent water must be prepared and distilled (or diffused, 3 mL) with each analytical batch of samples.

9.2.3. Corrective Action for Blanks

9.2.3.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.3.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. A midrange LCS consisting of 2.0 mL of the 1.0 mg/L standard (secondary source) brought to a final volume of 50 mL must be distilled with each analytical batch of samples.

9.3.3. Corrective Action for Laboratory Control Samples

9.3.3.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.3.2. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. A MS/MSD consisting of 50 mL or 1.0 g of sample brought up to 50 mL with reagent water and 2.0 mL of the 1.0 mg/L standard (either source) must be distilled with each analytical batch of samples.

9.4.2.1. **Note:** MS for CLP samples require 5 mL of 1.0 mg/l standard (true 100ug/L)

9.4.3. Corrective action for MS/MSDs

9.4.3.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

9.4.3.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data reported as DIL (diluted out).

9.4.3.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.5 Control Limits

9.5.1 Control limits are established by the laboratory as described in SOP, NC-QA-0018.

9.5.2 Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are listed in the Laboratory Quality Manual (LQM) and the latest is version easily accessible via the LIMs (QC Browser program).

9.6 Method Detection Limits (MDLs) and MDL Checks

9.6.1 MDLs and MDL Checks are established by the laboratory as described in SOP, NC-QA-0021.

9.6.2 MDLs are listed in the Laboratory Quality Manual (LQM) and the latest version is easily accessible via the LIMs (QC Browser program).

9.7 Nonconformance and Corrective Action

9.7.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager/

10. CALIBRATION AND STANDARDIZATION

10.1. For all non-CLP methods, a low and high standard are distilled from the same source as the calibration curve each day. Prepare the low standard (0.025 mg/L) by diluting 0.125 mL of 10.0 mg/L standard with reagent water to a final volume of 50 mL. Prepare the high standard (0.075 mg/L) by diluting 0.375 mL of a 10.0 mg/L standard with reagent water to a final volume of 50mL. Method 9012A *recommends* that the distilled standards (a high and a low) be compared to the curve with a ±10% agreement with the undistilled standards. The distilled standards are evaluated against all applicable batch QC.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described

11.3 Summary

11.3.1. For all non-CLP methods, the sample is distilled/refluxed under acidic conditions for one hour. For CLP and EPA 335.4 methods, the samples are distilled for one and a half hours. The released HCN is trapped in 0.25 N NaOH solution.

11.4. Sample Preparation Procedure applicable for use with the Midi distillation unit.

11.4.1. Checking for Interferences (For non-CLP samples)

11.4.1.1. Using pH paper strips, check the pH of the sample and record it as >12 or <12 on the analytical logsheet. If pH is <12, the deviation **must be addressed in the project narrative.**

11.4.1.2.For ALL samples (non-CLP and CLP samples): Test each sample for the presence of sulfides using lead acetate paper. If sulfides are present, treat the sample with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution. Avoid a large excess of cadmium carbonate and long contact time in order to minimize loss by complexation or occlusion of cyanide on the precipitated material.

11.4.2. Complex Cyanide Preparation Procedure [THIS PROCESS IS PERFORMED ONLY WHEN REQUESTED BY THE CLIENT]: Add 100 mL of water sample or 2.0 g of solid sample and 100 mL reagent water to a plastic, sealable container. Add 10 g NaOH pellets. Mix or tumble for 12-16 hours. Allow solids to settle out. Transfer 50 mL of the solution to the distillation tube. Distill sample (See section 11.2.4.1).

Note: If the sample requires an MS/MSD or MS/Dup, double or triple the volumes as stated above. A blank and LCS should be prepared and analyzed with samples using 10 g NaOH and 100 mL reagent water.

11.4.3. Amenable Cyanide (Chlorinated Aliquot) [THIS PROCESS IS PERFORMED ONLY WHEN REQUESTED BY THE CLIENT]

11.4.3.1.Place 50 mL (waters) or 1.0 g (solids/liquids) into a beaker. Add 50 mL of reagent water to non-waters. Place the beaker on a stir plate and begin mixing. Test the pH of the solution, if less than 12 add 1.25 N NaOH, drop by drop until pH $\geq$ 12. Drop by drop, add calcium hypochlorite until an excess of chlorine is reached. Test for chlorine excess using KI paper. Allow the sample to chlorinate for one hour. At the end of the chlorination period, add about 0.1 to 0.5 g of ascorbic acid to destroy excess chlorine. Test using KI paper. Keep the addition of ascorbic acid to a minimum. Pour the sample into a distillation flask and follow the total cyanide preparation method (Section 11.2.4). Also set up an unchlorinated aliquot of sample (50 mL or 1.0 g) following the total cyanide method.

11.4.4. Total Cyanide

11.4.4.1. Add 50 mL or 1.0 g of the sample and 2 - 3 boiling chips to the distillation tube. Bring all volumes to 50 mL with reagent water. Add 50 mL of 0.25 N NaOH solution to the absorber tube and assemble the scrubber. Assemble the cyanide distillation apparatus.

NOTE: If solid samples are being prepared under the Michigan program, the initial solid weight is 2.5 g

11.4.4.2. Turn on the cooling water to "60". Turn on the vacuum source and adjust the flow such that even stream of air bubbles are in the scrubber tube approximately 1/4 " of foam or 1-2 bubbles per second. At this time add any spiking solutions to the LCS or MS/MSDs samples directly into the inlet tube.

11.4.4.3. In the order stated, add 2.0 mL sulfamic acid solution (not for CLP ILM03.0 samples), 2.5 mL of concentrated sulfuric acid and 2.0 mL of magnesium chloride solution to the distillation tube. Be sure to rinse the inlet tube sparingly with reagent water between and after reagent additions.

Note: Complex cyanide samples require an additional 2.5 mL of sulfuric acid. Be sure the pH is < 2.

11.4.4.4. Flip the heater switch to "on" and turn the dial to an appropriate setting (according to manufacturer's instructions) to allow the apparatus to warm up. Be sure to adjust the air flow and water as necessary. Heat for one hour. After the heating period, the heater will turn off automatically. Allow to cool for fifteen minutes. Keep the vacuum and cooling water on.

NOTE: For CLP, heat samples for one and a half hours.

11.4.4.5. Disconnect the absorber. Pour solution into a 120 mL snap seal container. Do not rinse the scrubber tube or dilute NaOH in the snap seal. Be sure to properly label the bottles "total" or "amenable" along with sample ID, position and date.

11.4.5. Free Cyanide (Weak and Dissociable) [THIS PROCESS IS PERFORMED ONLY WHEN REQUESTED BY THE CLIENT]

11.4.5.1. Add 50 mL or 1.0 g of sample to the distillation tube. Bring all volumes up to 50 mL with reagent water. Add 50 mL of 0.25 N NaOH to the absorber tube and assemble.

11.4.5.2. Turn on cooling water to "60". Turn on the vacuum source. Add any spiking standards to the appropriate LCS or MS/MSDs at this time. Through the inlet tube, add 1.0 mL of sodium acetate buffer, 1.0 mL of zinc acetate, and 0.25 mL of methyl red indicator. Rinse the inlet tube sparingly with reagent water between and after reagent additions. If the sample is not red, carefully add 1:9 acetic acid dropwise until the sample does turn red.

11.4.5.3. Flip on the heater switch and turn the dial to "10.5" (This allows the apparatus to warm up). Adjust the air flow and water as needed. Heat for 1 hour. After the

heating period, the heater will shut off automatically. Allow the sample to cool for fifteen minutes with the air and water on.

11.4.5.4. Pour the scrubber contents into a 120 mL snap seal bottle. Do not rinse the scrubber. Label the bottle well and be sure to denote that it is a "free" sample distillate.

11.4.6 Cyanide Unit Clean-up

11.4.6.1 Cyanide distillation unit glassware is very fragile and expensive. It must be handled with care at all times.

11.4.6.2 Disassemble each set-up and rinse the sample down the drain with large amounts of water. Be sure to collect the solids in a screen and dispose of properly.

11.4.6.3 Wash each set-up with soap and hot water, and then rinse with 1:1 HCl. Rinse several times with reagent water. **After preparing samples found to be extremely high in cyanide, a subsequent rinse with 1.0N NaOH, followed by further rinsing with reagent water can help rinse away any residual cyanide.

11.4.6.4 Re-assemble the set-up.

11.4.6.5 Be sure to wash each set up as a separate unit and replace in the same position.

11.4.6.6 If a sample of known high cyanide concentration was distilled in a certain position, be sure to change the appropriate tubing on that position.

11.5 Analytical Documentation

11.5.1 Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.5.2 All standards are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3 Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4 Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files

14. POLLUTION PREVENTION

14.1. Using a Midi Cyanide distillation unit saves time and requires less sample and reagents for use.

15. WASTE MANAGEMENT

15.1. Acid waste must be collected in clearly labeled acid waste cans.

15.2. Solid waste materials (soil, gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.3. Refer to the Laboratory Sample and Waste Disposal plan

15.4. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1. References.

16.1.1. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Total and Amenable Cyanide, Automated UV; Method 9012A.

16.1.2. EPA 600; Cyanide, Total and Cyanide, Amenable to Chlorination; Methods 335.1 and 335.2

16.1.3. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition; Weak and Dissociable Cyanide; Method 4500-CN-I

16.1.4. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition; Complex Cyanide; Method 4500-CN-E

16.1.5. USEPA CLP SOW ILM03.0 Section D-Cyanide Midi Distillation

16.1.6. Corporate Quality Management Plan (QMP), current version

16.1.7. STL Laboratory Quality Manual (LQM), current version

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2 Glassware Washing, NC-QA-0014

16.2.3 Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4 Method Detection Limits and Instrument Detection Limits, NC-QA-0021

16.2.5 Navy/Army SOP, NC-QA-0016

16.2.6 Cyanide, Automated Method NC-WC-0031

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. BASF RFI Requirements

17.1.1. Amendments to this SOP to satisfy BASF RCRA Facility Investigation (RFI) requirements are based on comments from US EPA upon review of site requirements and laboratory procedures.

17.1.2. Amendment to the preparation of **all** samples

17.1.2.1. **Section 7.2.1**: Change 2.51 g of potassium cyanide to 2.11g potassium ferricyanide.

17.1.2.2. **Section 9.4**: The matrix spike/duplicate frequency has been increased from one per batch to one per 7 samples. Consult with the project manger for specific samples to be spiked. The goal is to spike each sampling area at the site at least once.

17.1.3. Amendment to the preparation of **Prussian blue area** samples

17.1.3.1. **Section 7.1.22**: Sodium thiocyanate (reagent grade): Prepare a spiking solution at a concentration 100X greater than the measured cyanide for the appropriate Prussian blue area samples.

17.1.3.2. **Section 9.5**: Three Prussian blue area samples will be spiked with 1 mL of the sodium thiocyanate solution (Section 17.1.3.1) in order to assess the potential impact of thiocyanate in the original samples on the measured cyanide concentration. These matrix spikes do not replace the cyanide matrix spikes mentioned above (Section 17.1.2.2). The increase (if any) in measured cyanide concentration relative to the original sample shall be reported as "percentage increase" using the following equation:

$$\%CN_{increase} = \frac{Conc_{SCN_spike} - Conc_{unspiked}}{Conc_{unspiked}} \times 100$$

where

$Conc_{SCN_spike}$ = *cyanide concentration measured in the thiocyanate spiked sample*

$Conc_{unspiked}$ = *cyanide concentration measured in the unspiked sample*

Note: Section 11.3.2.1 of NC-WC-0031 addresses sulfide testing and precipitation.

17.2. Method Deviation (9012A, 335.1, 335.2)

17.2.1. The reflux distillation apparatus used is the midi distillation.

17.2.2. The volume of sample used is reduced to 50 mL vs. 500 mL using the midi-distillation apparatus.

17.2.3. Method of Standard Addition is not performed for samples with matrix interference (sulfides).

APPENDIX A

Cyanide Distillation Form

(Example)

APPENDIX A

N:\WET-LOG\CYANIDE

QUANTERRA- NORTH CANTON
CYANIDE DISTILLATION LOGSHEET

PAGE 1 OF 2

Batch No. 0040131 waters 0040130 leach
Prep/Anal. Date 000207 000209
Start Time: 2 30 pm

LCS&MS/MSD No. CY0117
LCS Prep Date: 000208
End Time: 3:30 pm

SDG No. —
Analyst: MFG

Pos.	Sample No.	Type T	C	F	Sample Volume	Final Volume(mL)	pH >12	S	Cl2	Comments
1A	BLK	T	C	F	50mL	50mL	—			D8CDF B/C
2	LCS	T	C	F			—			2ml CY0117 1ppm 2°
3	LCSD	T	C	F			—			1 D8CDE 0040130
4	.025	T	C	F			—			.125ml CY0105 10ppm 1°
5	.075	T	C	F			—			.375ml CY0105 10ppm 1°
6	D833X	T	C	F			✓	✓	✓	
7	D833X MS	T	C	F			—			2ml CY0117 1ppm 2°
8	D833X MSD	T	C	F			—			" "
*9	D851G MS	T	C	F			—			" "
*10	D851G MSD	T	C	F			—			" "
*1A	D851G	T	C	F			✓	✓	✓	
*2	D851K	T	C	F			✓	✓	✓	
*3	leach blk	T	C	F			✓	✓	✓	D8CDE 1.01 0040130
4	D81MG	T	C	F			✓	✓	✓	
5	D81MK --	T	C	F			✓	✓	✓	
6	D81ML	T	C	F			✓	✓	✓	
7	D83RM	T	C	F			✓	✓	✓	
8	D833H	T	C	F			✓	✓	✓	
9	D833P	T	C	F			✓	✓	✓	
10	D83RI	T	C	F			✓	✓	✓	

Type: T=Total C= Chlorinated F= Free

S and Cl2 column- a check mark in these columns means the sample was checked for this interference.

If the interference is present, the analyst will address this in the comments section.

* leaches came down on 2.4.00

Level Review X
Calibration
Calculation
Date: 2.8.00

Level Review X
Calibration
Calculation
Date: 2.8.00

Rev 5-16-97

APPENDIX A

N:\WET-LOG\CYANIDE

QUANTERRA- NORTH CANTON
CYANIDE DISTILLATION LOGSHEET

PAGE 2 OF 2

Batch No. 0040131 waters 0040130 leaches
Prep/Anal. Date 000208 / 000209
Start Time: 2:30 pm

LCS&MS/MSD No. CR017
LCS Prep Date: 000208
End Time: 3:30pm

SDG No. —
Analyst: MFG

Pos.	Sample No.	Type T C F	Sample Volume	Final Volume(mL)	pH >12	S	Cl2	Comments
B 1X	D83R6	(T) C F	50 mL	50mL	✓	✓	✓	
2	D83R8	(T) C F			✓	✓	✓	
3	D856F	(T) C F			✓	✓	✓	
4	D856Q	(T) C F			✓	✓	✓	
5		T C F						
6		T C F						
7		T C F						
8		T C F						
9		T C F						
10		T C F						
1B		T C F						
2		T C F						
3		T C F						
4		T C F	MFG 000208					
5		T C F						
6		T C F						
7		T C F						
8		T C F						
9		T C F						
10		T C F						

Type: T=Total C= Chlorinated F= Free
S and Cl2 column- a check mark in these columns means the sample was checked for this interference.
If the interference is present, the analyst will address this in the comments section.

Level Review X
Calibration
Calculation
Date 2-8-00

Level Review X
Calibration ✓
Calculation ✓
Date 2-9-00

Rev. 5-16-97

Controlled Copy
Copy No:_____

Implementation Date: 10/16/00

SOP No: NC-WC-0084
Revision No: 3
Revision Date: 10/03/00

STL STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPHY

(SUPERSEDES: REVISION (2))

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Date

Reviewed by: Kurt C. Lee 10/5/00
Technology Specialist Date

Approved by: Gail Ann Poh 10/5/00
Quality Assurance Manager Date

Approved by: [signature] 10-10-00
Environmental Health and Safety Coordinator Date

Approved by: Christy A Grandi 10/11/00
Laboratory Director Date

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1. SCOPE AND APPLICATION

1.1. This method covers the determination of fluoride, chloride, nitrite, bromide, nitrate, ortho-phosphate and sulfate in drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, solids (after extraction 11.7) and leachates (when no acetic acid is used).

1.2. A listing of associated LIMs method codes is located in Section 8.2.

1.3. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.

2. SUMMARY OF METHOD

2.1. A 25 uL volume of sample is introduced into the ion chromatograph. The sample is pumped through three different ion exchange columns and into a conductivity detector. The first two columns, a precolumn or guard column and a separator column, are packed with low-capacity, strongly basic anion exchange resin. Ions are separated into discrete bands based on their affinity for the exchange sites of the resin. The last column is a suppresser column that reduces the background conductivity of the eluent to a low or negligible level and converts the anions in the sample to their corresponding acids. The separated anions in their acid form are measured using an electrical conductivity cell. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a calibration curve generated from known standards.

3. DEFINITIONS

3.1. Refer to the glossary in the Laboratory Quality Manual (LQM).

4. INTERFERENCES

4.1. Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.

4.2. The water dip or negative peak that elutes near, and can interfere with, the fluoride peak can usually be eliminated by the addition of concentrated eluent to each standard and sample.

4.3. Method interferences may be caused by contaminants in the reagent water, reagents, glassware and other sample processing apparatus that lead to discrete artifacts or an elevated baseline in the ion chromatograms.

4.4. Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other small organic anions. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant; however, it is the responsibility of the user to generate precision and accuracy information in each sample matrix.

4.5. The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

5.2. Eye protection that satisfied ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore; unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation when possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Balance -- Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.2. Ion Chromatograph -- Analytical system complete with ion chromatograph and all required accessories including analytical columns, compressed gases and detectors.

6.2.1. Anion guard column: A protector of the separator column. If omitted from the system the retention times will be shorter. Usually packed with same substrate as the separator column. 4 x 50 mm, Dionex IonPac AG14 P/N 46134, or equivalent.

6.2.2. Anion separator column: The separation shown in Figure 1 was generated using a Dionex IonPac AS14 column (P/N 46134). Equivalent column may be used if comparable resolution is obtained, and the requirements of Sect. 9.2 can be met.

6.2.3. Anion suppresser device: Dionex anion micro membrane suppresser (P/N 37106) or ASRS-Ultra Self-Regenerating Suppressor (4mm) P/N 53946 or equivalent.

6.2.4. Detector -- Conductivity cell: approximately 1.25 uL internal volume, Dionex, or equivalent.

6.2.5. Dionex –PeakNet 5.1Data Chromatography Software or equivalent.

6.3. Assorted laboratory glassware (pipettes, volumetric flasks, etc.).

7. REAGENTS AND STANDARDS

7.1. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2. Reagent water: Distilled or deionized water, free of the anions of interest. Water should contain particles no larger than 0.20 microns.

7.3. Eluent solution: sodium bicarbonate (CASRN 144-55-8) 1.0 mM, sodium carbonate (CASRN 497-19-8) 3.5 mM. Dissolve 1.680 g sodium bicarbonate ($NaHCO_3$) and 7.417 g of sodium carbonate (Na_2CO_3) in reagent water (7.2) and dilute to 100 mL in a volumetric flask. Take 10 mL of this concentrated eluent solution and dilute to 2 L for use as the working eluent solution or dissolve the entire bicarbonate/carbonate amount in 20 L of reagent water.

7.4. Stock solutions (1,000 mg/L): All stocks may be prepared as described below or purchased from commercial sources. Primary and secondary sources are required for each target analyte.

7.4.1. Fluoride stock solution (1.00 mL = 1.00 mg F^-): In a 1 liter volumetric flask, dissolve 2.2100 g of sodium fluoride (NaF) in reagent water, and dilute to volume with reagent water. Store in chemical- resistant glass or polyethylene.

7.4.2. Chloride stock solution (1.00 mL = 1.00 mg Cl^-): Dry sodium chloride (NaCl) for 12 hours at 105°C, and cool in a desiccator. In a 1 liter volumetric flask, dissolve 1.6485 g of the dry salt in reagent water and dilute to volume with reagent water.

7.4.3. Nitrite stock solution (1.00 mL = 1.00 mg NO_2^- - N): Place approximately 10.0 g of sodium nitrite (KNO_2) in a 125 mL beaker and dry to constant weight (about 24 hours) in a desiccator. In a 1 liter volumetric flask, dissolve 6.0790 g of the dried salt in reagent water and dilute to volume with reagent water. Store in a sterilized glass bottle. Refrigerate and prepare monthly.

- Nitrite is easily oxidized, especially in the presence of moisture, and only fresh reagents are to be used.
- Prepare sterile bottles for storing nitrite solutions by heating for 1 hour at 170°C in an air oven.

7.4.4. Bromide stock solution (1.00 mL = 1.00 mg Br^-): Dry approximately 5.0 g of sodium bromide (NaBr) for 12 hours at 105°C, and cool in a desiccator. In a 1 liter volumetric flask, dissolve 1.2876 g of the dried salt in reagent water and dilute to volume with reagent water.

7.4.5. Nitrate stock solution (1.00 mL = 1.00 mg NO_3^- - N): Dry approximately 10.00 g of sodium nitrate (KNO_3) at 105°C for 24 hours . In a 1 liter volumetric flask, dissolve 7.2200 g of the dried salt in reagent water and dilute to volume with reagent water.

7.4.6. Phosphate stock solution (1.00 mL = 1.00 mg PO_4 - P): Dry approximately 10.00 g of potassium dihydrogen phosphate (KH_2PO_4) for 1 hour at 105°C and cool in a desiccator. In a 1 liter volumetric flask, dissolve 4.3937 g of the dry salt in reagent water and dilute to volume with reagent water.

7.4.7. Sulfate stock solution (1.00 mL = 1.00 mg SO_4^{-2}): Dry approximately 5.00 g of potassium sulfate (K_2SO_4) at 105°C for 1 hour and cool in a desiccator. In a 1 liter volumetric flask, dissolve 1.8141 g of the dried salt in reagent water and dilute to volume with reagent water.

7.4.8. Commercial stock solution A: F^- - 25 mg/L, Cl^- - 500 mg/L, Br^- - 100 mg/L, NO_3^- - N- 25 mg/L, PO_4 – P - 25 mg/L, SO_4^{-2} - 500 mg/L

7.4.9. Commercial stock solution B: NO_2^- - N- 25 mg/L

7.4.10. Commercial IC Spike solution A: : F^- - 125 mg/L, Cl^- - 2500 mg/L, Br^- - 500 mg/L, NO_3^- - N- 125 mg/L, PO_4 – P - 125 mg/L, SO_4^{-2} - 2500 mg/L

7.4.11. Commercial IC Spike solution B: NO_2^- - N- 125 mg/L

7.5. Working standards: Prepare calibration standard #5 in a 10 mL volumetric flask and transfer to a vial. Adjust the amount of stock solution used to prepare the working standards if the stock concentration differs from 1000 mg/L as assumed Alternatively prepare Cal standard #5 by mixing 4.0 mL commercial stock A, 4.0 mL commercial stock B and 2.0 mL of reagent water.

Calibration Standard #5

Analyte	mL of Stock	Final Conc.
Fluoride	0.10mL	10.0 mg/L
Chloride	2.0 mL	200. mg/L
Nitrite	0.10 mL	10.0 mg/L
Bromide	0.40 mL	40.0 mg/L
Nitrate	0.10 mL	10.0 mg/L
Ortho-Phosphate	0.10 mL	10.0 mg/L
Sulfate	2.0 mL	200. mg/L

7.5.1. In 5 mL PolyVials prepare the following calibration standards in reagent grade water. Final concentrations of working standards are shown below.

Calibration Standard #4: take 2.50 mL of calibration standard #5 and add 2.50 mL of reagent water.

Calibration Standard #2: take 250 µL of calibration standard #5 and add 4.75 mL of reagent water.

Calibration Standard #1: take 25.0 µL of calibration standard #5 and add 4.95 mL of reagent water.

Calibration Standard #3: take 1.25 mL of calibration standard #5 and add 3.75 mL of reagent water.

Calibration Standard #1

Analyte	25.0 µL of Cal Std #5	Final Conc
Fluoride		0.05 mg/L
Chloride		1.0 mg/L
Nitrite		0.05 mg/L
Bromide		0.20 mg/L
Nitrate		0.05 mg/L
Ortho-Phosphate		0.05 mg/L
Sulfate		1.0 mg/L

Calibration Standard #2

Analyte	250 µL of Cal Std #5	Final Conc.
Fluoride		0.5 mg/L
Chloride		10. mg/L
Nitrite		0.5 mg/L
Bromide		2.0 mg/L
Nitrate		0.5 mg/L
Ortho-Phosphate		0.5 mg/L
Sulfate		10. mg/L

Calibration Standard #3

Analyte	1.25 mL of Cal Std #5	Final Conc.
Fluoride		2.5 mg/L
Chloride		50. mg/L
Nitrite		2.5 mg/L
Bromide		10. mg/L
Nitrate		2.5 mg/L
Ortho-Phosphate		2.5 mg/L
Sulfate		50. mg/L

Calibration Standard #4

Analyte	2.5 mL of Cal Std #5	Final Conc.
Fluoride		5.0 mg/L
Chloride		100 mg/L
Nitrite		5.0 mg/L
Bromide		40. mg/L
Nitrate		5.0 mg/L
Ortho-Phosphate		5.0 mg/L
Sulfate		100 mg/L

7.5.2. Prepare or purchase a secondary stock standard(s) using a standards source other than that used for the primary standards as described in Section 7.5. Dilute these stock standards to as indicated in the table below to prepare the mixture to be used for the LCS and CCV solution. Alternatively prepare this solution by mixing 0.50 mL commercial stock A, 0.50 mL commercial stock B and 4.0 mL of reagent water.

LCS & Continuing Calibration Verification Solution

Analyte	Final Conc. (V_f=5ml)
Fluoride	2.5 mg/L
Chloride	50. mg/L
Nitrite	2.5 mg/L
Bromide	10. mg/L
Nitrate	2.5 mg/L
Ortho Phosphate	2.5 mg/L
Sulfate	50. mg/L

7.5.3. Prepare or purchase a secondary stock standard(s) using a standards source other than that used for the primary standards as described in Section 7.5. Dilute these stock standards to prepare the mixture to be used for the Matrix Spike solution. Alternatively purchase these mixes (ready to use) from a commercial source. Add 100 uL of each IC Spike solution to 5 mL of sample when preparing the MS/MSD. Dilute as needed after spiking the sample.

Matrix Spike "True" Values

Analyte	Final Conc.
Fluoride	2.5 mg/L
Chloride	50. mg/L
Nitrite	2.5 mg/L
Bromide	10. mg/L
Nitrate	2.5 mg/L
Ortho Phosphate	2.5 mg/L
Sulfate	50. mg/L

NOTE: Stock standards, calibration standard #5 and LCS standard should be stored in the dark at 4° ± 2°C. Replace these standards when instrument response indicates target analyte degradation may have occurred or after the standard has expired (12 months

commercial mix or 6 months in house mix), which ever occurs first. Nitrite and ortho-phosphate are particularly light and oxygen sensitive.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to ensure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

8.2. Sample preservation and holding times for the anions that can be determined by this method for water samples are as follows:

QuanTIMs Method Code		Analyte	Preservation	Holding Time
EPA 300.0A	SW846 9056A	Fluoride	4° ± 2°C	28 days
C8	3C			
CX	3D	Chloride	4° ± 2°C	28 days
GO	3E	Nitrite	4° ± 2°C	48 hours
GM	3F	Bromide	4° ± 2°C	28 days
C9	3G	Nitrate	4° ± 2°C	48 hours
DO	3H	Ortho Phosphate	4° ± 2°C	48 hours
CY	3I	Sulfate	4° ± 2°C	28 days

Note: Soil leachates will follow the same preservation and holding times as the water samples; starting from the time of extraction.

9. QUALITY CONTROL

9.1. The STL QC Program document provides further details of the QC and corrective action guidelines presented in this SOP. Refer to this document if additional guidance is required.

9.2. Table I provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.3. Initial Demonstration of Capability

9.3.1. Prior to the analysis of any samples by Ion Chromatography, the following requirements must be meet:

9.3.1.1. Method Detection Limit (MDL): An MDL must be determined prior to analysis of any samples. The MDL is determined using seven replicates of reagent water spiked with the anions of interest that has been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis. The spike level must be greater than the calculated MDL but less than or equal to 10x the MDL. The result of the MDL determination must be below the STL reporting limit.

9.4. Batch definition: Preparation and QC batch definitions are provided in the STL QC Policy.

9.5. Method Blank (MB): One method blank must be processed with each preparation batch. The method blank consists of reagent grade water that has been taken through the entire preparation and analytical process. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest above the reporting limit.

9.6. Laboratory Control Sample (LCS): One LCS must be processed with each preparation batch and must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. If the result is outside established control limits the system is out of control and corrective action must occur. Until in-house limits are established, a control limit of 90 - 110% recovery must be applied. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable. The LCS consists of reagent grade water containing a known amount of target analytes that has been injected into the ion chromatography system. The LCS is prepared from a separate stock standard, or neat material, of a different manufacturer than the stock, or neat material, used to prepare the calibration standard.

9.7. Matrix Spike/Matrix Spike Duplicate (MS/MSD): One MS/MSD pair must be processed for each QC batch. A matrix spike (MS) is a field sample to which a known concentration of target analyte has been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed

along with the sample and matrix spike. Some client specific DQO's may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Spiking levels will be the same as the LCS values.

- If the MS/MSD recovery or RPD falls outside the acceptance range, the recovery of the analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 90-110% recovery and 20% RPD must be applied to the MS/MSD.

- If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported as NC (i.e. not calculated).

- If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted.

- If the recovery of the LCS is outside the limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

- If a MS/MSD is not possible due to limited sample volume then a LCS duplicate must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike limits.

9.8. Continuing Calibration Verification (CCV/CCB): Continuing calibration is verified by analyzing the calibration standard after every ten (10) samples . The CCV must fall within +/- 10% of the true value for each target analyte. A CCB is analyzed immediately following the CCV to monitor low level accuracy and system cleanliness. The CCB result must be below the reporting limit for that analyte. If either the CCV or CCB fail to meet criteria, the analysis must be terminated, the problem corrected and repreparation and analysis of all samples following the last CCV and CCB which were in control.

10. CALIBRATION AND STANDARDIZATION

10.1. Establish ion chromatographic operating parameters equivalent to those indicated in table 2. Refer to Table 3 for typical standard run retention times. Other than the presence of the analytical column the instrument conditions are the same.

10.2. For each analyte of interest, prepare a **minimum** of 3 calibration standards and a blank by adding accurately measured volumes of one or more stock standards to a volumetric flask and dilution to volume with reagent water. If a sample analyte concentration exceeds the calibration range the sample may be diluted to fall within the range. If this is not possible then three new calibration concentrations must be chosen, two of which must bracket the concentration of the sample analyte of interest. Each attenuation range of the instrument used to analyze a sample must be calibrated individually.

10.3. Using an injection volume of 25 uL of each calibration standard, tabulate peak height or area responses against the concentration. The results are used to prepare a calibration curve for each analyte. During this procedure, retention times must be recorded. All analytes will be calibrated using a quadratic regression forced through the origin. Correlation coefficients (R^2) must be 0.995 or better.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Table 2 summarizes the recommended operating conditions for the ion chromatograph. Included in this table are estimated retention times that can be achieved by this method. Other columns, chromatographic conditions, or detectors may be used if the requirements of Sect. 9.2 are met.

11.4. Check system calibration daily as outlined in Table 1 and, if required, recalibrate as described in Sect 10.

11.5. Load and inject a fixed amount (25 uL) of settled & filtered sample. If the sample is cloudy then it should be filtered prior to loading into the autosampler polyvial. Flush injection loop thoroughly, using each new sample. Use the same size loop for standards and samples. Record the resulting peak size in area or peak height units. An automated constant volume injection system may also be used.

11.6. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of various concentration. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms since retention time is concentration dependent for most analytes..

11.7. If the response for the peak exceeds the working range of the system, dilute the sample with an appropriate amount of reagent water and reanalyze.

11.8. If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, fortify the sample with an appropriate amount of standard and reanalyze.

NOTE: Retention time is affected by concentration. Nitrate and sulfate exhibit the greatest amount of change, although all anions are affected to some degree. See Table 3. In some cases this peak migration may produce poor resolution or identification.

11.9. The following extraction should be used for solid materials: Add an amount of reagent water equal to ten times the weight of dry solid material taken as a sample. This slurry is mixed for one hour using a magnetic stirring device or tumbler. Filter the resulting slurry before injecting using a 0.45 um membrane type filter. This can be the type that attaches directly to the end of the syringe

11.10. Should more complete resolution be needed between peaks the eluent (7.3) can be diluted. This will spread out the run but will also cause the later eluting anions to be retained longer. The analyst must determine to what extent the eluent is diluted. This dilution should not be considered a deviation from the method.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Prepare a calibration curve for each analyte by plotting instrument response against standard concentration. Compute sample concentration by comparing sample response with the standard curve. Multiply answer by appropriate dilution factor.

12.2. Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

12.3. Report results in mg/L for aqueous samples and mg/Kg for 1 hour leachates and mg/L for 18 hour leachates of solid samples.

12.4. Report NO_2^- as N

NO_3^- as N

$HPO_4^=$ as P

13. METHOD PERFORMANCE

13.1. The reporting limits for the following analytes are based on a 25 uL injection volume:

Analyte	Water RL	Soil RL
Fluoride	1.0 mg/L	10 mg/kg
Chloride	1.0 mg/L	10 mg/kg
Nitrite	0.5 mg/L	5 mg/kg
Bromide	0.5 mg/L	5 mg/kg
Nitrate	0.05 mg/L	0.5 mg/kg
O-Phosphate	0.5 mg/L	5 mg/kg
Sulfate	1.0 mg/L	10 mg/kg

13.2. The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. The analyst must be given two blind performance samples to analyze or process for analysis. Upon successful completion of the performance evaluation (PE) samples, these analyses will be documented as initial qualification,. Requalification must be performed annually thereafter for this procedure. The group/team leader must document the training and PE performance and submit the results to the QA Manager for inclusion in the associate's training files.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in this procedure must be segregated, and disposed of according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. Method 300.0, "Determination of Inorganic Anions by Ion Chromatography", Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio, Revision 2.1, August 1993.

16.2. Method 9056A, "Determination of Inorganic Anions by Ion Chromatography", SW846, Test Methods for Evaluating Solid Waste, Third Edition, Draft Revision 1, September 1999.

16.3. STL North Canton Laboratory Quality Manual (LQM), current version.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Attachment #1, method Flow Chart

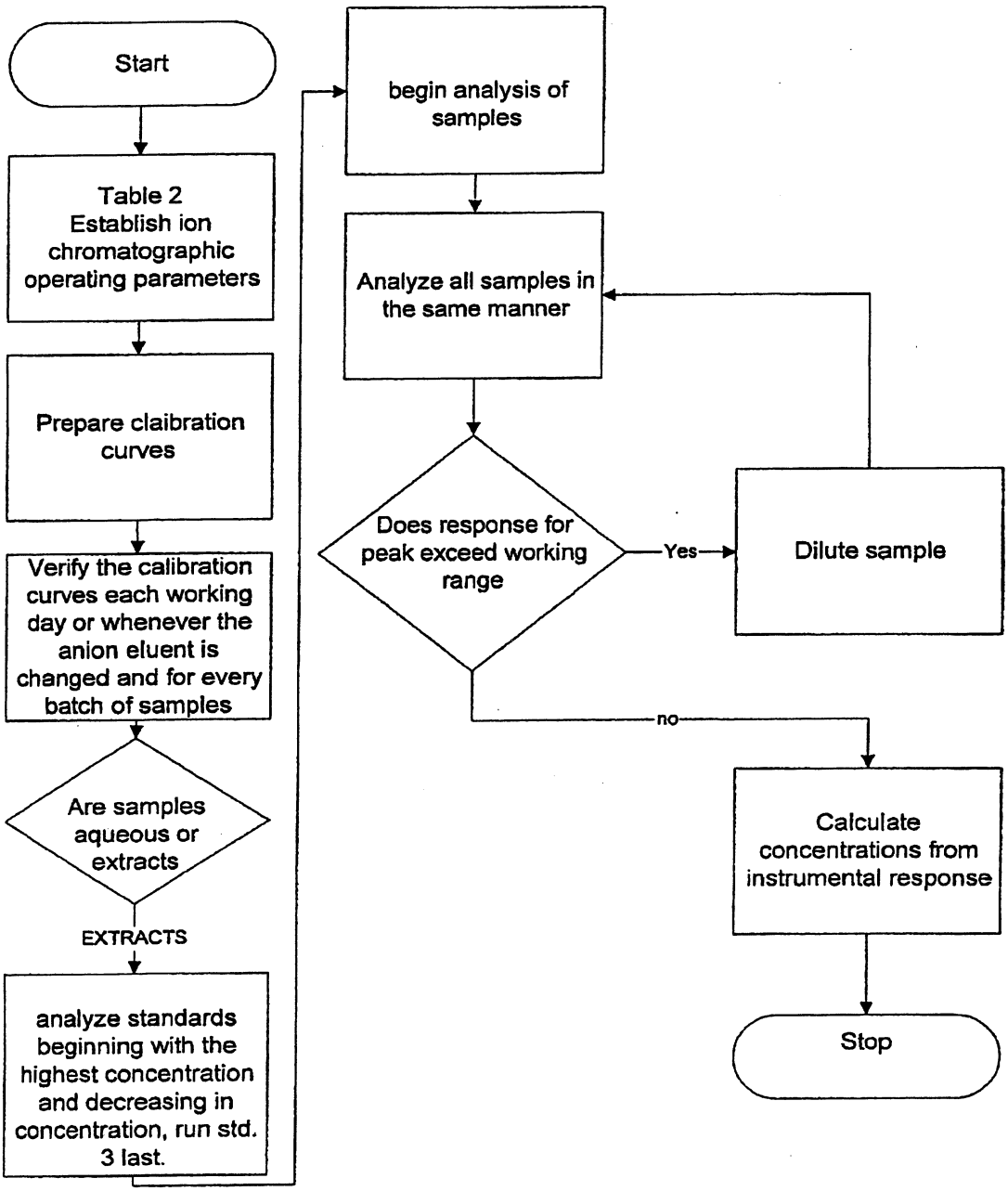
17.2. Table 1, Quality Control Samples

17.3. Table 2, Standard Instrument Operating Parameters

17.4. Table 3, Retention Time Matrix

17.5. Figure 1, Example Chromatogram

Determination Of Inorganic Anions By Ion Chromatography



ATTACHMENT #1

TABLE 1

QUALITY CONTROL SAMPLES

QC Samples	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration Verification (ICV)	At the start of each day following calibrating prior to sample analysis	+/- 10% of true value	Recalibrate and reanalyze
Initial Calibration Blank (ICB)	After Initial Calibration Verification and prior to sample analysis	< the Reporting Limit	Reprepare and reanalyze
Laboratory Control Sample (LCS)	1 per batch of 20 samples	Meets laboratory historical limits	Reanalyze all samples associated with unacceptable LCS
Matrix Spike Sample (MS/MSD)	1 MS/MSD pair per batch or 20 samples	Meets laboratory historical limits	Supervisor's technical judgment
Continuing Calibration Verification (CCV)	Between each group of 10 injections and at the end of the analytical sequence	+/- 10% of true value	Recalibrate and reanalyze all samples since the last acceptable CCV
Continuing Calibration Blank (CCB)	Between each group of 10 injections and at the end of the analytical sequence	< the Reporting Limit	Recalibrate and reanalyze all samples since the last acceptable CCB

TABLE 2

Standard Instrument Operating Parameters

Standard Conditions:

Eluent Pump Rate:	1.20 mL/min (DX-120 and DX-320)
Sample Loop:	25 uL
Eluent:	1.0mM sodium bicarbonate, 3.5mM sodium carbonate
Detector output	Baseline conductivity should be 15 - 20 uS prior to sample analysis.

TABLE 3

Standard Run Retention Time Matrix (minutes)*

Analyte	Concentration (mg/L) 0.05	0.2	0.5	1	2	2.5	5	10	20	40	50	100	200	RT window
F^-	2.75		2.75			2.75	2.75	2.75						
Cl^-				3.97				3.98			4.03	4.08	4.17	
NO_2^-	4.80		4.80			4.78	4.78	4.80						
Br^-		6.15			6.13			6.10	6.08	6.07				
NO_3^-	7.33		7.27			7.17	7.13	7.07						
o-PO_4^{2-}	9.53		9.53			9.52	9.50	9.48						
SO_4^{2-}				11.50				11.48			11.43	11.38	11.27	

* Analyte retention time is concentration dependent for most anions. Retention time increases with increasing concentration for chloride. Retention time decreases with increasing concentration for bromide, nitrate, ortho-phosphate and sulfate.

EXAMPLE ION CHROMATOGRAM

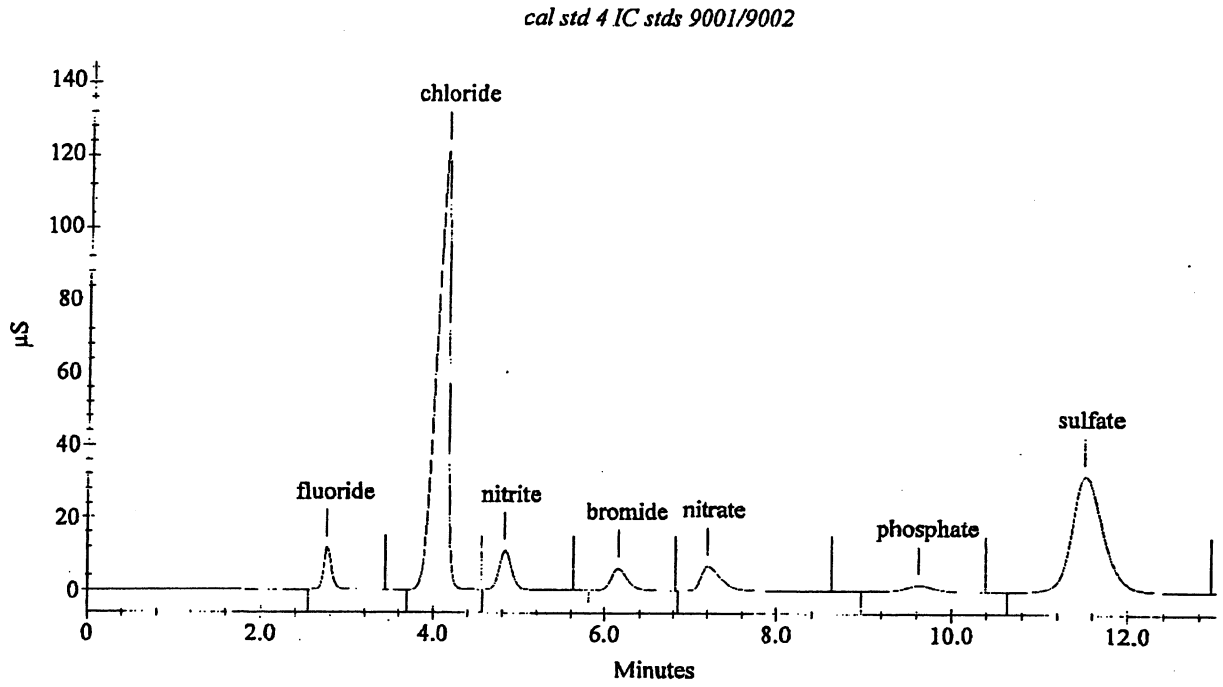


Figure #1

STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: CYANIDE AUTOMATED, PYRIDINE-BARBITURIC ACID METHOD

(SUPERSEDES: REVISION 6 DATED 06/28/99)

Prepared by: _____ 5/31/01
Date

Reviewed by: _____ 5/31/01
Technology Specialist Date

Approved by: _____ 5/31/01
Quality Assurance Manager Date

Approved by: _____ 5-31-01
Environmental Health and Safety Coordinator Date

Approved by: _____ 5-31-01
Laboratory Director Date

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Total, Amenable, and Free Inorganic Cyanide in solids, liquids, and waters. It is based on CLP ILM03.0, CLP ILM04.0, SW846 Method 9012A, 335.2, 335.4 and Standard Method 4500-CN-E. The working linear range is 0.005 - 0.1 mg/L for waters and 0.25 to 5 mg/kg for solids.

1.2. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

1.3. The associated method codes are CLP ILM03.0 (DV), CLP ILM04.0 (OU), SW846 Method 9012A (QP), ASTM D4282-83, EPA 335.1 and 335.2 (CG), EPA 335.4 (O2), and Standard Method 4500-CN-E (A4).

2. SUMMARY OF METHOD

2.1. The Cyanide, as HCN, is released by distilling/refluxing the sample with strong acid and is trapped in a sodium hydroxide solution.

2.2. The sodium hydroxide solution is analyzed colormetrically on an autoanalyzer using the pyridine-barbituric acid method.

3. DEFINITIONS

3.1. Refer to the glossary in the Laboratory Quality Manual (LQM), latest version.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Sulfides interfere, but can be eliminated by treating the sample with cadmium carbonate prior to analysis.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all STL associates.

5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. The following materials are known to be **corrosive: Sodium Hydroxide**

5.3.2. Cyanide is a highly toxic compound that must be handled with care. The analyst must be aware of the handling techniques.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. It is recommended that neat standards be purchased only as a last resort. The preparation of standards from neat materials and reagents {as well as glassware cleaning procedures that involved solvents such as methylene chloride} should be conducted in a fume hood with the sash closed as far as the operations will permit.

5.6. Standards in solution may be diluted in the open laboratory when syringes and the like are utilized.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Traacs 800 autoanalyzer

6.2. Probe 0.016"

6.3. Flowcell 10nm

6.4. Wavelength cell 570nm

6.5. Manifold-Multitest cartridge with cyanide tubing

6.6. 125 mL Erlenmeyer flasks

6.7. 100 mL, 250 mL, 1000 mL volumetric flasks

6.8. Volumetric pipettes: various

6.9. Top loading balance: capable of accurately weighing ± 0.01g.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Cadmium carbonate: powder

7.1.2. 1.25 N sodium hydroxide: Add 50 g of sodium hydroxide pellets (NaOH) to a 1 liter volumetric flask and dilute to volume with reagent water.

7.1.3. Phosphate buffer: Add 136 g of potassium phosphate - monobasic (KH_2PO_4) and 2.8 g of sodium phosphate – dibasic anhydrous (Na_2HPO_4) to 800 mL of reagent water in a 1 liter volumetric flask. Mix, bring to volume with reagent water. Add 3-5 drops of Brij-35 to 100 mL of Buffer prior to using.

7.1.4. Chloramine-T reagent: Add 1.0 g of chloramine-T to a 250 mL volumetric flask and dilute to volume with reagent water. Prepare fresh daily.

7.1.5. Pyridine reagent: Add 15.0 g of barbituric acid to a 1 liter volumetric flask. Add 75 mL of pyridine and 15 mL of concentrated hydrochloric acid (HCl) and mix. Bring to volume with reagent water and store at 4°C ± 2°C in an amber glass bottle.

7.1.6. 0.25 N sodium hydroxide: Add 200 mL of N NaOH to a 1 liter volumetric flask and dilute to volume with reagent water.

7.1.7. Brij-35

7.1.8. 30% Brij solution: Add 2 mL Brij-35 to 1000 mL reagent water.

7.1.9. Rhodanine indicator, purchased.

7.1.10. 0.0192 N silver nitrate, purchased

7.2. Standards

7.2.1. Primary Source Cyanide Stock Standard, 1000 mg/L: Add 2.51 g of potassium cyanide (KCN) and 2.0 g of potassium hydroxide (KOH) to a 1000 mL volumetric flask and dilute to volume with reagent water. Mix well and store in glass amber container. Stable for 1-3 months.

Note: This stock standard must be standardized prior to use. See Appendix I.

7.2.2. Secondary Source Cyanide Standard, 1000 mg/L: Follow 7.2.1 using an alternate source of Potassium Cyanide (KCN).

Note: This stock standard must be standardized prior to use. See Appendix I.

7.2.3. Calibration Standards (Water and Solid Matrices)

7.2.3.1. Pipet the appropriate amount of cyanide standard into 100 mL volumetric. Add 20 mL 1.25N NaOH to each calibration standard (except the 1.0 mg/L) and bring to volume with reagent water. Prepare weekly.

Concentration CN-	mL CN-	Final Volume
100 mg/L	10 mL of 1000 mg/L	100 mL
10 mg/L	10 mL of 100 mg/L	100 mL
1.0 mg/L	10 mL of 10 mg/L	100 mL
*0.1 mg/L	10 mL of 1 mg/L	100 mL
*0.05 mg/L	5 mL of 1 mg/L	100 mL
*0.025 mg/L	25 mL of 0.1 mg/L	100 mL
*0.01 mg/L	10 mL of 0.1 mg/L	100 mL

*0.005 mg/L	10 mL of 0.05 mg/L	100 mL

*Denotes calibration standards

NOTE: The 1 ppm CN standard is only good for the day it is prepared and is not preserved with NaOH.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Solid and liquid samples are not chemically preserved. Water samples are preserved with NaOH to a pH > 12. All samples are stored at 4°C ± 2°C in plastic or glass containers.

8.2. The holding time is fourteen days from sampling to analysis.

*NOTE: Holding time for CLP is twelve days from receipt to analysis.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.2. CLP

9.2.2.1. Each SDG (sample delivery group) must have one method blank (MB) processed per matrix and per twenty samples. If the SDG is distilled over multiple "racks", the method blanks must be distilled with the samples. If more

than one SDG is on one rack, process one method blank per SDG and per matrix. The solid method blank is called "PBS" (prep blank solid) and the water method blank is called "PBW" (prep blank water).

NOTE: A "rack" is one setup of cyanide samples and QC, and can encompass 1-3 of the midi-distillation units.

9.2.3. A reagent water blank consisting of 50 mL reagent water must be distilled and analyzed with each analytical batch of samples. See SOP NC-WC-0032 for distillation instructions.

9.2.4. Corrective Action for Blanks

9.2.4.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.4.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One LCS from an independent source must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. A midrange LCS consisting of a 0.04 mg/L (2.0 mL of 1.0 mg/L to 50 mL) must be distilled and analyzed with each analytical batch of samples. See SOP NC-WC-0032 for distillation instruction.

9.3.3. The acceptance limits for drinking water samples is 90 – 110%. This is also the same value for CLP.

9.3.4. CLP

9.3.4.1. A LCS (which doubles as an ICV) is processed with each "rack". Even if multiple SDG's are on the "rack", only one LCS (ICV) needs to be distilled.

9.3.4.2. For solids process a solid LCS per SDG. The value for this LCS changes as the solid lot number changes each time a new one is ordered. The value is noted on the product certification. If more than one SDG is processed on a "rack", process a solid LCS for each one. If a SDG has solid sample distilled on a different "rack", then additional solid LCS's will need to be distilled.

9.3.5. Corrective Action for LCS

9.3.5.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.5.2. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1. One MS/MSD pair must be processed for each batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis.

9.4.2. A MS/MSD consisting of 50 mL or 1.0 g sample and 0.04 mg/L spike (2.0 mL of 1.0 mg/L to 50 mL) will be distilled and analyzed with every batch. See SOP NC-WC-0032 for distillation instructions. This is also the same value for CLP.

9.4.3. The acceptance limits for drinking water samples is 90 – 110%.

9.4.4. Corrective action for MS/MSDs

9.4.4.1. If the analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch.

9.4.4.2. If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data is reported as DIL (diluted out).

9.4.4.3. If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.4.5. CLP

9.4.5.1. Each SDG must have a sample duplicate and a matrix spike processed per matrix. Sometimes this requirement is waived by the client or PM but, if uncertain, make sure that these are done. Usually , the sample to be spiked is assigned, but not always. If a MS/MSD is created in receiving, either batch the MSD and "N/A" the result or have the MSD deleted. In either case, create a sample duplicate for the sample that is spiked when batching. If processin a SDG over more than one "rack", then there is no need to process additional matrix spikes and sample duplicates from "rack" (unless specifically requested). Typically, only one spiked/duped sample is required per SDG, even if the SDG is distilled over different days.

9.4.5.2. Matrix spike for CLP consists of 50 mL or 1.0 g of sample and 100 µg/L spike (5.0 mL of 1.0 mg/L standard).

9.5. QC Acceptance Criteria

9.5.1. Control limits are established by the laboratory as described in NC-QA-0018.

9.5.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are listed in the Laboratory Quality Manual (LQM) and the latest is version easily accessible via the LIMs (QC Browser program.).

9.6. Method Detection Limits (MDLs) and MDL Checks

9.6.1. MDLs and MDL Checks are established by the laboratory as described in SOP, NC-QA-0021.

9.6.2. MDLs are listed in the Laboratory Quality Manual (LQM) and the latest version is easily accessible via the LIMs (QC Browser program).

9.7. Nonconformance and Corrective Action

9.7.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. The instrument is calibrated using five cyanide standards and a blank at the time of analysis.

10.2. Initial Calibration

10.2.1. The instrument is calibrated at the beginning of each run and is verified at the beginning of the run by using a midrange ICV. The correlation coefficient of the original curve must be $\geq$ 0.995 or recalibration is required.

10.3. Continuing Calibration

10.3.1. The run is checked every ten samples and at the end of the run using a midrange CCV to verify continued linearity. It cannot vary from the original curve by more than $\pm$ 15% or recalibration is required. The previous 10 samples must be reanalyzed and bracketed by a CCV that passes criteria. The CCV is composed of the 0.025 ppm secondary standard.

10.3.2. System cleanliness is checked every ten samples and at the end of the run using a CCB. It cannot contain the analyte of interest above the reporting limit or recalibration is required. The previous 10 samples must be reanalyzed and bracketed by a CCB that passes criteria. The CCB is 0.25N NaOH.

NOTE: Base and gain values may change with new reagents, standards, tubing changes, or board cleaning. These values are for reference only.

10.4. High and Low Standard

10.4.1. The distillation technique is checked by distilling a high and low standard and comparing the values obtained to the standard curve. If the percent recovery is not within ± 10%, the analyst will submit a Nonconformance Memo for the analytical run.

10.4.2. If the percent recovery is outside ±15%, the standard and samples will be redistilled and analyzed.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. See cyanide distillation SOP: NC-WC-0032.

11.3.1.1. The sample is distilled/refluxed under acidic conditions for one hour. The released HCN is trapped in 50 mL of 0.25 N NaOH solution. EPA Method 335.4 says to reflux for one and a half hours.

NOTE: CLP samples are distilled for one and a half hours.

11.3.2. Sample Preparation Procedure

11.3.2.1. All Samples: Test each sample for the presence of sulfides using lead acetate paper. If sulfides are present, treat the sample with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution. Avoid a large excess of

cadmium carbonate and long contact time in order to minimize loss by complexation or occlusion of cyanide on the precipitated material

11.4. Sample Analysis

11.4.1. Summary

11.4.1.1. The sample distillates are analyzed on the autoanalyzer for cyanide using the automated pyridine-barbituric method.

11.4.2. Recommended Instrument Conditions

11.4.2.1. See Manufacturer's information for operation instructions.

11.4.2.2. Start Up Solutions (and Wash Solutions)

11.4.2.2.1. All lines in 0.25N NaOH solution:

11.4.2.2.2. Then all lines in 30% Brij solution.

11.4.2.3. Running Solutions

11.4.2.3.1. Two DI water lines (orange/green) in 30% Brij solution

11.4.2.3.2. Red/Red in pyridine reagent

11.4.2.3.3. Orange/red in chloramine-T reagent

11.4.2.3.4. White/white in phosphate buffer solution

11.4.2.4. Base and Gain

11.4.2.4.1. Performed on the 0.1 mg/L standard

11.4.2.4.2. Approximate values: Base = 75 Gain = 150

*These values are for reference only and may change with new reagent or standard preparations.

11.4.2.5. Trouble shooting for poor Base and Gain or baseline noise

11.4.2.5.1.Place reagent lines 1-3 back in Brig solution and run on high for five minutes. Place all 5 lines (1-5) and probe line in 1.25 N NaOH solution and run on high for five minutes. Place lines back in appropriate "start-up" solutions and run on high for five minutes.

Place lines back in reagents and perform another base and gain when the baseline has stabilized.

11.4.3. Sample Analysis Procedure

11.4.3.1.See Manufacturer's information for operating instructions.

11.4.3.2.A calibration curve is analyzed at the beginning of each run. The correlation coefficient must be > 0.995 to continue.

11.4.3.3.CCV's and CCB's are analyzed at the beginning, end, and between every 10 samples. CCV's must be prepared from a secondary source.

11.4.3.4.Sample distillates higher than the highest calibration standard (0.1 mg/L) must be diluted with 0.25 N NaOH and re-analyzed.

11.4.3.5.Any samples analyzed after a high sample must be re-analyzed if carryover is suspected.

11.4.3.6.CLP

11.4.3.6.1. If more than one SDG is distilled on one rack, they may be analyzed together. Each SDG should have its own method blank(s), its own LCS-S (if samples are solids), and its own sample duplicate and matrix spike. If the SDG has been split over multiple distillation "racks", then you may not have a duped/spiked sample for each batch. You should have method blanks and, if solids, LCS's for each batch. An ICV (LCS) is distilled with each "rack". If more than one SDG is distilled on a "rack", then the ICV (LCS) distilled with that rack must be analyzed with the samples. If the samples from a multiple SDG "rack" are analyzed together, then the ICV needs to be analyzed only once; if they are analyzed on different runs, then the ICV (LCS) needs to be analyzed with each run.

11.4.3.7.Tray Protocol (Run Order)

Normal Tray Protocol

1. Calibration
2. ICV (CCV)
3. ICB (CCB)
4. MD
5. LCS
6. UP to 8 more samples
7. CCV
8. CCB
9. Up to 10 samples
10. CCV
11. CCb

CLP Tray Protocol

12. Calibration
13. Distilled ICV (LCS)
14. ICB
15. CCV
16. CCB
17. PBW

18. PBS (If solids are in SDG)

19. LCS-S (If solids are in SDG)

20. Sample

21. Sample duplicate

22. Sample MS

23. Sample MS @ 2x dilution

24. Sample A-spiked*

25. Up to 2 more samples

26. CCV

27. CCb

28. Up to ten more samples

29. CCV

30. CCb

*A-spike = 2 mL of sample + 2mL of A-spike solution

A-spike solution = 0.8 mL .25N NaOH, +3.2 mL 0.05 ppm 1° standard

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.5.2. All standards are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4. Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. $$\textit{Total Cyanide, mg / L} = \frac{\textit{mg / L } CN^- \textit{ from printout x 50}}{\textit{mL of sample distilled}} \textit{ x D}$$

12.2. $$\textit{Total Cyanide, mg / kg} = \frac{\textit{mg / L } CN^- \textit{ from printout x 50}}{\textit{g of sample distilled.}} \textit{ x D}$$

12.3. $$\textit{Amenable Cyanide, mg / L} = \textit{Total } CN^- \textit{ (mg / L) - Chlorinated } CN^- \textit{ (mg / L)}$$

Where:

mg/L = *can also be mg/kg*

D = *Dilution Factor* = $\frac{\textit{Final Volume of Dilution}}{\textit{Volume of Sample Distillate Used}}$

NOTE: Free cyanide has the same calculations as Total cyanide

12.4. LCS Recovery:

$$\frac{\textit{Instrument Value}}{\textit{0.04 (true)}} \textit{ x 100} = \textit{\% Recovery}$$

12.5. CCV Recovery:

$$\frac{\textit{Instrument Value}}{\textit{0.025 (true)}} \textit{ x 100 = \% Recovery}$$

NOTE: CCV recovery must be between 85 - 115% for data to be acceptable. If CCV recovery is not within these limits, reanalysis is required 335.4 says 10%.

12.6. ***MS/MSD Recovery for Waters and solids:***

$$\frac{\textit{A - B}}{\textit{0.040 (true)}} \textit{ x 100 = \% Recovery}$$

Where:

A = ***Instrument value MS/MSD***

B = ***Sample instrument value***

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

13.2.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. All distillates can be rinsed down the drain with large amounts of water unless they contain high levels of Cyanide. If they are highly concentrated, they must be disposed of in the liquid waste container.

15.2. Solvent waste must be disposed of in clearly labeled waste cans.

15.3. Acid waste must be collected in clearly labeled acid waste containers.

15.4. Solid materials (gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.5. Refer to the Laboratory Sample and Waste Disposal plan.

15.6. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of STL. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1. References

16.1.1. SW846, Test Methods for Evaluating Solid Waste Method 9012 A, and its updates

16.1.2. EPA 600; Cyanide, Total and Cyanide, Amenable to Chlorination; Methods 335.2, March 1983

16.1.3. Standard Methods for the Examination of Water and Wastewater, Eighteenth Edition: Weak and Dissociable Cyanide; Method 4500-CN-E

16.1.4. USEPA CLP SOW ILM03.0 and ILM04.0, Section D - Cyanide Midi Distillation

16.1.5. Corporate Quality Management Plan (QMP), current version.

16.1.6. STL Laboratory Quality Manual (LQM), current version

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-0014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.4. Method Detection Limits and Instrument Detection Limits, NC-QA-0021

16.2.5. Navy/Army SOP, NC-QA-0016

16.2.6. NC-WC-0032, Cyanide Preparation Method

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Reporting limits

17.1.1. The reporting limit (RL) is 0.01 mg/L for waters (50 mL used) and 0.50 mg/kg for solids (1.0 g used). The reporting limit for solid samples performed only under the Michigan program is 200 µg/kg for solids (2.5 g used). The lowest level of the calibration curve can be used as the reporting limit upon request.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Troubleshooting guide for poor Base and Gain or baseline noise

17.2.1. Place reagent lines 1-3 back in Brig solution and run on high for five minutes. Place all 5 lines (1-5) and probe line in 1.25 N NaOH solution and run on high for five minutes. Place lines back in appropriate "start-up" solutions and run on high for five minutes.

Place lines back in reagents and perform another base and gain when the baseline has been stabilized.

17.3. Method Deviation (9012A335.2)

17.3.1. Method of Standard Addition is not performed for samples with matrix interference (sulfides).

Appendix I - Cyanide Standardization

1. Pipet 10.0 mL of the 1000 ppm stock cyanide standard into a 250 mL Erlenmeyer flask and add 90 mL of reagent water.
2. Add 0.5 mL (10 drops) of Rhodanine indicator.
3. Titrate with 0.0192 N silver nitrate (using a micro burette) until the color changes from yellow to pink/orange.

4. Titrate a blank (100 mL reagent water) following steps 2 and 3.

5. Calculation:

$$\textbf{Cyanide, mg / L} = \frac{\textbf{(A - B) (1000)}}{\textbf{mL Cyanide Solution (10)}}$$

Where:

A = mL titrant for standard

B = mL titrant for blank

6. If the cyanide concentration is not 1000 ppm, adjust concentration accordingly.

ATTACHMENT 3

LABORATORY REFERENCE DATA

STL Reference Data Summary

Structured Analysis Code: I-25-QK-01-01

Target Analyte List: NC: OLM03.1/4.2 Volatile List

Matrix: WATER
Extraction: PURGE AND TRAP - 25 mL purge (Waters)
Method: Volatile Organics, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 4121			Detection Limits				Check List 1630							Spike List 1631						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
11	Acetone	10	ug/L	0.51	ug/L	20010221		Y	20	ug/L	22	200	95		Y	20	ug/L	45	128	30
196	Benzene	1	ug/L	0.16	ug/L	20010215	C	Y	20	ug/L	80	116	20	C	Y	20	ug/L	78	118	20
323	Bromodichloromethane	1	ug/L	0.17	ug/L	20010215		Y	20	ug/L	87	130	30		Y	20	ug/L	80	146	30
340	Bromoform	1	ug/L	0.18	ug/L	20010215		Y	20	ug/L	76	150	30		Y	20	ug/L	58	176	30
343	Bromomethane	1	ug/L	0.2	ug/L	20010223		Y	20	ug/L	64	129	30		Y	20	ug/L	55	145	30
372	2-Butanone	10	ug/L	0.41	ug/L	20010215		Y	20	ug/L	28	237	65		Y	20	ug/L	71	123	30
459	Carbon disulfide	1	ug/L	0.2	ug/L	20010215		Y	20	ug/L	73	139	30		Y	20	ug/L	69	138	41
463	Carbon tetrachloride	1	ug/L	0.12	ug/L	20010221		Y	20	ug/L	75	149	30		Y	20	ug/L	63	176	30
521	Chlorobenzene	1	ug/L	0.14	ug/L	20010221	C	Y	20	ug/L	76	117	20	C	Y	20	ug/L	76	117	20
535	Dibromochloromethane	1	ug/L	0.26	ug/L	20010215		Y	20	ug/L	81	138	30		Y	20	ug/L	71	158	30
550	Chloroethane	1	ug/L	0.26	ug/L	20010215		Y	20	ug/L	66	126	30		Y	20	ug/L	59	142	30
569	Chloroform	1	ug/L	0.14	ug/L	20010221		Y	20	ug/L	84	128	30		Y	20	ug/L	83	141	30
574	Chloromethane	1	ug/L	0.13	ug/L	20010215		Y	20	ug/L	48	123	30		Y	20	ug/L	40	137	39
669	Cyclohexane	1	ug/L	0.13	ug/L	20010221														
539	1,2-Dibromo-3-chloropropane	2	ug/L	0.62	ug/L	20010112														
870	1,2-Dibromoethane	1	ug/L	0.22	ug/L	20010215														
904	1,2-Dichlorobenzene	1	ug/L	0.11	ug/L	20010221														
907	1,3-Dichlorobenzene	1	ug/L	0.15	ug/L	20010215														
910	1,4-Dichlorobenzene	1	ug/L	0.17	ug/L	20010215														
924	Dichlorodifluoromethane	1	ug/L	0.26	ug/L	20010215														
933	1,1-Dichloroethane	1	ug/L	0.16	ug/L	20010215		Y	20	ug/L	86	123	30		Y	20	ug/L	88	127	30
936	1,2-Dichloroethane	1	ug/L	0.21	ug/L	20010215		Y	20	ug/L	79	136	30		Y	20	ug/L	71	160	30
948	cis-1,2-Dichloroethene	0.5	ug/L	0.22	ug/L	20010215		Y	20	ug/L	85	113	30		Y	20	ug/L	87	114	30
950	trans-1,2-Dichloroethene	0.5	ug/L	0.15	ug/L	20010215		Y	20	ug/L	79	120	30		Y	20	ug/L	85	116	30
943	1,1-Dichloroethene	1	ug/L	0.24	ug/L	20010215	C	Y	20	ug/L	63	130	20	C	Y	20	ug/L	62	130	20
986	1,2-Dichloropropane	1	ug/L	0.17	ug/L	20010215		Y	20	ug/L	82	115	30		Y	20	ug/L	87	114	30
998	cis-1,3-Dichloropropene	1	ug/L	0.12	ug/L	20010215		Y	20	ug/L	84	130	30		Y	20	ug/L	82	130	30
1000	trans-1,3-Dichloropropene	1	ug/L	0.27	ug/L	20010215		Y	20	ug/L	84	130	30		Y	20	ug/L	73	147	30
1332	Ethylbenzene	1	ug/L	0.12	ug/L	20010221		Y	20	ug/L	86	116	30		Y	20	ug/L	86	132	30
1515	2-Hexanone	10	ug/L	0.46	ug/L	20010215		Y	20	ug/L	35	200	52		Y	20	ug/L	81	128	30
1578	Isopropylbenzene	1	ug/L	0.12	ug/L	20010215														
1774	Methyl acetate	10	ug/L	0.41	ug/L	20010215														
1799	Methylcyclohexane	1	ug/L	0.15	ug/L	20010221														
1811	Methylene chloride	1	ug/L	0.34	ug/L	20010215		Y	20	ug/L	78	118	30		Y	20	ug/L	82	115	30
1845	4-Methyl-2-pentanone	10	ug/L	0.23	ug/L	20010215		Y	20	ug/L	78	141	32		Y	20	ug/L	82	135	30
2772	Methyl tert-butyl ether	5	ug/L	0.13	ug/L	20010215														

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Structured Analysis Code: I-25-QK-01-01

Target Analyte List: NC: OLM03.1/4.2 Volatile List

Matrix: WATER
Extraction: PURGE AND TRAP - 25 mL purge (Waters)
Method: Volatile Organics, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 4121			Detection Limits			Check List 1630							Spike List 1631						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2355	Styrene	1	ug/L	0.15	ug/L	20010215		Y	20	ug/L	85	117	30		Y	20	ug/L	83	120	30
2439	1,1,2,2-Tetrachloroethane	1	ug/L	0.25	ug/L	20010215		Y	20	ug/L	85	118	30		Y	20	ug/L	88	116	30
2445	Tetrachloroethene	1	ug/L	0.23	ug/L	20010215		Y	20	ug/L	88	113	30		Y	20	ug/L	85	121	30
2489	Toluene	1	ug/L	0.18	ug/L	20010215	C	Y	20	ug/L	74	119	20	C	Y	20	ug/L	70	119	20
2515	1,2,4-Trichlorobenzene	1	ug/L	0.17	ug/L	20010215														
2518	1,1,1-Trichloroethane	1	ug/L	0.15	ug/L	20010221		Y	20	ug/L	78	140	30		Y	20	ug/L	71	162	30
2522	1,1,2-Trichloroethane	1	ug/L	0.3	ug/L	20010215		Y	20	ug/L	83	122	30		Y	20	ug/L	86	129	30
2525	Trichloroethene	1	ug/L	0.14	ug/L	20010215	C	Y	20	ug/L	75	122	20	C	Y	20	ug/L	62	130	20
1428	Trichlorofluoromethane	1	ug/L	0.28	ug/L	20010215														
2566	1,1,2-Trichloro-1,2,2-trifluoroethane	1	ug/L	0.32	ug/L	20010215														
2613	Vinyl chloride	1	ug/L	0.15	ug/L	20010215		Y	20	ug/L	61	120	30		Y	20	ug/L	88	126	30
2627	Xylenes (total)	1	ug/L	0.33	ug/L	20010221		Y	60	ug/L	87	116	30		Y	60	ug/L	89	121	30
337	4-Bromofluorobenzene						X	Y	10	ug/L	74	116	0	X	Y	20	ug/L	74	116	0
2735	1,2-Dichloroethane-d4						X	Y	10	ug/L	61	128	0	X	Y	10	ug/L	61	128	0
2740	Toluene-d8						X	Y	10	ug/L	76	110	0	X	Y	10	ug/L	76	110	0
2863	Dibromofluoromethane						X	Y	10	ug/L	73	122	0	X	Y	10	ug/L	73	122	0

STL Reference Data Summary

Structured Analysis Code: I-51-QL-01-01

Target Analyte List: NC: OLM03.1/4.2 Semivolatile List

Matrix: WATER
Extraction: LIQ/LIQ, CONT (Acids,Phenols) - Acid
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 4122			Detection Limits				Check List 1114							Spike List 1115						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
1	Acenaphthene	10	ug/L	0.8	ug/L	20010424	C	Y	50	ug/L	39	118	35	C	Y	50	ug/L	26	118	35
5	Acenaphthylene	10	ug/L	0.71	ug/L	20010424		Y	50	ug/L	51	110	30		Y	50	ug/L	49	111	51
24	Acetophenone	10	ug/L	2.6	ug/L	20010329														
122	Anthracene	10	ug/L	0.68	ug/L	20010424		Y	50	ug/L	54	116	33		Y	50	ug/L	49	118	51
158	Atrazine	10	ug/L	2.5	ug/L	20010329														
3398	Benzaldehyde	10	ug/L	2.8	ug/L	20010329														
202	Benzo(a)anthracene	10	ug/L	0.7	ug/L	20010424		Y	50	ug/L	49	112	34		Y	50	ug/L	48	115	51
205	Benzo(b)fluoranthene	10	ug/L	0.94	ug/L	20010424		Y	50	ug/L	51	115	38		Y	50	ug/L	44	123	54
208	Benzo(k)fluoranthene	10	ug/L	0.84	ug/L	20010424		Y	50	ug/L	52	116	34		Y	50	ug/L	46	123	53
210	Benzo(ghi)perylene	10	ug/L	0.85	ug/L	20010424		Y	50	ug/L	47	118	35		Y	50	ug/L	44	122	55
211	Benzo(a)pyrene	10	ug/L	0.73	ug/L	20010424		Y	50	ug/L	50	116	34		Y	50	ug/L	44	122	51
3474	1,1'-Biphenyl	10	ug/L	2.6	ug/L	20010329														
289	bis(2-Chloroethoxy)methane	10	ug/L	0.87	ug/L	20010424		Y	50	ug/L	54	112	32		Y	50	ug/L	57	114	49
293	bis(2-Chloroethyl) ether	10	ug/L	0.97	ug/L	20010424		Y	50	ug/L	59	114	30		Y	50	ug/L	57	120	51
302	bis(2-Ethylhexyl) phthalate	10	ug/L	2.7	ug/L	20010222		Y	50	ug/L	49	124	39		Y	50	ug/L	43	128	84
348	4-Bromophenyl phenyl ether	10	ug/L	0.88	ug/L	20010424		Y	50	ug/L	56	113	33		Y	50	ug/L	51	119	51
403	Butyl benzyl phthalate	10	ug/L	0.88	ug/L	20010424		Y	50	ug/L	46	121	40		Y	50	ug/L	41	127	84
5101	Caprolactam	10	ug/L	2.5	ug/L	20010329														
2751	Carbazole	10	ug/L	0.82	ug/L	20010424		Y	50	ug/L	50	118	34		Y	50	ug/L	48	119	53
518	4-Chloroaniline	10	ug/L	0.61	ug/L	20010424		Y	50	ug/L	21	110	41		Y	50	ug/L	10	110	99
578	4-Chloro-3-methylphenol	10	ug/L	0.99	ug/L	20010205	C	Y	50	ug/L	29	124	55	C	Y	50	ug/L	21	124	55
589	2-Chloronaphthalene	10	ug/L	0.85	ug/L	20010424		Y	50	ug/L	50	116	30		Y	50	ug/L	51	119	51
600	2-Chlorophenol	10	ug/L	1	ug/L	20010220	C	Y	50	ug/L	19	124	43	C	Y	50	ug/L	19	124	43
602	4-Chlorophenyl phenyl ether	10	ug/L	0.79	ug/L	20010424		Y	50	ug/L	56	112	31		Y	50	ug/L	51	118	51
633	Chrysene	10	ug/L	0.75	ug/L	20010424		Y	50	ug/L	49	118	34		Y	50	ug/L	49	118	52
860	Dibenz(a,h)anthracene	10	ug/L	0.79	ug/L	20010205		Y	50	ug/L	48	124	36		Y	50	ug/L	45	127	57
863	Dibenzofuran	10	ug/L	1.1	ug/L	20010215		Y	50	ug/L	53	112	30		Y	50	ug/L	51	117	51
891	Di-n-butyl phthalate	10	ug/L	0.67	ug/L	20010424		Y	50	ug/L	50	116	36		Y	50	ug/L	41	121	53
918	3,3'-Dichlorobenzidine	50	ug/L	0.93	ug/L	20010205		Y	50	ug/L	10	110	75		Y	50	ug/L	10	110	99
971	2,4-Dichlorophenol	10	ug/L	0.85	ug/L	20010424		Y	50	ug/L	51	118	43		Y	50	ug/L	52	121	88
1082	Diethyl phthalate	10	ug/L	0.65	ug/L	20010424		Y	50	ug/L	14	135	47		Y	50	ug/L	10	148	81
1145	2,4-Dimethylphenol	10	ug/L	0.85	ug/L	20010220		Y	50	ug/L	10	123	44		Y	50	ug/L	10	125	62
1149	Dimethyl phthalate	10	ug/L	0.76	ug/L	20010424		Y	50	ug/L	10	150	51		Y	50	ug/L	10	159	99
1167	4,6-Dinitro-2-methylphenol	50	ug/L	1.3	ug/L	20010329		Y	50	ug/L	22	143	79		Y	50	ug/L	20	158	99
1187	2,4-Dinitrophenol	50	ug/L	3	ug/L	20010215		Y	50	ug/L	10	146	99		Y	50	ug/L	10	168	99
1191	2,4-Dinitrotoluene	10	ug/L	0.79	ug/L	20010424	C	Y	50	ug/L	47	131	32	C	Y	50	ug/L	31	131	32

Structured Analysis Code: I-51-QL-01-01

Target Analyte List: NC: OLM03.1/4.2 Semivolatile List

Matrix: WATER
Extraction: LIQ/LIQ, CONT (Acids,Phenols) - Acid
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 4122			Detection Limits				Check List 1114							Spike List 1115						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
1193	2,6-Dinitrotoluene	10	ug/L	0.57	ug/L	20010424		Y	50	ug/L	59	125	32		Y	50	ug/L	58	127	82
1162	Di-n-octyl phthalate	10	ug/L	1.1	ug/L	20010424		Y	50	ug/L	49	130	42		Y	50	ug/L	39	144	89
1414	Fluoranthene	10	ug/L	0.81	ug/L	20010424		Y	50	ug/L	54	118	33		Y	50	ug/L	49	122	53
1417	Fluorene	10	ug/L	1	ug/L	20010215		Y	50	ug/L	53	114	31		Y	50	ug/L	51	119	51
1482	Hexachlorobenzene	10	ug/L	0.74	ug/L	20010205		Y	50	ug/L	54	114	34		Y	50	ug/L	48	123	51
1489	Hexachlorobutadiene	10	ug/L	0.73	ug/L	20010220		Y	50	ug/L	36	113	47		Y	50	ug/L	35	118	56
1492	Hexachlorocyclopentadiene	50	ug/L	1.1	ug/L	20010327		Y	50	ug/L	10	114	51		Y	50	ug/L	10	113	97
1497	Hexachloroethane	10	ug/L	0.74	ug/L	20010424		Y	50	ug/L	29	115	49		Y	50	ug/L	31	118	92
1535	Indeno(1,2,3-cd)pyrene	10	ug/L	0.63	ug/L	20010219		Y	50	ug/L	45	120	43		Y	50	ug/L	39	126	59
1566	Isophorone	10	ug/L	0.63	ug/L	20010205		Y	50	ug/L	54	111	33		Y	50	ug/L	56	112	50
1829	2-Methylnaphthalene	10	ug/L	0.64	ug/L	20010424		Y	50	ug/L	52	110	32		Y	50	ug/L	45	119	51
1851	2-Methylphenol	10	ug/L	1.1	ug/L	20010219		Y	50	ug/L	50	110	34		Y	50	ug/L	42	113	73
1857	4-Methylphenol	10	ug/L	1.3	ug/L	20010219		Y	50	ug/L	49	110	35		Y	50	ug/L	29	122	55
1932	Naphthalene	10	ug/L	0.75	ug/L	20010424		Y	50	ug/L	49	112	35		Y	50	ug/L	50	176	52
1960	2-Nitroaniline	50	ug/L	0.7	ug/L	20010205		Y	50	ug/L	53	122	34		Y	50	ug/L	48	125	83
1964	3-Nitroaniline	50	ug/L	0.71	ug/L	20010205		Y	50	ug/L	10	140	39		Y	50	ug/L	10	140	99
1968	4-Nitroaniline	50	ug/L	1.3	ug/L	20010220		Y	50	ug/L	32	116	49		Y	50	ug/L	20	122	99
1972	Nitrobenzene	10	ug/L	0.81	ug/L	20010424		Y	50	ug/L	56	119	32		Y	50	ug/L	56	125	81
1998	2-Nitrophenol	10	ug/L	2.1	ug/L	20010219		Y	50	ug/L	46	129	49		Y	50	ug/L	51	131	77
2001	4-Nitrophenol	50	ug/L	1.9	ug/L	20010329	C	Y	50	ug/L	19	144	34	C	Y	50	ug/L	10	145	34
2028	N-Nitrosodiphenylamine	10	ug/L	1.4	ug/L	20010215		Y	50	ug/L	49	117	36		Y	50	ug/L	49	117	51
2024	N-Nitrosodi-n-propylamine	10	ug/L	0.67	ug/L	20010424	C	Y	50	ug/L	30	115	36	C	Y	50	ug/L	18	115	36
3597	2,2'-oxybis(1-Chloropropane)	10	ug/L	0.78	ug/L	20010424														
2118	Pentachlorophenol	10	ug/L	2.4	ug/L	20010329	C	Y	50	ug/L	10	140	56	C	Y	50	ug/L	10	140	56
2154	Phenanthrene	10	ug/L	0.82	ug/L	20010424		Y	50	ug/L	53	112	32		Y	50	ug/L	52	117	51
2155	Phenol	10	ug/L	2.6	ug/L	20010219	C	Y	50	ug/L	10	131	43	C	Y	50	ug/L	10	131	43
2252	Pyrene	10	ug/L	1.1	ug/L	20010424	C	Y	50	ug/L	46	130	31	C	Y	50	ug/L	27	138	31
2555	2,4,5-Trichlorophenol	10	ug/L	1.2	ug/L	20010219		Y	50	ug/L	46	122	48		Y	50	ug/L	45	125	74
2559	2,4,6-Trichlorophenol	10	ug/L	1.7	ug/L	20010219		Y	50	ug/L	46	118	48		Y	50	ug/L	46	122	98
1425	2-Fluorobiphenyl						X	Y	50	ug/L	30	110	0	X	Y	50	ug/L	30	110	0
1426	2-Fluorophenol						X	Y	75	ug/L	13	110	0	X	Y	75	ug/L	13	110	0
2512	2,4,6-Tribromophenol						X	Y	75	ug/L	21	122	0	X	Y	75	ug/L	21	122	0
2736	Nitrobenzene-d5						X	Y	50	ug/L	32	112	0	X	Y	50	ug/L	32	112	0
2737	Phenol-d5						X	Y	75	ug/L	10	113	0	X	Y	75	ug/L	10	113	0
2738	Terphenyl-d14						X	Y	50	ug/L	10	144	0	X	Y	50	ug/L	10	144	0

STL Reference Data Summary

Structured Analysis Code: I-61-QH-72-01

Target Analyte List: Q: Aroclors Only Standard List

Matrix: WATER
Extraction: LIQ/LIQ, CONT w/ACID STRIP (PCB) - Nominal
Method: PCBs (8082)
QC Program: MICHIGAN
Location: STL North Canton

Target List 2207		Detection Limits				Check List 928							Spike List 929						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2082	Aroclor 1016	0.2	ug/L	0.054	ug/L	20000229	C	Y	10	ug/L	61	118	20	C	Y	10	ug/L	56	119	20
2085	Aroclor 1221	0.2	ug/L	0.11	ug/L	20000229														
2088	Aroclor 1232	0.4	ug/L	0.056	ug/L	20000229														
2091	Aroclor 1242	0.2	ug/L	0.075	ug/L	20000229														
2094	Aroclor 1248	0.2	ug/L	0.061	ug/L	20000229														
2097	Aroclor 1254	0.2	ug/L	0.082	ug/L	20000229														
2100	Aroclor 1260	0.2	ug/L	0.044	ug/L	20000229	C	Y	10	ug/L	61	124	27	C	Y	10	ug/L	31	138	27
2732	Decachlorobiphenyl						X	Y	0.2	ug/L	24	128	0	X	Y	0.2	ug/L	24	128	0
2739	Tetrachloro-m-xylene						X	Y	0.2	ug/L	45	120	0	X	Y	0.2	ug/L	45	120	0

STL Reference Data Summary

Structured Analysis Code: I-04-QO-01-01

Target Analyte List: Q: CLP ICP Metals TAL Standard List

Matrix: WATER
Extraction: METALS, TOTAL RECOVERABLE
Method: Inductively Coupled Plasma (6010B)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 2600		Detection Limits				Check List 906							Spike List 907						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
88	Aluminum	0.2	mg/L	0.028	mg/L	20000224		Y	2000	ug/L	80	120	20		Y	2000	ug/L	75	125	20
128	Antimony	0.06	mg/L	0.0022	mg/L	20000412		Y	500	ug/L	80	120	20		Y	500	ug/L	75	125	20
194	Barium	0.20	mg/L	0.003	mg/L	20000412		Y	2000	ug/L	80	120	20		Y	2000	ug/L	75	125	20
222	Beryllium	0.005	mg/L	0.00054	mg/L	20000224		Y	50	ug/L	80	120	20		Y	50	ug/L	75	125	20
411	Cadmium	0.005	mg/L	0.00028	mg/L	20000412		Y	50	ug/L	80	120	20		Y	50	ug/L	75	125	20
413	Calcium	5.0	mg/L	0.250	mg/L	20000224		Y	50000	ug/L	80	120	20		Y	50000	ug/L	75	125	20
2952	Chromium	0.01	mg/L	0.0014	mg/L	20000412		Y	200	ug/L	80	120	20		Y	200	ug/L	75	125	20
637	Cobalt	0.05	mg/L	0.0013	mg/L	20000224		Y	500	ug/L	80	120	20		Y	500	ug/L	75	125	20
643	Copper	0.025	mg/L	0.0042	mg/L	20000224		Y	250	ug/L	80	120	20		Y	250	ug/L	75	125	20
1539	Iron	0.1	mg/L	0.088	mg/L	20000412		Y	1000	ug/L	77	127	20		Y	1000	ug/L	75	125	20
1618	Magnesium	5.0	mg/L	0.030	mg/L	20000224		Y	50000	ug/L	80	120	20		Y	50000	ug/L	75	125	20
1659	Manganese	0.015	mg/L	0.0009	mg/L	20000412		Y	500	ug/L	80	120	20		Y	500	ug/L	75	125	20
1956	Nickel	0.04	mg/L	0.0022	mg/L	20000412		Y	500	ug/L	80	120	20		Y	500	ug/L	75	125	20
2214	Potassium	5	mg/L	0.041	mg/L	20000224		Y	50000	ug/L	80	120	20		Y	50000	ug/L	75	125	20
2285	Silver	0.01	mg/L	0.0015	mg/L	20000224		Y	50	ug/L	80	120	20		Y	50	ug/L	75	125	20
2315	Sodium	5	mg/L	0.630	mg/L	20000224		Y	50000	ug/L	80	120	20		Y	50000	ug/L	75	125	20
2607	Vanadium	0.05	mg/L	0.00082	mg/L	20000224		Y	500	ug/L	80	120	20		Y	500	ug/L	75	125	20
2649	Zinc	0.02	mg/L	0.012	mg/L	20000224		Y	500	ug/L	80	120	20		Y	500	ug/L	75	125	20

STL Reference Data Summary

Structured Analysis Code: I-04-QM-01-01

Target Analyte List: Q: CLP Trace ICP Metals TAL Standard List

Matrix: WATER
Extraction: METALS, TOTAL RECOVERABLE
Method: Inductively Coupled Plasma (6010B Trace)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 2610		Detection Limits					Check List 906							Spike List 907						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
140	Arsenic	0.01	mg/L	0.0039	mg/L	20010218		Y	2000	ug/L	80	120	20		Y	2000	ug/L	75	125	20
1605	Lead	0.003	mg/L	0.0025	mg/L	20000412		Y	500	ug/L	80	120	20		Y	500	ug/L	75	125	20
2281	Selenium	0.005	mg/L	0.0045	mg/L	20000224		Y	2000	ug/L	80	120	20		Y	2000	ug/L	75	125	20
2477	Thallium	0.010	mg/L	0.005	mg/L	20000224		Y	2000	ug/L	80	120	20		Y	2000	ug/L	75	125	20

STL Reference Data Summary

Structured Analysis Code: I-19-O8-01-01
Target Analyte List: All Analytes

Matrix: WATER
Extraction: METALS, TOTAL (Method exclusive) - Waters
Method: Mercury (7470A, Cold Vapor) - Liquid
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits					Check List 1021							Spike List 1026						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
1701	Mercury	0.0002	mg/L	0.000069	mg/L	20001031		Y	5	ug/L	70	118	20		Y	1	ug/L	53	135	20

STL Reference Data Summary

Structured Analysis Code: I-06-QP-01-01
Target Analyte List: All Analytes

Matrix: WATER
Extraction: DISTILLATION, MICRO/MIDI - Acid
Method: Cyanide, Total (9012A, Automated)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits					Check List 1180							Spike List 1181						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
667	Total Cyanide	0.01	mg/L	0.0017	mg/L	20001206		Y			65	114	20		Y	0.04	mg/L	40	130	99

STL Reference Data Summary

Structured Analysis Code: I-06-N4-01-01
Target Analyte List: All Analytes

Matrix: WATER
Extraction: DISTILLATION, MICRO/MIDI - Acid
Method: Cyanide, Amenable (9012)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List			Detection Limits				Check List 1180							Spike List 1181						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
664	Cyanide Amenable to Chlorination	0.01	mg/L	0.01	mg/L	20010927		Y			65	114	20		Y	0.04	mg/L	40	130	99

STL Reference Data Summary

Structured Analysis Code: I-88-CX-01-01
Target Analyte List: All Analytes

Matrix: WATER
Extraction: NO SAMPLE PREPARATION PERFORMED / DIRECT INJE
Method: Chloride (300.0, Ion Chromatography)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits					Check List 1045							Spike List 1046						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
512	Chloride	1	mg/L	0.17	mg/L	20001201	C	Y			90	110	20	C	Y	50	mg/L	56	137	20

STL Reference Data Summary

Structured Analysis Code: A-I5-QK-01-01

Target Analyte List: NC: OLM03.1/4.2 Volatile List

Matrix: SOLID
Extraction: Indiana Modified Method 5035 (IN 5035-M)
Method: Volatile Organics, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: STL North Canton

	Target List 4121		Detection Limits				Check List 924							Spike List 925						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
11	Acetone	20	ug/kg	2.1	ug/kg	20010222		Y	20	ug/kg	58	130	30		Y	50	ug/kg	10	200	66
196	Benzene	5	ug/kg	0.65	ug/kg	20010122	C	Y	20	ug/kg	75	129	20	C	Y	50	ug/kg	55	138	20
323	Bromodichloromethane	5	ug/kg	1.2	ug/kg	20010308		Y	20	ug/kg	72	125	30		Y	50	ug/kg	47	131	51
340	Bromoform	5	ug/kg	1.6	ug/kg	20010308		Y	20	ug/kg	43	149	30		Y	50	ug/kg	26	141	64
343	Bromomethane	5	ug/kg	1.8	ug/kg	20010308		Y	20	ug/kg	24	152	30		Y	50	ug/kg	15	152	72
372	2-Butanone	20	ug/kg	2.7	ug/kg	20010308		Y	20	ug/kg	27	200	46		Y	50	ug/kg	21	195	60
459	Carbon disulfide	5	ug/kg	0.78	ug/kg	20010222		Y	20	ug/kg	50	137	30		Y	50	ug/kg	27	149	73
463	Carbon tetrachloride	5	ug/kg	1	ug/kg	20010125		Y	20	ug/kg	57	137	30		Y	50	ug/kg	32	143	68
521	Chlorobenzene	5	ug/kg	0.52	ug/kg	20010122	C	Y	20	ug/kg	75	127	22	C	Y	50	ug/kg	49	139	22
535	Dibromochloromethane	5	ug/kg	1.1	ug/kg	20010308		Y	20	ug/kg	49	135	30		Y	50	ug/kg	44	135	61
550	Chloroethane	5	ug/kg	2.2	ug/kg	20010308		Y	20	ug/kg	31	144	30		Y	50	ug/kg	32	140	66
569	Chloroform	5	ug/kg	0.82	ug/kg	20010122		Y	40	ug/kg	73	115	30		Y	50	ug/kg	59	128	46
574	Chloromethane	5	ug/kg	1.3	ug/kg	20010122		Y	20	ug/kg	15	136	30		Y	50	ug/kg	28	130	81
669	Cyclohexane	10	ug/kg	1	ug/kg	20010125														
539	1,2-Dibromo-3-chloropropane	10	ug/kg	1.6	ug/kg	20010222														
870	1,2-Dibromoethane	5	ug/kg	1.2	ug/kg	20010308														
904	1,2-Dichlorobenzene	5	ug/kg	0.71	ug/kg	20010122														
907	1,3-Dichlorobenzene	5	ug/kg	0.8	ug/kg	20010122														
910	1,4-Dichlorobenzene	5	ug/kg	0.67	ug/kg	20010122														
924	Dichlorodifluoromethane	5	ug/kg	1.1	ug/kg	20010222														
933	1,1-Dichloroethane	5	ug/kg	0.58	ug/kg	20010122		Y	20	ug/kg	77	119	30		Y	50	ug/kg	56	130	54
936	1,2-Dichloroethane	5	ug/kg	0.73	ug/kg	20010122		Y	20	ug/kg	78	121	30		Y	50	ug/kg	56	126	38
948	cis-1,2-Dichloroethene	2.5	ug/kg	0.84	ug/kg	20010122		Y	20	ug/kg	77	114	30		Y	50	ug/kg	48	127	52
950	trans-1,2-Dichloroethene	2.5	ug/kg	0.73	ug/kg	20010122		Y	20	ug/kg	68	117	30		Y	50	ug/kg	47	127	58
943	1,1-Dichloroethene	5	ug/kg	1.2	ug/kg	20010308	C	Y	20	ug/kg	55	142	27	C	Y	50	ug/kg	43	147	27
986	1,2-Dichloropropane	5	ug/kg	0.75	ug/kg	20010122		Y	20	ug/kg	78	116	30		Y	50	ug/kg	54	125	43
998	cis-1,3-Dichloropropene	5	ug/kg	0.64	ug/kg	20010122		Y	20	ug/kg	71	125	30		Y	50	ug/kg	30	138	49
1000	trans-1,3-Dichloropropene	5	ug/kg	0.86	ug/kg	20010125		Y	20	ug/kg	67	125	30		Y	50	ug/kg	34	134	57
1332	Ethylbenzene	5	ug/kg	0.59	ug/kg	20010122		Y	20	ug/kg	79	114	30		Y	50	ug/kg	36	133	72
1515	2-Hexanone	20	ug/kg	1.7	ug/kg	20010308		Y	20	ug/kg	29	200	41		Y	50	ug/kg	20	190	70
1578	Isopropylbenzene	5	ug/kg	0.7	ug/kg	20010122														
1774	Methyl acetate	10	ug/kg	1.1	ug/kg	20010122														
1799	Methylcyclohexane	10	ug/kg	0.59	ug/kg	20010122														
1811	Methylene chloride	5	ug/kg	1.3	ug/kg	20010308		Y	20	ug/kg	58	130	30		Y	50	ug/kg	45	129	49
1845	4-Methyl-2-pentanone	20	ug/kg	1.9	ug/kg	20010308		Y	20	ug/kg	68	142	60		Y	50	ug/kg	42	143	60
2772	Methyl tert-butyl ether	20	ug/kg	0.84	ug/kg	20010122														

Structured Analysis Code: A-I5-QK-01-01

Target Analyte List: NC: OLM03.1/4.2 Volatile List

Matrix: SOLID
Extraction: Indiana Modified Method 5035 (IN 5035-M)
Method: Volatile Organics, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 4121			Detection Limits				Check List 924								Spike List 925						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2355	Styrene	5	ug/kg	0.5	ug/kg	20010122		Y	20	ug/kg	80	114	30		Y	50	ug/kg	23	136	65
2439	1,1,2,2-Tetrachloroethane	5	ug/kg	1.8	ug/kg	20010308		Y	20	ug/kg	70	133	30		Y	50	ug/kg	33	162	90
2445	Tetrachloroethene	5	ug/kg	1.2	ug/kg	20010308		Y	20	ug/kg	72	120	30		Y	50	ug/kg	31	137	81
2489	Toluene	5	ug/kg	0.53	ug/kg	20010122	C	Y	20	ug/kg	71	130	24	C	Y	50	ug/kg	46	147	24
2515	1,2,4-Trichlorobenzene	5	ug/kg	0.89	ug/kg	20010122														
2518	1,1,1-Trichloroethane	5	ug/kg	0.92	ug/kg	20010122		Y	20	ug/kg	67	123	30		Y	50	ug/kg	48	132	57
2522	1,1,2-Trichloroethane	5	ug/kg	1.2	ug/kg	20010308		Y	20	ug/kg	82	116	30		Y	50	ug/kg	58	128	52
2525	Trichloroethene	5	ug/kg	0.76	ug/kg	20010122	C	Y	20	ug/kg	70	131	23	C	Y	50	ug/kg	46	143	23
1428	Trichlorofluoromethane	5	ug/kg	1.1	ug/kg	20010122														
2566	1,1,2-Trichloro-1,2,2-trifluoroethane	5	ug/kg	0.93	ug/kg	20010125														
2613	Vinyl chloride	5	ug/kg	1.4	ug/kg	20010122		Y	20	ug/kg	24	152	30		Y	50	ug/kg	30	136	80
2627	Xylenes (total)	5	ug/kg	1.9	ug/kg	20010122		Y	60	ug/kg	80	114	30		Y	150	ug/kg	33	135	78
337	4-Bromofluorobenzene						X	Y	50	ug/kg	47	158	0	X	Y	50	ug/kg	47	158	0
2735	1,2-Dichloroethane-d4						X	Y	50	ug/kg	61	130	0	X	Y	50	ug/kg	61	130	0
2740	Toluene-d8						X	Y	50	ug/kg	60	143	0	X	Y	50	ug/kg	60	143	0
2863	Dibromofluoromethane						X	Y	50	ug/kg	59	138	0	X	Y	50	ug/kg	59	138	0

STL Reference Data Summary

Structured Analysis Code: A-13-QL-01-01

Target Analyte List: NC: OLM03.1/4.2 Semivolatile List

Matrix: SOLID
Extraction: SONICATION - Low Level
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 4122			Detection Limits				Check List 1114							Spike List 1115						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
1	Acenaphthene	330	ug/kg	51	ug/kg	20010227	C	Y	1700	ug/kg	44	108	44	C	Y	1700	ug/kg	13	133	44
5	Acenaphthylene	330	ug/kg	47	ug/kg	20010227		Y	1700	ug/kg	55	110	30		Y	1700	ug/kg	43	120	75
24	Acetophenone	330	ug/kg	50	ug/kg	20010219														
122	Anthracene	330	ug/kg	49	ug/kg	20010227		Y	1700	ug/kg	57	116	30		Y	1700	ug/kg	34	137	83
158	Atrazine	330	ug/kg	150	ug/kg	20010329														
3398	Benzaldehyde	330	ug/kg	170	ug/kg	20010329														
202	Benzo(a)anthracene	330	ug/kg	55	ug/kg	20010220		Y	1700	ug/kg	56	110	30		Y	1700	ug/kg	30	136	63
205	Benzo(b)fluoranthene	330	ug/kg	58	ug/kg	20010220		Y	1700	ug/kg	54	116	30		Y	1700	ug/kg	31	139	67
208	Benzo(k)fluoranthene	330	ug/kg	53	ug/kg	20010220		Y	1700	ug/kg	54	110	36		Y	1700	ug/kg	30	133	84
210	Benzo(ghi)perylene	330	ug/kg	55	ug/kg	20010220		Y	1700	ug/kg	48	124	31		Y	1700	ug/kg	23	142	89
211	Benzo(a)pyrene	330	ug/kg	60	ug/kg	20010220		Y	1700	ug/kg	56	115	30		Y	1700	ug/kg	28	142	86
3474	1,1'-Biphenyl	330	ug/kg	140	ug/kg	20010329														
289	bis(2-Chloroethoxy)methane	330	ug/kg	47	ug/kg	20010227		Y	1700	ug/kg	59	110	30		Y	1700	ug/kg	46	116	97
293	bis(2-Chloroethyl) ether	330	ug/kg	51	ug/kg	20010227		Y	1700	ug/kg	62	111	30		Y	1700	ug/kg	49	123	93
302	bis(2-Ethylhexyl) phthalate	330	ug/kg	54	ug/kg	20010220		Y	1700	ug/kg	53	115	30		Y	1700	ug/kg	36	131	54
348	4-Bromophenyl phenyl ether	330	ug/kg	61	ug/kg	20010131		Y	1700	ug/kg	58	110	30		Y	1700	ug/kg	47	121	95
403	Butyl benzyl phthalate	330	ug/kg	51	ug/kg	20010220		Y	1700	ug/kg	54	113	30		Y	1700	ug/kg	34	136	94
5101	Caprolactam	330	ug/kg	180	ug/kg	20010329														
2751	Carbazole	330	ug/kg	52	ug/kg	20010227		Y	1700	ug/kg	56	117	31		Y	1700	ug/kg	40	130	85
518	4-Chloroaniline	330	ug/kg	51	ug/kg	20010220		Y	1700	ug/kg	28	110	33		Y	1700	ug/kg	10	110	99
578	4-Chloro-3-methylphenol	330	ug/kg	51	ug/kg	20010131	C	Y	1700	ug/kg	43	110	55	C	Y	1700	ug/kg	17	128	55
589	2-Chloronaphthalene	330	ug/kg	48	ug/kg	20010227		Y	1700	ug/kg	52	116	30		Y	1700	ug/kg	43	125	75
600	2-Chlorophenol	330	ug/kg	52	ug/kg	20010227	C	Y	1700	ug/kg	43	110	54	C	Y	1700	ug/kg	17	116	54
602	4-Chlorophenyl phenyl ether	330	ug/kg	52	ug/kg	20010227		Y	1700	ug/kg	59	110	30		Y	1700	ug/kg	47	119	74
633	Chrysene	330	ug/kg	46	ug/kg	20010424		Y	1700	ug/kg	56	110	30		Y	1700	ug/kg	28	139	63
860	Dibenz(a,h)anthracene	330	ug/kg	51	ug/kg	20010220		Y	1700	ug/kg	50	129	33		Y	1700	ug/kg	31	142	99
863	Dibenzofuran	330	ug/kg	50	ug/kg	20010227		Y	1700	ug/kg	57	110	30		Y	1700	ug/kg	35	132	58
891	Di-n-butyl phthalate	330	ug/kg	150	ug/kg	20010329		Y	1700	ug/kg	58	114	30		Y	1700	ug/kg	44	128	74
918	3,3'-Dichlorobenzidine	1600	ug/kg	39	ug/kg	20010220		Y	1700	ug/kg	22	110	33		Y	1700	ug/kg	10	110	99
971	2,4-Dichlorophenol	330	ug/kg	50	ug/kg	20010220		Y	1700	ug/kg	60	111	30		Y	1700	ug/kg	45	122	85
1082	Diethyl phthalate	330	ug/kg	150	ug/kg	20010329		Y	1700	ug/kg	57	112	30		Y	1700	ug/kg	45	122	76
1145	2,4-Dimethylphenol	330	ug/kg	80	ug/kg	20010430		Y	1700	ug/kg	41	110	35		Y	1700	ug/kg	31	119	99
1149	Dimethyl phthalate	330	ug/kg	160	ug/kg	20010329		Y	1700	ug/kg	60	111	30		Y	1700	ug/kg	48	124	75
1167	4,6-Dinitro-2-methylphenol	1600	ug/kg	160	ug/kg	20010329		Y	1700	ug/kg	37	126	37		Y	1700	ug/kg	10	142	99
1187	2,4-Dinitrophenol	1600	ug/kg	110	ug/kg	20010329		Y	1700	ug/kg	15	131	53		Y	1700	ug/kg	10	149	99
1191	2,4-Dinitrotoluene	330	ug/kg	46	ug/kg	20010227	C	Y	1700	ug/kg	48	111	45	C	Y	1700	ug/kg	10	171	45

Structured Analysis Code: A-13-QL-01-01

Target Analyte List: NC: OLM03.1/4.2 Semivolatile List

Matrix: SOLID
Extraction: SONICATION - Low Level
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

	Target List 4122	Detection Limits					Check List 1114							Spike List 1115						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
1193	2,6-Dinitrotoluene	330	ug/kg	48	ug/kg	20010131		Y	1700	ug/kg	61	121	30		Y	1700	ug/kg	45	124	99
1162	Di-n-octyl phthalate	330	ug/kg	54	ug/kg	20010220		Y	1700	ug/kg	47	129	33		Y	1700	ug/kg	34	143	99
1414	Fluoranthene	330	ug/kg	56	ug/kg	20010227		Y	1700	ug/kg	57	118	30		Y	1700	ug/kg	25	150	92
1417	Fluorene	330	ug/kg	48	ug/kg	20010227		Y	1700	ug/kg	57	110	30		Y	1700	ug/kg	35	133	80
1482	Hexachlorobenzene	330	ug/kg	56	ug/kg	20010131		Y	1700	ug/kg	57	111	30		Y	1700	ug/kg	45	123	74
1489	Hexachlorobutadiene	330	ug/kg	45	ug/kg	20010227		Y	1700	ug/kg	56	113	30		Y	1700	ug/kg	43	126	86
1492	Hexachlorocyclopentadiene	1600	ug/kg	60	ug/kg	20010220		Y	1700	ug/kg	10	113	57		Y	1700	ug/kg	10	110	99
1497	Hexachloroethane	330	ug/kg	48	ug/kg	20010227		Y	1700	ug/kg	61	110	30		Y	1700	ug/kg	36	134	97
1535	Indeno(1,2,3-cd)pyrene	330	ug/kg	70	ug/kg	20010220		Y	1700	ug/kg	44	128	54		Y	1700	ug/kg	20	147	73
1566	Isophorone	330	ug/kg	44	ug/kg	20010227		Y	1700	ug/kg	58	110	30		Y	1700	ug/kg	45	116	95
1829	2-Methylnaphthalene	330	ug/kg	48	ug/kg	20010227		Y	1700	ug/kg	58	110	30		Y	1700	ug/kg	30	137	79
1851	2-Methylphenol	330	ug/kg	46	ug/kg	20010220		Y	1700	ug/kg	51	110	30		Y	1700	ug/kg	41	116	99
1857	4-Methylphenol	330	ug/kg	47	ug/kg	20010227		Y	1700	ug/kg	56	110	30		Y	1700	ug/kg	36	123	86
1932	Naphthalene	330	ug/kg	49	ug/kg	20010227		Y	1700	ug/kg	59	110	30		Y	1700	ug/kg	31	138	65
1960	2-Nitroaniline	1600	ug/kg	46	ug/kg	20010227		Y	1700	ug/kg	58	113	30		Y	1700	ug/kg	42	126	99
1964	3-Nitroaniline	1600	ug/kg	140	ug/kg	20010329		Y	1700	ug/kg	42	110	37		Y	1700	ug/kg	17	113	99
1968	4-Nitroaniline	1600	ug/kg	68	ug/kg	20010424		Y	1700	ug/kg	42	116	40		Y	1700	ug/kg	14	125	99
1972	Nitrobenzene	330	ug/kg	46	ug/kg	20010227		Y	1700	ug/kg	62	110	30		Y	1700	ug/kg	49	120	99
1998	2-Nitrophenol	330	ug/kg	46	ug/kg	20010131		Y	1700	ug/kg	55	118	30		Y	1700	ug/kg	44	125	99
2001	4-Nitrophenol	1600	ug/kg	190	ug/kg	20010329	C	Y	1700	ug/kg	22	128	64	C	Y	1700	ug/kg	10	148	64
2028	N-Nitrosodiphenylamine	330	ug/kg	49	ug/kg	20010227		Y	1700	ug/kg	58	113	30		Y	1700	ug/kg	37	135	94
2024	N-Nitrosodi-n-propylamine	330	ug/kg	44	ug/kg	20010220	C	Y	1700	ug/kg	38	110	50	C	Y	1700	ug/kg	12	128	50
3597	2,2'-oxybis(1-Chloropropane)	330	ug/kg	52	ug/kg	20010227														
2118	Pentachlorophenol	330	ug/kg	120	ug/kg	20010329	C	Y	1700	ug/kg	10	123	87	C	Y	1700	ug/kg	10	144	87
2154	Phenanthrene	330	ug/kg	52	ug/kg	20010227		Y	1700	ug/kg	56	110	30		Y	1700	ug/kg	24	145	81
2155	Phenol	330	ug/kg	49	ug/kg	20010227	C	Y	1700	ug/kg	35	110	50	C	Y	1700	ug/kg	10	148	50
2252	Pyrene	330	ug/kg	51	ug/kg	20010220	C	Y	1700	ug/kg	42	122	66	C	Y	1700	ug/kg	10	218	66
2555	2,4,5-Trichlorophenol	330	ug/kg	55	ug/kg	20010227		Y	1700	ug/kg	52	115	37		Y	1700	ug/kg	39	123	99
2559	2,4,6-Trichlorophenol	330	ug/kg	63	ug/kg	20010220		Y	1700	ug/kg	51	112	30		Y	1700	ug/kg	43	123	99
1425	2-Fluorobiphenyl						X	Y	1700	ug/kg	43	110	0	X	Y	1700	ug/kg	43	110	0
1426	2-Fluorophenol						X	Y	2500	ug/kg	11	116	0	X	Y	2500	ug/kg	11	116	0
2512	2,4,6-Tribromophenol						X	Y	2500	ug/kg	35	116	0	X	Y	2500	ug/kg	35	116	0
2736	Nitrobenzene-d5						X	Y	1700	ug/kg	42	110	0	X	Y	1700	ug/kg	42	110	0
2737	Phenol-d5						X	Y	2500	ug/kg	25	115	0	X	Y	2500	ug/kg	25	115	0
2738	Terphenyl-d14						X	Y	1700	ug/kg	37	137	0	X	Y	1700	ug/kg	37	137	0

STL Reference Data Summary

Structured Analysis Code: A-71-QH-01-01

Target Analyte List: Q: Aroclors Only Standard List

Matrix: SOLID
Extraction: SONICATION w/ACID STRIP (PCB)
Method: PCBs (8082)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 2207		Detection Limits				Check List 928							Spike List 929						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2082	Aroclor 1016	33	ug/kg	5.3	ug/kg	20000222	C	Y	333	ug/kg	49	122	39	C	Y	333	ug/kg	26	144	39
2085	Aroclor 1221	33	ug/kg	19	ug/kg	20000222														
2088	Aroclor 1232	33	ug/kg	11	ug/kg	20000222														
2091	Aroclor 1242	33	ug/kg	18	ug/kg	20000222														
2094	Aroclor 1248	33	ug/kg	4.6	ug/kg	20000222														
2097	Aroclor 1254	33	ug/kg	20	ug/kg	20000222														
2100	Aroclor 1260	33	ug/kg	7.4	ug/kg	20000222	C	Y	333	ug/kg	51	127	33	C	Y	333	ug/kg	37	138	33
2732	Decachlorobiphenyl						X	Y	6.67	ug/kg	23	141	0	X	Y	6.67	ug/kg	23	141	0
2739	Tetrachloro-m-xylene						X	Y	6.67	ug/kg	31	127	0	X	Y	6.67	ug/kg	31	127	0

STL Reference Data Summary

Structured Analysis Code: A-46-QO-01-01

Target Analyte List: Q: CLP ICP Metals TAL Standard List

Matrix: SOLID
Extraction: METALS, TOTAL - Soils
Method: Inductively Coupled Plasma (6010B)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 2600			Detection Limits				Check List 906							Spike List 907						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
88	Aluminum	20	mg/kg	1.1	mg/kg	20000224		Y	200	mg/kg	80	120	20		Y	200	mg/kg	75	125	20
128	Antimony	6	mg/kg	0.49	mg/kg	20000224		Y	50	mg/kg	80	120	20		Y	50	mg/kg	75	125	20
194	Barium	20	mg/kg	0.13	mg/kg	20000224		Y	200	mg/kg	80	120	20		Y	200	mg/kg	75	125	20
222	Beryllium	0.5	mg/kg	0.046	mg/kg	20000224		Y	5	mg/kg	80	120	20		Y	5	mg/kg	75	125	20
411	Cadmium	0.5	mg/kg	0.043	mg/kg	20000224		Y	5	mg/kg	80	120	20		Y	5	mg/kg	75	125	20
413	Calcium	500	mg/kg	37	mg/kg	20000224		Y	5000	mg/kg	80	120	20		Y	5000	mg/kg	75	125	20
2952	Chromium	1	mg/kg	0.38	mg/kg	20000412		Y	20	mg/kg	80	120	20		Y	20	mg/kg	75	125	20
637	Cobalt	5	mg/kg	0.15	mg/kg	20000224		Y	50	mg/kg	80	120	20		Y	50	mg/kg	75	125	20
643	Copper	2.5	mg/kg	0.27	mg/kg	20000412		Y	25	mg/kg	80	120	20		Y	25	mg/kg	75	125	20
1539	Iron	10	mg/kg	6.6	mg/kg	20000412		Y	100	mg/kg	73	137	20		Y	100	mg/kg	75	125	20
1618	Magnesium	500	mg/kg	12	mg/kg	20000224		Y	5000	mg/kg	80	120	20		Y	5000	mg/kg	75	125	20
1659	Manganese	1.5	mg/kg	0.15	mg/kg	20000224		Y	50	mg/kg	80	120	20		Y	50	mg/kg	75	125	20
1956	Nickel	4	mg/kg	0.27	mg/kg	20000224		Y	50	mg/kg	80	120	20		Y	50	mg/kg	75	125	20
2214	Potassium	500	mg/kg	5.1	mg/kg	20000224		Y	5000	mg/kg	80	120	20		Y	5000	mg/kg	75	125	20
2285	Silver	1	mg/kg	0.15	mg/kg	20000224		Y	5	mg/kg	80	120	20		Y	5	mg/kg	75	125	20
2315	Sodium	500	mg/kg	50	mg/kg	20000224		Y	5000	mg/kg	80	120	20		Y	5000	mg/kg	75	125	20
2607	Vanadium	5	mg/kg	0.13	mg/kg	20000224		Y	50	mg/kg	80	120	20		Y	50	mg/kg	75	125	20
2649	Zinc	2	mg/kg	1.2	mg/kg	20000412		Y	50	mg/kg	80	120	20		Y	50	mg/kg	75	125	20

STL Reference Data Summary

Structured Analysis Code: A-46-QM-01-01

Target Analyte List: Q: CLP Trace ICP Metals TAL Standard List

Matrix: SOLID
Extraction: METALS, TOTAL - Soils
Method: Inductively Coupled Plasma (6010B Trace)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 2610		Detection Limits				Check List 906							Spike List 907						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
140	Arsenic	1.0	mg/kg	0.3	mg/kg	20000224		Y	200	mg/kg	80	120	20		Y	200	mg/kg	75	125	20
1605	Lead	0.3	mg/kg	0.24	mg/kg	20000224		Y	50	mg/kg	80	120	20		Y	50	mg/kg	75	125	20
2281	Selenium	0.5	mg/kg	0.31	mg/kg	20000224		Y	200	mg/kg	80	120	20		Y	200	mg/kg	75	125	20
2477	Thallium	1.0	mg/kg	0.5	mg/kg	20000224		Y	200	mg/kg	80	120	20		Y	200	mg/kg	75	125	20

STL Reference Data Summary

Structured Analysis Code: A-70-O9-01-01
Target Analyte List: All Analytes

Matrix: SOLID
Extraction: METALS, TOTAL (Method Exclusive) - Solids
Method: Mercury (7471A, Cold Vapor) - Solids
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits					Check List 1021							Spike List 1026						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
1701	Mercury	0.1	mg/kg	0.0084	mg/kg	20000613		Y	.833333	mg/kg	52	127	20		Y	0.1666	mg/kg	10	209	20

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STL Reference Data Summary

Structured Analysis Code: A-06-QP-01-01
Target Analyte List: All Analytes

Matrix: SOLID
Extraction: DISTILLATION, MICRO/MIDI - Acid
Method: Cyanide, Total (9012A, Automated)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List			Detection Limits				Check List 1180							Spike List 1181						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
667	Total Cyanide	0.5	mg/kg	0.17	mg/kg	20000727		Y			65	124	20		Y	2.0	mg/kg	23	155	94

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STL Reference Data Summary

Structured Analysis Code: A-06-N4-01-01

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: DISTILLATION, MICRO/MIDI - Acid
Method: Cyanide, Amenable (9012)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List			Detection Limits				Check List 1180							Spike List 1181						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3623	Amenable Cyanide	0.5	mg/kg	0.5	mg/kg	20010927		Y			65	124	20		Y	2.0	mg/kg	23	155	94
664	Cyanide Amenable to Chlorination	0.5	mg/kg	0.5	mg/kg	20010927		Y			65	124	20		Y	2.0	mg/kg	23	155	94

STL Reference Data Summary

Structured Analysis Code: A-82-VR-01-01
Target Analyte List: All Analytes

Matrix: SOLID
Extraction: LEACHATE, DI (Routine)
Method: Carbon, Total Organic "TOC" (WALKLEY) - Solids
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List			Detection Limits				Check List 1215							Spike List 1215						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
470	Total Organic Carbon	100	mg/kg	100	mg/kg	20010924	C	Y			51	128	20	C	Y			51	128	20

STL Reference Data Summary

Structured Analysis Code: D-1U-QH-01-01

Target Analyte List: Q: Aroclors Only Standard List

Matrix: WIPE
Extraction: EXTRACTION, SOLID/SOLVENT (Auto shaker) w/ACID STR
Method: PCBs (8082)
QC Program: STANDARD TEST SET
Location: STL North Canton

Target List 2207		Detection Limits				Check List 928							Spike List 929						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2082	Aroclor 1016	4	ug	4	ug	20001222	C	Y	10	mg/kg	90	138	30	C	Y			26	144	39
2085	Aroclor 1221	4	ug	4	ug	20001222														
2088	Aroclor 1232	4	ug	4	ug	20001222														
2091	Aroclor 1242	4	ug	4	ug	20001222														
2094	Aroclor 1248	4	ug	4	ug	20001222														
2097	Aroclor 1254	4	ug	4	ug	20001222														
2100	Aroclor 1260	4	ug	4	ug	20001222														
2732	Decachlorobiphenyl						C	Y	10	mg/kg	88	125	30	C	Y			37	138	33
2739	Tetrachloro-m-xylene						X	Y	0.2	mg/kg	64	181	0	X	Y	0.2	mg/kg	64	181	0
							X	Y	0.2	mg/kg	66	135	0	X	Y	0.2	mg/kg	66	135	0

ATTACHMENT 4

FIELD AUDIT CHECKLIST

FIELD CHECKLIST

Briefing with Project Manager

SIGNATURE OF AUDITOR _____ DATE OF AUDIT _____

PROJECT MANAGER _____ PROJECT NO. _____

PROJECT LOCATION _____

TYPE OF INVESTIGATION _____
(authority, agency)

Yes ___ No ___ N/A ___ 1. Was a project plan prepared? If yes, what items are addressed in the Plan?

_____

_____

_____

Yes ___ No ___ N/A ___ 2. Were additional instructions given to project participants (i.e., changes in project plan)? If yes, describe these changes.

_____

_____

_____

Yes ___ No ___ N/A ___ 3. Is there a written list of sampling locations and descriptions? If yes, describe where documents are.

_____

_____

_____

Yes ___ No ___ N/A ___ 4. Is there a map of sampling location? If yes, where is the map?

_____

_____

_____

FIELD CHECKLIST

Field Observations

Yes ___ No ___ N/A ___ 1. Are the number, frequency and types of field measurements and observations taken as specified in the project plan or as directed by the project manager? If yes, where are they recorded?

Yes ___ No ___ N/A ___ 2. Are samples collected in the types of containers specified for each type of analysis? If no, what kind of sample containers are used?

Yes ___ No ___ N/A ___ 3. Are samples preserved as required? If no or N/A, explain.

Yes ___ No ___ N/A ___ 4. Are the number, frequency and types of samples collected as specified in the project plan or as directed by the project manager? If no, explain why not.

Yes ___ No ___ N/A ___ 5. Are samples packed for preservation when required (i.e., packed in ice, etc.)? If no or N/A, explain why.

Yes ___ No ___ N/A ___ 6. Is sample custody maintained? How?

FIELD CHECKLIST

Document Control

Yes ___ No ___ N/A ___ 1. Are samples identified with sample tags? If no, how are samples identified?

Yes ___ No ___ N/A ___ 2. Are sample tags completed (e.g., station no., location, date, time, analyses, signatures on samples, type, preservatives, etc.)? If yes, describe types of information recorded.

Yes ___ No ___ N/A ___ 3. Are samples collected listed on a chain-of-custody record? If yes, describe the type of chain-of-custody record used and what information is recorded.

Yes ___ No ___ N/A ___ 4. If used, are the sample tag numbers recorded on the chain-of-custody documents?

Yes ___ No ___ N/A ___ 5. Does information on sample tags and chain-of-custody records match?

Yes ___ No ___ N/A ___ 6. Does the chain-of-custody record indicate the method of sample shipment?

Yes ___ No ___ N/A ___ 7. Is the chain-of-custody record included with the samples in the shipping container?

Yes ___ No ___ N/A ___ 8. If used, do the sample traffic reports agree with the sample tags?

Yes ___ No ___ N/A ___ 9. If used, are blank samples identified?

Yes ___ No ___ N/A ___ 10. If collected, are duplicate samples identified on sample tags and chain-of-custody records?

Yes ___ No ___ N/A ___ 11. If used, are spiked samples identified?

Yes ___ No ___ N/A ___ 12. Are logbooks signed by the individual who checked out the logbook from the project coordinator?

Yes ___ No ___ N/A ___ 13. Are logbooks dated upon receipt from the project coordinator?

Yes ___ No ___ N/A ___ 14. Are logbooks project-specific (by logbook or by page)?

Yes ___ No ___ N/A ___ 15. Are logbook entries dated and identified by author?

Yes ___ No ___ N/A ___ 16. Are photographs documented in logbooks (e.g., time, date, description of subject, photographer, etc.)?

Yes ___ No ___ N/A ___ 17. If film from a self-developing camera is used, are photos matched with logbook documentation?

Yes ___ No ___ N/A ___ 18. Are sample tag numbers recorded? If yes, describe where they are recorded.

Yes ___ No ___ N/A ___ 19. Are calibration of pH meters, conductivity meters, etc., documented? If yes, describe where this is documented.

Yes ___ No ___ N/A ___ 20. Are amendments to the project plan documented? If yes, describe where the amendments are documented.

FIELD CHECKLIST

Debriefing with Project Manager

Yes ___ No ___ N/A ___ 1. Was a debriefing held with project manager and/or other participants?

Yes ___ No ___ N/A ___ 2. Were any recommendations made to the project participants during the debriefing? If yes, list recommendations.

ATTACHMENT 5

LABORATORY AUDIT CHECKLIST

LABORATORY AUDIT CHECKLIST

SIGNATURE OF AUDITOR _____ DATE OF AUDIT _____

PROJECT MANAGER _____ PROJECT NO. _____

PROJECT LOCATION _____

TYPE OF INVESTIGATION _____
(authority, agency)

The enclosed laboratory audit checklists are organized into the categories by laboratory functional area. The attached audit checklists have been numbered according to the following outline:

1.0 Sample Management
2.0 Organic Sample Preparation
3.0 Gas Chromatography
4.0 Mass Spectrometry
5.0 Metals Preparation
6.0 Metals Analysis
7.0 Wet Chemistry
8.0 Report Generation
9.0 Computer Systems

Special Checklists

Form A Laboratory Temperature Monitoring Checklist
Form B Laboratory Notebook Checklist
Form C Laboratory Balance Checklist
Form D Reagent and Analytical Standards Checklist
Form E Laboratory Safety Checklist

LABORATORY AUDIT CHECKLIST

1.0 Sample Management

Shipment of Sample Bottles and Coolers

Check off which of the following items are utilized in this area and complete the associated forms.			
		Refrigerators, freezers, ovens,	Complete Form A
		Logbooks	Complete Form B
		Laboratory Balances	Complete Form C
		Reagents and Standards	Complete Form D
		Safety Equipment	Complete Form E

Y	N	Item	Comments
		Is there a SOP describing preparation and shipment of sample coolers?	
		Are sample bottles precleaned and is cleaning documented?	
		Are cooler shipment records maintained?	
		Are coolers and bottles sent out under chain-of-custody?	
		Are preservatives provided with the cooler?	
		Are sample collectors supplied with preservatives and sampling instructions for their use?	
		Is there a SOP describing the amount and concentration of preservation agents added to bottles?	
		Are chain of custody forms provided along with instructions for proper completion?	
		Are trip blanks included with the cooler?	
		If so, is the purity of the trip blank water checked before shipment and can it be documented?	
		Does the laboratory only utilize new sample containers?	

LABORATORY AUDIT CHECKLIST

1.0 Sample Management (continued)

Receipt and Storage of Samples

Check off which of the following items are utilized in this area and complete the associated forms.			
		Refrigerators, freezers, ovens,	Complete Form A
		Logbooks	Complete Form B
		Laboratory Balances	Complete Form C
		Reagents and Standards	Complete Form D
		Safety Equipment	Complete Form E

Y	N	Item	Comment
		Do the laboratory personnel indicate a familiarity with the requirements set forth in the laboratory QA program?	
		Is there a written SOP readily available to the login personnel?	
		Is the SOP followed as written?	
		If discrepancies are noted between COC records and the samples, are they documented and appropriate personnel notified?	
		Is the pH checked on all appropriate incoming samples and documented?	
		Is the pH check done in such a manner that avoids sample contamination?	
		Is HCl preservation utilized for volatile sample preservation? If not, is a 7-day hold time being applied?	
		Are incoming volatile vials checked for the presence of air bubbles?	
		If air bubbles are detected, is the client notified?	
		If there a mechanism to receive samples after normal work hours and weekends?	
		Is the filing system for original custody forms and paperwork traceable?	
		Is a fume hood available for opening of hazardous samples?	
		Are eyewashes and/or showers available in the immediate area?	
		Is there a central log-in notebook and is it document controlled?	
		Are unique laboratory sample identification numbers assigned and properly recorded on sample container labels and sample log-in documentation?	

LABORATORY AUDIT CHECKLIST

1.0 Sample Management (continued)

Y	N	Item	Comment
		Is there a SOP describing how the lab handles samples with short holding times?	
		Are samples checked for proper preservation upon receipt? (Required by NJ)	
		If samples are not properly preserved, is there a SOP describing how it is handled?	
		Are files maintained for log-in documentation?	
		Are all samples and extracts stored in a secure manner? (Locked refrigerators and freezers)	
		Are all samples and extracts stored in separate storage units?	
		Is there limited access to sample and extract storage areas?	
		Is there an internal custody procedure for samples and extracts and is it documented in a SOP? If so, where are records filed?	
		Is there a procedure for documenting where samples and extracts are stored?	
		Are volatile samples stored separately from other samples or extracts?	
		Are volatile water samples stored separately from soil volatile samples?	
		Are refrigerator holding blanks utilized to monitor potential sample contamination in each volatile sample storage unit?	
		Are standards or reagents stored with samples or extracts?	
		Are samples maintained in a cooled condition until holding time expires?	
		Are archived samples stored in a secure manner?	
		Is there a policy describing how long samples are held after a report is issued?	
		Are samples being held after report issuance maintained in a cooled condition?	
		Is there a SOP describing the archival and disposal of samples and sample extracts?	

LABORATORY AUDIT CHECKLIST

1.0 Sample Management (continued)

Y	N	Item	Comment
		Does the disposal SOP describe how the lab disposes of samples of unknown content?	
		Is there a record of when every sample has been discarded?	
		Do supervisory personnel review the paperwork in the dept. and is this review documented?	

LABORATORY AUDIT CHECKLIST

2.0 Organic Sample Preparation

Check off which of the following items are utilized in this area and complete the associated forms.			
		Refrigerators, freezers, ovens,	Complete Form A
		Logbooks	Complete Form B
		Laboratory Balances	Complete Form C
		Reagents and Standards	Complete Form D
		Safety Equipment	Complete Form E

Y	N	Item	Comment
		Are SOPs available detailing the procedures used in this area?	Obtain a listing of all procedures performed in the area. Compare the list with available SOPs and estimate a percentage complete below. Percent Compliance _____
Are the following QC samples processed with each analytical batch?			
		Method Blank	
		QC Check Sample	
		Matrix Spike	
		Matrix Spike Duplicate	
		Are the solvent storage cabinets properly vented as appropriate to avoid possible laboratory contamination?	
		Are gas lines checked for leaks each time a tank is changed?	
		If the lab utilizes desiccators, do they appear to be properly maintained?	
		Is there an SOP for glassware washing and is it posted in the washing area?	
		Are operating manuals available to the analysts for all major equipment?	
		Is there a record of instrument servicing maintained in a logbook and is it document controlled?	
		Is appropriate corrective action taken if a surrogate is outside of acceptable limits?	
		Are boiling chips kept to a minimum size to prevent volumetric distortion and minimize sample contamination potential? (only 1 boiling chip, the size of a grain of salt should be used)	
		Are graduated cylinders utilized for measuring out sample volumes for water extractions?	

LABORATORY AUDIT CHECKLIST

2.0 Organic Sample Preparation (continued)

Y	N	Item	Comment
		Are amber screw cap vials utilized for solvent extracts? (screw cap vials prevent solvent losses associated with crimp top vials)	
		If the volume of solvent extracts is reduced utilizing gas blowdown systems, do the gas lines contain molecular sieves to remove contaminants from the gas supply?	
		Are surrogates added to samples utilizing class A pipets in a manner to ensure quantitative transfer of the surrogates?	
		Are various dry reagents such as florisil, sodium sulfate and glass wool or thimbles, prextracted prior to use?	
		Is florisil PR 60/100 mesh utilized and is it stored in a dark glass bottle with foil lined cap and initially activated at 676 deg. C and at 130 deg. before use?	
		Is anhydrous sodium sulfate used which is dried at 400 deg. C for 4 hours and stored at 130 deg. before use?	
		Are only approved solvent lot numbers being utilized by the laboratory?	
		Is laboratory glassware rotated so that method blanks are truly representative of the effectiveness of routine glassware cleaning procedures? (dedicated glassware must not be used for QC samples) and only 1 blank can be extracted per batch.	
		When utilizing liquid/liquid continuous extractors is all the solvent remaining after extraction in both the extractor and the boiling flask concentrated down to the final volume?	
		Do the laboratory personnel indicate a familiarity with the requirements set forth in the corporate QA program?	

LABORATORY AUDIT CHECKLIST

3.0 Gas Chromatography

Check off which of the following items are utilized in this area and complete the associated forms.			
		Refrigerators., freezers, ovens,	Complete Form A
		Logbooks	Complete Form B
		Laboratory Balances	Complete Form C
		Reagents and Standards	Complete Form D
		Safety Equipment	Complete Form E

Y	N	Item	Comment
		Do the laboratory personnel indicate a familiarity with the requirements set forth in the laboratory QA program?	
		Are SOPs available detailing the procedures used in this area?	Obtain a listing of all procedures performed in the area. Compare the list with available SOPs and estimate a percentage complete below. Percent Compliance _____
		Are gas lines checked for leaks each time a tank is changed?	
		Are instruments properly vented or are appropriate traps in place?	
		Can the lab demonstrate the absence of volatile organics in the lab reagent water?	
		Are SOPs available detailing the procedures used in this area?	
		Are the solvent storage cabinets properly vented as appropriate to avoid possible laboratory contamination?	
		Are operating manuals available to the analysts for all major equipment?	
		Is there a record of instrument servicing maintained in a logbook and is it document controlled?	
		Are volatile analysis being conducted using the appropriate packed or capillary columns as specified by the particular method?	
		Is there a policy in place concerning carryover prevention in the event a high level sample precedes a relatively low level sample?	
		Is quantitation correct for the method? (based upon the average RF of a calibration curve or the RF from a continuing calibration standard)	

LABORATORY AUDIT CHECKLIST

3.0 Gas Chromatography (continued)

Y	N	Item	Comment
		Is the initial calibration conducted at five or three concentrations as appropriate for the method?	
		Are all reported results within the calibration range of the highest and lowest standard?	
		Are calibrations performed at the required frequency specified in the methods?	
		Is appropriate corrective action taken if a calibration is out of criteria?	
		Is a method blank run with every batch of samples?	
		Is there specified acceptance limits for the method blank?	
		Is there a policy stating that the laboratory does not "blank correct" sample data?	
		Is appropriate corrective action taken if a surrogate is outside of acceptance limits?	
		Is appropriate corrective action taken if internal standard area is outside of acceptance limits?	
		Is a run log maintained for all instrumentation?	
		Are GC identifications confirmed on a second dissimilar column?	
		Are PCB identifications performed by manual pattern recognition?	
		Are a sufficient number of peaks used to calculate a PCB result? (minimum of 3)	
		Are various extract cleanup techniques such as copper, florisil, alumina, sulfuric acid, used routinely?	
		Do gas lines contain molecular sieves or other filters to remove contaminants from the gas supply?	
		If 25 ml purge vessels are utilized for methods normally requiring a 5 ml purge, have MDL studies been performed to verify that required MDLs are achieved?	

4.0 Mass Spectrometry

Volatile GC/MS Area

Check off which of the following items are utilized in this area and complete the associated forms.

		Refrigerators., freezers, ovens,	Complete Form A
		Logbooks	Complete Form B
		Laboratory Balances	Complete Form C
		Reagents and Standards	Complete Form D
		Safety Equipment	Complete Form E

Y	N	Item	Comment
		Do the laboratory personnel indicate a familiarity with the requirements set forth in the laboratory QA program?	
		Are SOPs available detailing the procedures used in this area?	Obtain a listing of all procedures performed in the area. Compare the list with available SOPs and estimate a percentage complete below. Percent Compliance _____
Are the following QC samples processed with each analytical batch?			
		Method Blank	
		QC Check Sample	
		Matrix Spike	
		Matrix Spike Duplicate	
		Are gas lines checked for leaks each time a tank is changed?	
		Are the instruments properly vented or are appropriate traps in place?	
		Can the lab demonstrate the absence of volatile organics in the lab reagent water?	
		Are the solvent storage cabinets properly vented as appropriate to avoid possible laboratory contamination?	
		Is the laboratory utilizing the most recent version of the EPA/NIH mass spectral database? If not what version is being used?	
		Are operating manuals available to the analysis for all major equipment?	
		Is there a record of instrument servicing maintained in a logbook and is it document controlled?	
		Are volatile analyses being conducted using the appropriate packed or capillary columns as specified by the particular method?	
		Is there a policy in place concerning carryover prevention in the event a high level sample precedes a relatively low level sample?	

LABORATORY AUDIT CHECKLIST

4.0 Mass Spectrometry (continued)

Volatile GC/MS Area

Y	N	Item	Comment
		Is the initial calibration conducted at five or three concentrations as appropriate for the method?	
		Is quantitation correct for the method? (based upon the average RF of a calibration curve or the RF from a continuing calibration standard)	
		Are calibrations and tuning performed according the required frequency specified in the methods?	
		Is appropriate corrective action taken if a calibration is out of criteria?	
		Is appropriate corrective action taken if a continuing calibration is out of criteria?	
		Is a method blank run with every batch of samples?	
		Is there specified acceptance limits for the method blank?	
		Is there a policy stating that the laboratory does not "blank correct" sample data?	
		Is the appropriate corrective action taken if a surrogate is outside of acceptance limits?	
		Is the appropriate corrective action taken if internal standard area is outside of acceptance limits?	
		Are library searches examined by the mass spectrometry expert before results are reported and is there documentation of this review?	
		Is a run log maintained for all instrumentation and is it documented controlled?	
		Is raw data stored in a secure manner under appropriate conditions to prevent loss of data?	
		Are the reported results within the calibration range of the highest and lowest standard?	
		Are appropriate dilutions performed?	
		Do gas lines contain molecular sieves or other filters to remove contaminants in gas supply?	
		If 25-ml purge vessels are utilized for methods normally requiring a 5-ml purge, have MDL studies been performed to verify that required MDLs are achieved?	

LABORATORY AUDIT CHECKLIST

4.0 Mass Spectrometry (continued)

Semi-Volatile GC/MS Area			
Check off which of the following items are utilized in this area and complete the associated forms.			
		Refrigerators., freezers, ovens,	Complete Form A
		Logbooks	Complete Form B
		Laboratory Balances	Complete Form C
		Reagents and Standards	Complete Form D
		Safety Equipment	Complete Form E

Y	N	Item	Comment
		Do the laboratory personnel indicate a familiarity with the requirements set forth in the laboratory QA program?	
		Are SOPs available detailing the procedures used in this area?	Obtain a listing of all procedures performed in the area. Compare the list with available SOPs and estimate a percentage complete below. Percent Compliance _____
		Are gas lines checked for leaks each time a tank is changed?	
		Are the instruments properly vented or are appropriate traps in place?	
		Can the lab demonstrate the absence of volatile organics in the lab reagent water?	
		Are the solvent storage cabinets properly vented as appropriate to avoid possible laboratory contamination?	
		Is the laboratory utilizing the most recent version of the EPA/NIH mass spectral database? If not what version is being used?	
		Are operating manuals available to the analysis for all major equipment?	
		Is there a record of instrument servicing maintained in a logbook and is it document controlled?	
		Are volatile analyses being conducted using the appropriate packed or capillary columns as specified by the particular method?	
		Is there a policy in place concerning carryover prevention in the event a high level sample precedes a relatively low-level sample?	
		Is the initial calibration conducted at five or three concentrations as appropriate for the method?	

LABORATORY AUDIT CHECKLIST

4.0 Mass Spectrometry (continued)

Semi-Volatile GC/MS Area

Y	N	Item	Comment
		Is quantitation correct for the method? (based upon the average RF of a calibration curve or the RF from a continuing calibration standard)	
		Are calibrations and tuning performed according the required frequency specified in the methods?	
		Is appropriate corrective action taken if a calibration is out of criteria?	
		Is appropriate corrective action taken if a continuing calibration is out of criteria?	
		Is a method blank run with every batch of samples?	
		Is there specified acceptance limits for the method blank?	
		Is there a policy stating that the laboratory does not "blank correct" sample data?	
		Is the appropriate corrective action taken if a surrogate is outside of acceptance limits?	
		Is the appropriate corrective action taken if internal standard area is outside of acceptance limits?	
		Are library searches examined by the mass spectrometry expert before results are reported and is there documentation of this review?	
		Is a run log maintained for all instrumentation and is it documented controlled?	
		Is raw data stored in a secure manner under appropriate conditions to prevent loss of data?	
		Are the reported results within the calibration range of the highest and lowest standard?	
		Are appropriate dilutions performed?	
		Do gas lines contain molecular sieves or other filters to remove contaminants in gas supply?	

LABORATORY AUDIT CHECKLIST

5.0 Metals Preparation

Check off which of the following items are utilized in this area and complete the associated forms.			
		Refrigerators, freezers, ovens,	Complete Form A
		Logbooks	Complete Form B
		Laboratory Balances	Complete Form C
		Reagents and Standards	Complete Form D
		Safety Equipment	Complete Form E

Y	N	Item	Comment
		Is the storage area for acids properly ventilated?	
		Do the laboratory personnel indicate a familiarity with the requirements set forth in the laboratory QA program?	
Are the following QC samples processed with each analytical batch?			
		Method Blank	
		QC Check Sample	
		Matrix Spike	
		Matrix Spike Duplicate or Matrix Duplicate	
		Are SOPs available detailing the procedures used in this area?	Obtain a listing of all procedures performed in the area. Compare the list with available SOPs and estimate a percentage complete below. Perent Compliance _____
		Is there a SOP for glassware washing and is it posted in the washing area?	
		Are high purity trace metal grade acids utilized for preservation and digestion of samples for metals?	
		Is glassware for metals analysis acid washed?	
		If automatic dispensers are utilized for acids, are they properly maintained?	
		Does the lab ensure that powder free gloves are used to avoid possible zinc contamination of samples?	
		Are operating manuals available to the analysts for all major equipment?	

5.0 Metals Preparation (continued)

Y	N	Item	Comment
		Is there a record of instrument servicing maintained in a logbook and is it document controlled?	
		Is a digestion log maintained for all samples and is it document controlled?	
		Is an acid digestion performed for drinking water samples requiring total metals?	
		Are samples for dissolved metals digested after filtration?	EPA required, except for DW where turbidity is below a certain level.

LABORATORY AUDIT CHECKLIST

6.0 Metals Analysis

Check off which of the following items are utilized in this area and complete the associated forms.			
		Refrigerators., freezers, ovens,	Complete Form A
		Logbooks	Complete Form B
		Laboratory Balances	Complete Form C
		Reagents and Standards	Complete Form D
		Safety Equipment	Complete Form E

Y	N	Item	Comment
		Do the laboratory personnel indicate a familiarity with the requirements set forth in the laboratory QA program?	
		Are SOPs available detailing the procedures used in this area?	Obtain a listing of all procedures performed in the area. Compare the list with available SOPs and estimate a percentage complete below. Percent Compliance _____
		Are gas lines checked for leaks each time a tank is changed?	
		Are vents above AA units functioning properly (200 ft3/min.)?	
		Are SOPs available detailing the procedures used in this area?	
		Are operating manuals available to the analysis for all major equipment?	
		Is there a record of instrument servicing maintained in a logbook and is it document controlled?	
		Is there a policy in place concerning carryover prevention in the event a high level sample precedes a relatively low-level sample?	
		Are calibrations performed according the required frequency specified in the methods?	
		Is appropriate corrective action taken if a calibration is out of criteria?	
		Are continuing calibrations run at an adequate frequency?	

LABORATORY AUDIT CHECKLIST

6.0 Metals Analysis (continued)

Y	N	Item	Comment
		Is appropriate corrective action taken if a continuing calibration is out of criteria?	
		Is a method blank run with every batch of samples?	
		Is there specified acceptance limits for the method blank?	
		Is there a policy stating that the laboratory does not "blank correct" sample data?	
		Is a run log maintained for all instrumentation and is it documented controlled?	
		Are post-digestion spikes performed for graphic furnace analyses?	
		Is there a corrective action when PDS fail? Is it to flag results or perform standard additions?	
		Are ICP interference standards run at the required frequency specified in the method?	
		Are serial dilutions performed?	
		Are check standards run at a frequency of 10% for graphite furnace to monitor when the furnace should be changed?	
		For mercury analysis, is the mercury vapor safely removed from the lab atmosphere?	
		Are all reported results within the calibration range of the highest and lowest standard?	

LABORATORY AUDIT CHECKLIST

7.0 Wet Chemistry

Check off which of the following items are utilized in this area and complete the associated forms.			
		Refrigerators., freezers, ovens,	Complete Form A
		Logbooks	Complete Form B
		Laboratory Balances	Complete Form C
		Reagents and Standards	Complete Form D
		Safety Equipment	Complete Form E

Y	N	Item	Comment
		Are SOPs available detailing the procedures used in this area?	Obtain a listing of all procedures performed in the area. Compare the list with available SOPs and estimate a percentage complete below. Percent Compliance _____
Are the following QC samples processed with each analytical batch?			
		Method Blank	
		QC Check Sample	
		Matrix Spike	
		Matrix Spike Duplicate or Matrix Duplicate	
		If the lab utilizes dessicators, do they appear to be properly maintained?	
		Is there a SOP for glassware washing and is it posted in the washing area?	
		Are operating manuals available to the analysts for all major equipment?	
		Is there a record of instrument servicing maintained in a logbook and is it document controlled?	
		Is there a policy in place concerning carryover prevention in the event a high level sample precedes a relatively low-level sample?	
		Are calibrations performed according the required frequency specified in the methods?	
		Is appropriate corrective action taken if a calibration is out of criteria?	

LABORATORY AUDIT CHECKLIST

7.0 Wet Chemistry (continued)

Y	N	Item	Comment
		Is a method blank run with every batch of samples?	
		Is there specified acceptance limits for the method banks?	
		Is there a policy stating that the laboratory does not "blank correct" sample data?	
		Is a dedicated analysis log maintained for all parameters determined and are they document controlled?	
		Is the pH meter checked daily, or before use, by setting the meter to pH 7.0, then measuring pH at 4 and 10?	
		Is the conductivity meter calibrated daily against a 0.001MKCl solution and the data recorded in a logbook?	
		Is the turbidometer calibrated daily, or before use, with 40 NTU formazine standard and the data recorded in a logbook?	
		Is the dissolved oxygen meter calibrated weekly against the Winkler method and data recorded in a logbook?	
		Are all reported results within the calibration range of the highest and lowest standard?	

LABORATORY AUDIT CHECKLIST

8.0 Report Generation

Check off which of the following items are utilized in this area and complete the associated forms.			
		Logbooks	Complete Form B
		Safety Equipment	Complete Form E

Y	N	Item	Comment
		Do the laboratory personnel indicate a familiarity with the requirements set forth in the laboratory QA program?	
		Are SOPs available detailing the procedures used in this area?	Obtain a listing of all procedures performed in the area. Compare the list with available SOPs and estimate a percentage complete below. Percent Compliance _____
		Is there a document describing in detail the contents and order of deliverables provided with various types of reporting levels or packages?	
		Do all reports contain a cover letter and case narrative?	
		Is chain of custody provided with all reports?	

LABORATORY AUDIT CHECKLIST

9.0 Computer Systems

Check off which of the following items are utilized in this area and complete the associated forms.

		Logbooks	Complete Form B
		Safety Equipment	Complete Form E
Y	N	Item	Comment
		Do the laboratory personnel indicate a familiarity with the requirements set forth in the laboratory QA program?	
		Are SOPs available detailing the procedures used in this area?	Obtain a listing of all procedures performed in the area. Compare the list with available SOPs and estimate a percentage complete below. Percent Compliance _____
		Are computer programs validated before use?	
		Are operating manuals available to the computer specialists for all computer equipment?	
		Is there a record of computer servicing maintained in a logbook and is it document controlled?	
		Is the computer system backed up at a regular frequency and if so, what is the frequency? Backup copy maintained off Site?	
		Is the system protected from unauthorized access utilizing a password system?	
		Are backup power supplied utilized to prevent loss of information in the event of a power failure?	
		Is the system protected from a lightning strike?	
		If an in-house information management system is utilized, are instructions available to the users?	
		Is there a formal training program for new users?	
		Is there any mechanism to prevent computer clocks from being altered to reflect incorrect dates and times?	
		If such alteration would occur, is there a mechanism to detect it in a very timely fashion?	
		Are computer specialists at the facility familiar with the EPA computer requirements set forth in the GALP manual (good automated lab practices)?	

LABORATORY AUDIT CHECKLIST

Form A - Laboratory Temperature Monitoring Checklist

Identify the Laboratory Area under Review:			
Identify the number of units in this laboratory area:			Refrigerators: Freezers: Ovens:
Y	N	Item	Comment
		Do the laboratory personnel indicate a familiarity with the requirements set forth in the laboratory SOP for temperature monitoring?	
		Are temperatures of all lab temperature units such as refrigerators, ovens, freezers and incubators monitored daily and documented in a logbook?	
		Are all units individually identified with a unique identification number on the outside of the unit?	
		Have thermometers been calibrated against NIST within the last year and documented?	
		Are temperature excursion noted and appropriate action taken?	
		Does all laboratory equipment contain thermometers, which are uniquely identified?	
		Are all thermometers immersed in appropriate media?	
		Is the laboratory aware of whether the temperature has been checked routinely?	

LABORATORY AUDIT CHECKLIST

Form B – Laboratory Notebook Checklist

Identify the Laboratory Area under Review:			
Identify the Number of Notebooks in this Area:			
Y	N	Item	Comment
		Do the logbooks comply with GLP standards for logbooks?	
		Are the contents of the notebook clearly identified?	
		Are lab notebooks maintained in black ink?	
		Are notebooks permanently bound and contain preprinted and consecutively numbered pages?	
		Are lab notebooks neat, orderly and legible?	
		Are entries noting anomalies routinely recorded?	
		Are mistakes in notebooks crossed out with one line signed and dated?	
		Do all notebooks have a unique document control number?	
		Is there evidence of secondary review of the notebook?	
		Are unused portions of pages struck out with a line or Z'd out?	

LABORATORY AUDIT CHECKLIST

Form C - Laboratory Balance Checklist

Identify the Laboratory Area under Review:			
Identify the Number of Balances in this Area:			
Y	N	Item	Comment
		Are all balances being calibrated at the correct frequency?	
		Have the calibrations been conducted at the correct level?	
		Are the acceptance limits presented in the logbook?	
		Are balances and S weights maintained in a clean environment?	
		Does each balance have a unique identification number?	
		Has each balance been serviced in the past year and is a sticker affixed to the balance?	

LABORATORY AUDIT CHECKLIST

Form D – Laboratory Reagents and Standards Checklist

Identify the Laboratory Area under Review:			
Y	N	Item	Comment
		Is there a SOP describing how reagents and standards are prepared and stored?	
		Is there a mechanism for verifying a prepared standard or purchased commercial standard prior to its use in the lab?	
		Is a standards notebook maintained for all areas, which describes the preparation and standardization of standard solutions and is it document controlled?	
		Are stock standards traceable to their source?	
		Are standards and reagents properly labeled with received date, concentration, date prepared, preparer's initials and expiration date?	
		Are standards stored separately from samples or sample extracts?	
		Are all sources of reagent water checked daily?	
		Does water meet the conductance requirements outlined in the SOP and is it documented?	
		Are the water quality records document controlled?	
		Are reagent grade or higher-grade chemicals used to prepare standards?	
		Is there evidence that all reagents and standards are within their expiration dates?	
		Are chemicals being used in analyses tested to ensure they contain no contaminants, which may interfere with the analyses, prior to use?	

LABORATORY AUDIT CHECKLIST

Form E – Laboratory Safety Checklist

Identify the Laboratory Area under Review:			
Y	N	Item	Comment
		Is there sufficient safety equipment in this area such as fume hoods, shower, eyewash, first aid kit, etc.?	
		Is there an emergency spill kit available in the vacinity?	
		Are lab fume hoods checked monthly for proper airflow and is it documented?	
		Is there a cutoff level for determining airflow (100fpm)?	
		Is the interior of fume hoods maintained in a clutter free manner?	
		Are safety glasses worn in the laboratory areas?	
		Are appropriate gloves worn suitable for the reagents used in the laboratory?	
		Are the storage areas and benchtops maintained in an orderly manner such that safety hazards are avoided?	
		Have fire extinguishers, safety showers, eyewashes been checked for proper operation?	
		Are self-contained breathing apparatus available for use in emergency situations?	
		Is all standards preparation performed in a fume hood?	
		Are material safety data sheets (MSDS) accessible and up to date?	
		Are all instruments properly vented?	
		Have all personnel participated in formal safety training?	

APPENDIX D
DATA MANAGEMENT PLAN

GM POWERTRAIN BEDFORD PLANT
105 GM DRIVE
BEDFORD, INDIANA

EPA ID# IND006036099

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1.0 INTRODUCTION

This Data Management Plan (DMP) is submitted as an appendix to and forms part of the Resource Conversation and Recovery Act (RCRA) Facility Investigation (RFI) Work Plan prepared by General Motors Corporation (GM) for the GM Powertrain – Bedford Plant (Bedford Facility).

This DMP identifies procedures to be employed for managing information, reports, and correspondence (documents) associated with the RFI to be conducted at the Facility. These documents must be readily accessible and the integrity and accuracy of these documents must be maintained. This will be achieved by restricting access to the materials and implementing data management procedures.

This DMP is comprised of two separate tasks which are:

i) data management; and

ii) document control.

The data management task consists of procedures used to collect, handle, and safeguard all data generated by field and laboratory programs. The task of document control involves implementing procedures to physically track all documents associated with the RFI which GM possesses. These two tasks will be expanded upon in the following sections.

2.0 DATA MANAGEMENT

The data management task of this DMP is to implement procedures relating to collection, recording, retrieval, presentation, and security of all field and laboratory data generated. The data to be managed includes but it is not limited to:

i) field data;

ii) laboratory analytical data; and

iii) quality assurance/quality control data.

These three categories of data and security procedures will be reviewed as discussed in the following sections.

2.1 DATA COLLECTION

2.1.1 FIELD LOGBOOKS

Data generated in the field will be recorded in the field logbook. Photographs of the RFI work also form part of the field database. The field logbook is the primary means of recording field-related information. The field logbooks will contain data such as the following:

i) general field observations;

ii) field measurements and observations;

iii) sample location and corresponding sample number;

iv) relevant comments pertaining to the samples collected;

v) weather conditions;

vi) a listing of all personnel involved in Facility-related activities;

vii) an accurate log of all telephone conversations and Facility meetings;

viii) field decisions made and pertinent information associated with the decisions; and

ix) relevant field forms completed documenting collected field data.

The field logbooks will be numbered consecutively and maintained in accordance with the project Quality Assurance Project Plan (QAPP) (see Appendix C).

Field forms may be used during implementation of various field activities (e.g., boring logs, groundwater sample record forms, etc.). The use of and an example of each field form is included in each appropriate Standard Operating Procedure (SOP). When data are recorded on field forms, appropriate documentation of the use of such forms will be recorded in the field log books as described above.

2.1.2 STILL PHOTOGRAPHS

Historic still photographs provide a means of visually recording Facility conditions and operations.

To ensure quick and accurate retrieval, all photographs taken during RFI work will be documented, catalogued, and stored. Documentation shall consist of the following:

i) identification of Facility and project;
ii) identification of the area and/or activity photographed;
iii) orientation and direction of photograph;
iv) date and time of photograph;
v) photographer's name;
vi) weather conditions; and
vii) a unique number identifying the photograph.

Cataloging of photographs shall be done in a manner that ensures ease of accessibility. Storage of the photographs shall be in a secure location.

2.1.3 ANALYTICAL DATA

Analytical data will be compiled on three and one-half inch floppy discs and/or compact discs (CD) in addition to the original laboratory reports. In all cases where Facility data are stored on diskettes, a backup copy of each computer diskette will be maintained. Each computer diskette will be consecutively numbered, identified as a primary or

backup diskette, and identified as to the contents. Backup diskettes will be stored separately from primary diskettes.

2.2 DATA STORAGE AND RETRIEVAL

The data collected and/or generated as part of the RFI will be stored in a secure manner in a location where all project materials can easily be retrieved.

Field logbooks, field forms, analytical reports, reference materials, correspondence, reports, and other documents will be filed in chronological order in the project file. Computer disks will be stored in a secure area and will back up any computer information stored on the computer systems. The computer-stored information will be filed under the project name and identification number.

2.3 DATA PRESENTATION

The data collected and/or generated as part of the RFI will be presented in tabular and/or graphical formats.

Analytical lab reports will be presented as an appendix to the RFI Report. Summary tables of the analytical data will be developed to present the data in a useable format. Analytical data will be manipulated using a database management system, which uses EQuIS software.

Graphical data presentation in the form of figures, plans, or charts/graphs will also be included in the RFI Report. Information that will be presented in this format includes: sample areas, sampling locations, contaminant distribution, stratigraphic logs, geologic cross-sections, and any other information where a graphical presentation would be appropriate.

2.4 DATA SECURITY

The integrity of the data generated during the RFI will be maintained by restricting access to other than read-only documents. Only personnel actively involved in the project will be permitted access to the original data. The data will be accessed in

accordance with CRA's, Exponent's, and ENVIRON's standard procedures for protecting the integrity of the data.

3.0 DOCUMENT CONTROL

Documents used for and generated during the RFI will be stored and maintained in a unique project file. Access to the documents will be restricted to personnel actively engaged in the project and procedures will be implemented to track the documents.

GM will maintain the document file.

The documents to be covered by the document control procedures can be categorized as follows:

i) background information files;

ii) primary data documents; and

iii) project documents generated during the course of the project.

These three categories will be further discussed in the following sections.

3.1 BACKGROUND INFORMATION FILES

The background information for the RFI consists of the following:

i) reports of previous Facility sampling programs;

ii) copies of Facility files from the Facility;

ii) copies of Facility files from the United States Environmental Protection Agency (U.S. EPA)/Indiana Department of Environmental Management (IDEM), United States Department of the Interior – Fish and Wildlife Services (U.S. DOI); and

iii) miscellaneous correspondence.

These documents will be included in the document control system.

3.2 PRIMARY DATA DOCUMENTS

Primary data documents for the RFI will consist of the following:

i) field logbooks/field forms;
ii) analytical reports;
iii) chain-of-custody forms;
iv) U.S. EPA/IDEM/U.S. DOI correspondence;
v) personnel medical records;
vi) logs of meetings and telephone conversations;
vii) quality assurance/quality control data;
viii) inventory of samples;
ix) Facility plans and data diskettes; and
x) survey notes.

These documents will be included in the document control system.

3.3 PROJECT DOCUMENTS

Project documents include reports generated during the RFI and forwarded to the U.S. EPA. These documents will be included in the document control system.

HEALTH AND SAFETY PLAN

GM POWERTRAIN – BEDFORD PLANT
105 GM DRIVE
BEDFORD, INDIANA

EPA ID# IND 006036099

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APPENDIX C	HEAT STRESS AND COLD STRESS PROCEDURES

1.0 GENERAL

An investigation program will be implemented at the General Motors Powertrain (GM) Bedford Plant (Facility) located in Bedford, Indiana. The activities outlined in the Scope of Work (SOW) will involve drilling and sampling within and outside the limits of the Facility. During this program, personnel may come in contact with soil, sediment, groundwater, surface water, and other materials/debris that may contain hazardous materials. This Health & Safety Plan (HASP) has been developed to minimize the potential for exposures to field personnel and serves as the general HASP for the Bedford RCRA Facility Investigation (RFI). This HASP is provided as a starting point for the use of any personnel completing RFI activities potentially encountering contaminated materials. All contractors and subcontractors should develop HASPs which are specific to their SOW and are expected to take responsibility for the health and safety of their employees. A contractor or subcontractor may agree to follow the provisions contained in this HASP, but must still provide a written SOW that details their activities and health and safety procedures that will be implemented as part of their activities

This HASP is designed to ensure the following:

i) that field personnel are not adversely exposed to the constituents of concern as well as the physical and biological hazards present;

ii) that public welfare or the environment are not adversely impacted by migration of contaminated materials due to work activities; and

iii) that operations, procedures, and equipment will meet the requirements of 29 CFR 1910.120, Hazardous Waste Operations and Emergency Response, and the applicable subparts of 29 CFR 1926 and 29 CFR 1910.

For the purpose of this HASP, all investigative activities carried out at the Facility involving contact with potentially contaminated materials will be considered contaminated operations requiring personal protective equipment (PPE).

Investigation operations at the Bedford Facility will be conducted in accordance with the provisions of the HASP. Cost and/or scheduling considerations will not be considered as justification for modifying this plan.

2.0 FACILITY CHARACTERIZATION AND HAZARD ANALYSIS

The Bedford Facility is located at 105 GM Drive, in Bedford, Indiana as depicted in Figure 1. The Facility is located in a commercial and industrial setting. A Facility plan is included as Figure 2 of this HASP.

The Bedford Facility is an active facility and has been operating as an aluminum foundry since 1942. Current products include transmission cases, engine blocks, and pistons.

A summary of the constituents of concern that have been identified and their related health effects are presented in Table 1 of this HASP.

The activities to be performed as part of the SOW may include:

- oversight of soil boring advancement;
- oversight of monitoring well installation; and
- sampling activities.

Risks associated with these activities will be minimized by implementing engineering controls, safe work practices, and the proper use of personal protective equipment (PPE). Table 2 summarizes the potential hazards associated with activities at the Facility.

3.0 BASIS

The Occupational Safety and Health Administration (OSHA) Standards and Regulations contained in Title 29, CFR, Parts 1910 and 1926 (29 CFR 1910 and 1926) including the amended sections in 29 CFR 1910.120 and current Recommended Exposure Limits (RELs) as provided by the National Institute for Occupational Safety and Health (NIOSH) provide the basis for this HASP. Some of the specifications within this section are in addition to OSHA regulations and reflect the positions of the United States Environmental Protection Agency (USEPA), the American Conference of Governmental Industrial Hygienists (ACGIH) and the United States Coast Guard (USCG) regarding procedures required to ensure safe operations at potential hazardous waste sites.

The safety and health of the public and field personnel and the protection of the environment will take precedence over cost and schedule considerations for all project work.

4.0 RESPONSIBILITIES AND ADMINISTRATION

The following individuals are designated to carry out stated job responsibilities for the onsite engineer, CRA, related to this project.

-Senior Project Engineer:	James McGuigan
-Project Manager:	William Steinmann
-Project Industrial Hygienist:	Matthew Lazaric
-Project Geologist	TBD

A field individual will be designated as the Health and Safety Officer (HSO). The HSO will supervise the implementation of the HASP and will be responsible for all decisions regarding operations and work stoppages due to health and safety considerations.

The responsibilities of the HSO are as follows:

i) be responsible for controlling and maintaining access to the work area;

ii) be responsible for implementation of the HASP at the initiation of field work;

iii) conduct the pre-entry safety briefing for all field personnel with regard to the HASP and other safety requirements to be observed during field sampling, including:

   a) potential hazards,
   b) personal hygiene principles,
   c) PPE,
   d) respiratory protection equipment usage, and
   e) emergency response procedures.

iv) review and modify the HASP as more information becomes available concerning the hazardous materials involved;

v) supervision and enforcement of safety equipment usage;

vi) supervision and inspection of equipment cleaning;

vii) personnel training in safety equipment usage and emergency procedures;

viii) monitoring of the health and safety program under direction of an industrial hygienist;

ix) suspend work activity if unsafe working conditions develop;

x) inform employees, nearby workers, and visitors of the nature of chemical exposure risk as required by the "Right-to-Know" Law;

xi) recommend a medical examination when required for an employee;

xii) coordination of the Emergency Response Plan (Section 15.0);

xiii) assure that safety equipment is provided, maintained and accessible to field personnel;

xiv) maintain a log with a sign in/out sheet for personnel performing activities and visitors entering the work areas;

xv) assure that employees comply with the "buddy system" while working at the Facility;

xvi) investigate all accidents, injuries, illnesses, spills, fires, incidents, and near misses; and

xvii) Ensure all subcontractors have a Health and Safety Plan.

5.0 MEDICAL SURVEILLANCE

In accordance with requirements detailed in 29 CFR 1910.120(f), all personnel who may be exposed to potentially hazardous materials will have received, within one year prior to starting field activities, medical surveillance by a licensed physician or physician's group.

Medical records for CRA field personnel will be maintained at the Niagara Falls, New York office. The medical records will detail the tests that were taken and will include a copy of the consulting physician's statement regarding the tests and the employee's suitability for work.

All personnel involved in field work will have all necessary medical examinations prior to commencing work which requires respiratory protection or exposure to hazardous materials. Personnel not obtaining medical certification will not perform work within contaminated areas.

Interim medical surveillance will be completed if an individual exhibits poor health or high stress responses due to field work or when accidental exposure to elevated concentrations of contaminants occurs.

6.0 TRAINING

All field personnel shall complete training sessions in accordance with 29 CFR 1910.120(e). This training shall consist of a minimum of 40 hours of instruction and three days of actual field experience under the direct supervision of a trained, experienced supervisor. Documentation will be maintained in the site trailer stating that all field personnel have complied with this regulation.

Prior to commencing work activities, a pre-entry safety briefing will be conducted. Topics covered during the pre-entry safety briefing will include:

i) health and safety hazards;

ii) level of PPE required;

iii) safe use of equipment;

iv) decontamination procedures; and

v) emergency response procedures.

CRA personnel who attend this briefing will sign the HASP Acknowledgment Form presented as Appendix A. Any visitors to the Facility entering the exclusion zone will be required to undergo the same training program discussed above and will be required to sign a Visitor's Signoff Sheet (Appendix A) certifying they have completed the appropriate training and have read this HASP.

All personnel working under the RFI at the Bedford Facility shall attend regular safety ("tailgate") meetings. These meetings will be conducted by the HSO, and will cover specific health and safety issues, field activities, changes in field conditions, and a review of topics covered in the pre-entry briefing. Topics discussed in the safety meetings will be documented along with the signatures of personnel who attend.

7.0 WORK AREAS

As required, specific work areas (defined below) will be delineated by temporary fencing or a flagged line.

a) Exclusion Zone (EZ) - This zone will include all areas where potentially contaminated soil or materials are to be handled and all areas where contaminated equipment or personnel travel.

b) Contaminant Reduction Zone (CRZ) - This zone will occur at the interface of the EZ and Support Zone (SZ) and will provide access for the transfer of construction materials and field equipment to the EZ, the decontamination of vehicles prior to leaving the EZ, the decontamination of personnel and clothing prior to entering the SZ, and for the physical segregation of the SZ and EZ.

c) Support Zone (SZ) - This area is the portion of the work area defined as the area outside the zone of significant air and soil contamination. The SZ will be clearly delineated and procedures implemented to prevent active or passive migration of contamination from the work area.

8.0 PERSONAL PROTECTIVE EQUIPMENT

Engineering controls and work practices designed to reduce and maintain employee exposure at or below the Permissable Exposure Limits (PELs) for the constituents of concern (Table 1) will be implemented. Whenever engineering controls and work practices are not feasible, a reasonable combination of engineering controls, work practices, and PPE shall be used to reduce and maintain employee exposure at or below the PELs for the contaminants of concern.

All field personnel shall be equipped with PPE appropriate for the nature of work being completed. All safety equipment and protective clothing shall be kept clean, well-maintained, and their integrity intact.

Safety equipment and apparel as required will be Level D, Modified Level D and Level C PPE (as determined by the action levels set forth in Section 9.0) within the Exclusion Zone. Any decision regarding the adjustment of PPE levels will be made by the Project Manager, in conjunction with the Project Industrial Hygienist (IH).

Level D PPE

Type	*Properties*	*Item*
Foot protection	Steel-toe/reinforced shank	Boots
Head protection	Meets ANSI Z89.1 standard	Hard hat
Hand protection	Puncture/tear resistant	Leather/cotton gloves
Eye protection	Meets ANSI Z87.1 standard	Glasses/goggles with side shields

Modified Level D PPE

Type	*Properties*	*Item*
Foot protection	Steel-toe/reinforced shank	Boots
Foot protection	Chemical resistant (latex)	Overboots
Head protection	Meets ANSI Z89.1 standard	Hard hat
Hand protection	Chemical resistant (nitrile)	Inner gloves
Hand protection	Chemical resistant, puncture/ tear resistant (nitrile)	Outer gloves
Eye protection	Meets ANSI Z87.1 standard	Glasses/goggles with side shields
Body protection	Chemical resistant (tyvek)	Coverall

Level C PPE

Type	*Properties*	*Item*
Foot protection	Steel-toe/reinforced shank	Boots
Foot protection	Chemical resistant (latex)	Overboots
Head protection	Meets ANSI Z89.1 standard	Hard hat
Hand protection	Chemical resistant (nitrile)	Inner gloves
Hand protection	Chemical resistant, puncture/ tear resistant (nitrile)	Outer gloves
Eye protection	Meets ANSI Z87.1 standard	Glasses/goggles with side shields
Body protection	Chemical resistant (tyvek)	Coverall
Respiratory protection	NIOSH approved	Full-face/half-face air-purifying respirator with organic vapor/ acid gas/HEPA filter

Additional PPE guidelines to be implemented include:

i) prescription eyeglasses in use at the Facility will be safety glasses with side shields;

ii) protective gloves (leather palm) will be worn over nitrile gloves by field personnel involved in drilling activities;

iii) during periods of respirator usage, respirator cartridges and filters will be changed daily, or upon breakthrough, whichever occurs first;

iv) field personnel who have not passed a respirator fit test will not be permitted to enter or work in the EZ;

v) personnel required to wear a respirator will not be permitted to have beards, long sideburns, or mustaches that interfere with the proper fit of the respirator;

vi) all used PPE will be decontaminated or discarded at the end of each work day; and

vii) duct tape will be used to ensure that disposable coveralls and gloves are tightly secured when personnel are working within the EZ.

9.0 RESPIRATOR PROGRAM

Prior to arriving at the Facility, all field personnel will have received training in the use of, and have been fit tested for a full-facepiece respirator. The written respiratory program that complies with 29 CFR 1910.134 is included as Appendix B to this HASP (copies of the certifications will be kept on file at the field trailer).

During intrusive activities (i.e. excavating), a photo-ionization detector (PID) will be used to monitor for organic vapors and some inorganic gases and a particulate monitor will be used to monitor particulate concentrations present in the breathing space. Background concentrations will be established prior to commencing work activities at each work location.

Sustained (greater than 5 minutes) air monitoring action levels to determine the level of respiratory protection necessary during field activities will be:

Sustained Photoionization Organic Vapor Reading Above Background	***Protection Level***
0 – 10 ppm	Level D, Modified Level D
10 – 125 ppm	full-facepiece air purifying respirator (Level C)
>125 ppm	shut down activities

Sustained Particulate Reading Above Background	***Protection Level***
0 – 0.5 mg/m3	Level D, Modified Level D
0.5 – 2.5 mg/m3	full-facepiece air purifying respirator (Level C)
>2.5 mg/m3	shut down activities

Work will be stopped and the work area will be allowed to vent if monitoring indicates that organic vapors are present at concentrations which present Immediate Danger to Life and Health (IDLH) conditions, or in excess of the protection factor afforded by the air purifying respirator (whichever is lower). The action level to shut down activities has been decreased to a reasonable level for the work being conducted.

Air monitoring should continue, at a safe distance, if operations are stopped due to action level exceedences, to determine if a threat to the surrounding community exists.

10.0 JUSTIFICATION

The activities associated with the SOW are not expected to cause field personnel to be exposed to the constituents of concern in concentrations exceeding their respective PELs or RELs. The action levels have been calculated low enough so that the accuracy and sensitivity of the air monitoring equipment would not cause the field personnel to be inadvertently overexposed to the constituents of concern, even in a worst case scenario.

These action levels assume that all NIOSH criteria for using an air purifying respirator (APR) have been met. An APR can typically be worn in concentrations of up to 10 times the PEL for a given contaminant. Because of differences in sensitivities with direct reading instruments, a 50 percent safety factor is included when determining action levels. Therefore, the calculation to determine when a respirator should no longer be used is presented below (for a constituents of concern with PELs of 25 ppm and 0.5 mg/m3):

25 ppm (PEL) x 10 (protection factor) x 0.5 (50% safety factor) = 125 ppm

0.5 mg/m3 (PEL) x 10 (protection factor) x 0.5 (50% safety factor) = 2.5 ppm

The primary routes of exposure of contaminants to individuals performing field remediation tasks include direct contact, ingestion, and inhalation. The risk of exposure due to direct contact and ingestion will be minimized through the proper use of PPE as described in Section 8.0 and by exercising ordinary caution during sampling activities. In order to minimize exposure by the inhalation pathway, the respirator and air monitoring programs discussed in Sections 9.0 and 12.0 will be undertaken.

11.0 PERSONAL HYGIENE

All personnel performing or supervising work within the EZ shall observe and adhere to the personal hygiene-related provisions of this section.

Personnel found to be disregarding the personal hygiene-related provisions of this HASP will, at the discretion of the HSO, be barred from the Facility.

The following equipment/facilities are available for the personal hygiene of CRA's Facility personnel:

i) suitable disposable outerwear, gloves, respiratory protection and footwear on a daily basis for the use of field personnel;

ii) disposal containers for used disposable outerwear; and

iii) potable water and a suitable sanitation facility.

The following regulations will be enforced for all personnel actively participating in the field sampling program:

i) personnel will wear appropriate PPE when in the EZ;

ii) used disposable outerwear will not be reused if deemed to be unsuitable to provide the necessary protection, and when removed, will be placed inside disposal containers provided for that purpose;

iii) smoking, eating, and drinking will be prohibited within the EZ. These activities will be permitted only within designated areas; and

iv) personnel will thoroughly cleanse their hands, face, neck area, and other exposed areas before smoking, eating, or drinking, and before leaving the CRZ.

12.0 AIR MONITORING

Air quality will be monitored at the initiation of each work activity and periodically thereafter.

The air monitoring program will consist of monitoring with a PID and a particulate monitor. Operation and calibration procedures will be according to manufacturers' instructions. Calibration and maintenance records will be kept in the field log.

Identification of volatile organic vapor or particulate levels in excess of the action levels cited in Section 9.0 shall be reported to the HSO who, in conjunction with the Project IH, will determine when PPE should be upgraded or operations be shut down and restarted.

If work is stopped because action levels have been exceeded, air monitoring will continue from a safe distance to determine if there is a threat to the surrounding community.

13.0 COMMUNICATIONS

Emergency numbers including the police department, fire department, ambulance, hospital, and appropriate regulatory agencies (Table 3) will be prominently posted near the Facility telephone(s).

To ensure familiarity with the hospital route, all personnel covered under this HASP will drive the route to the hospital, prior to the initiation of field activities.

14.0 EMERGENCY AND FIRST AID EQUIPMENT

Safety equipment will be available for use by field personnel and will be located and maintained in the SZ. The safety equipment will include, but is not limited to, the following:

i) a portable emergency eye wash;

ii) one ABC type dry chemical fire extinguishers; and

iii) a first-aid kit for a minimum of 10 personnel.

15.0 EMERGENCY RESPONSE PLAN

Prior to commencing work activities, CRA will ensure that all of field personnel are briefed on emergency procedures. The emergency procedures are intended to provide immediate response to a serious occurrence such as injury, explosion or fire. A list of emergency contact numbers is presented as Table 3 of this HASP.

In the event of injury to field personnel, the HSO is responsible for the following:

i) evacuate all personnel upwind;

ii) exit Facility;

iii) contact the designated hospital and describe the injury;

iv) decontaminate personnel if possible, and administer appropriate first aid. If personnel cannot be decontaminated, alert hospital to possible problems of contamination;

v) transport personnel to the medical facility along a predefined route (Figure 3); and

vi) notify GM plant personnel.

16.0 EQUIPMENT AND PERSONNEL DECONTAMINATION

During the field investigation program, procedures will be implemented to reduce the amount of contact of both personnel and equipment with potentially contaminated materials. These procedures include the following:

i) proper work practices that will lead to minimal direct contact with potentially contaminated material; and

ii) use of disposable equipment and clothing as much as practicable.

All equipment used for the collection of samples for chemical analysis will be cleaned according to the following protocol:

- wash and scrub with low phosphate detergent;
- tap water rinse;
- thorough rinse with deionized distilled water;
- air dry; and
- wrap in aluminum foil for transport.

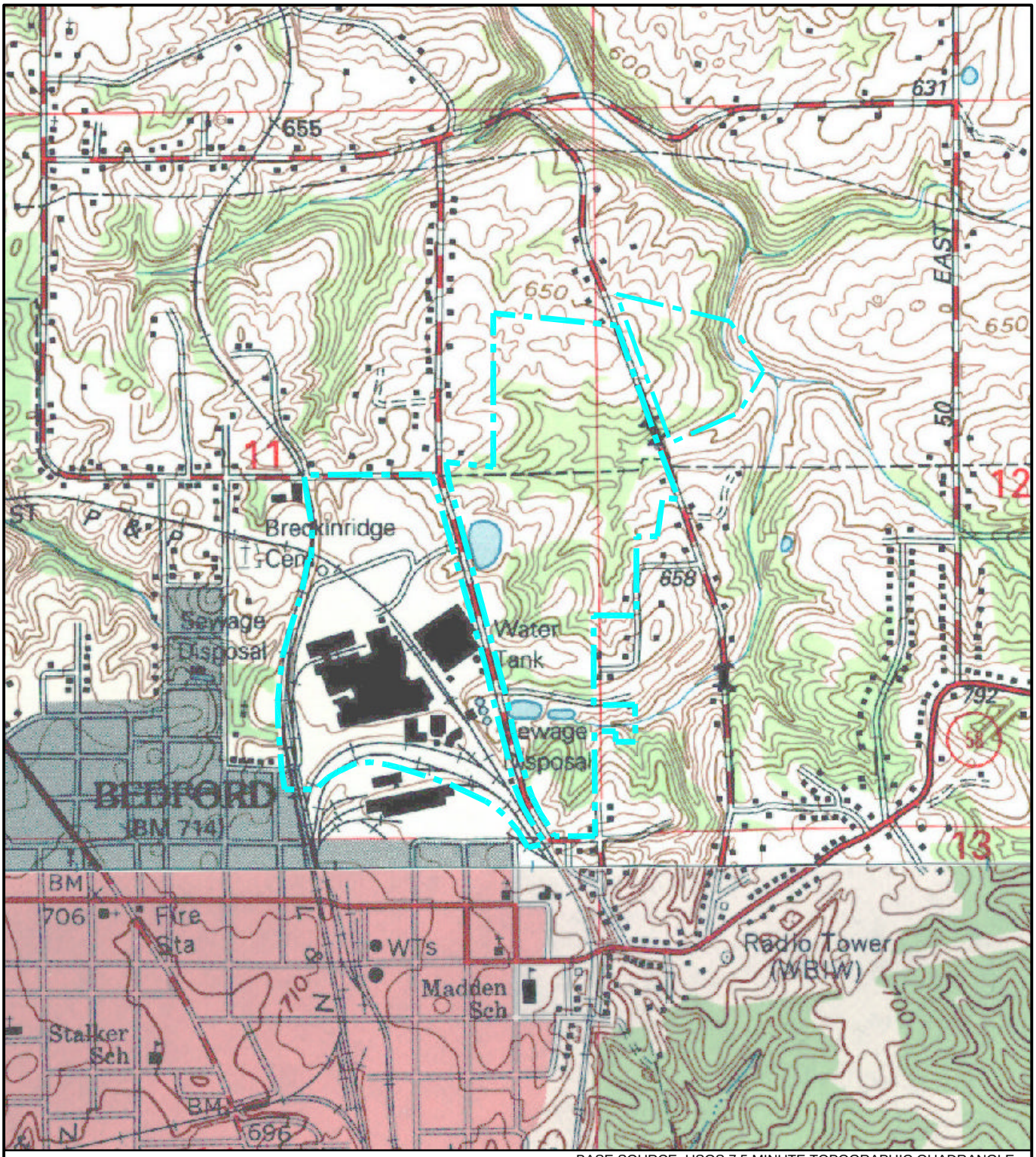
Tap water may be used from any municipal water treatment system. The use of an untreated water supply is not an acceptable substitute.

Personnel will remove their PPE and wash their hands, face, neck area and other exposed areas before entering the lunch and break areas to eat, drink, or smoke.

17.0 CONTAMINATION MIGRATION CONTROL

All vehicles and equipment used within the EZ shall be decontaminated in the CRZ as determined necessary by the HSO prior to leaving the Facility. Decontamination, when required, will consist of the thorough cleaning of those parts of the equipment which come in contact with potentially contaminated material. The HSO will certify that each piece of equipment has been decontaminated prior to removal from the Facility.

Personnel engaged in vehicle decontamination will wear PPE including suitable disposable clothing, respiratory protection, and face shields.

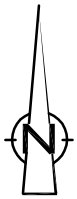


LEGEND:

APPROXIMATE PROPERTY BOUNDARY



BASE SOURCE: USGS 7.5 MINUTE TOPOGRAPHIC QUADRANGLE;
BARTLETTSVILLE, INDIANA 1994
BEDFORD EAST, INDIANA 1978
BEDFORD WEST, INDIANA 1993
OOLITIC, INDIANA 1987





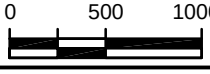


figure 1

FACILITY LOCATION MAP
GM POWERTRAIN BEDFORD FACILITY
Bedford, Indiana

13968-00(006)GN-CO001 JUL 11/2001



N⁰	Revision	Date	Initial

LEGEND

- PROPERTY BOUNDARY
- BEDFORD CITY LIMIT
- FENCE LINE
- RAILROAD TRACKS
- DIRT ROADS
- ROADS / PAVED AREAS
- STREAM
- CONTOUR LINE

SCALE VERIFICATION

THIS BAR MEASURES 1" ON ORIGINAL. ADJUST SCALE ACCORDINGLY.

Approved

DRAWING STATUS

Status	Date	Initial

GM POWERTRAIN BEDFORD PLANT

BEDFORD, INDIANA

FACILITY PLAN

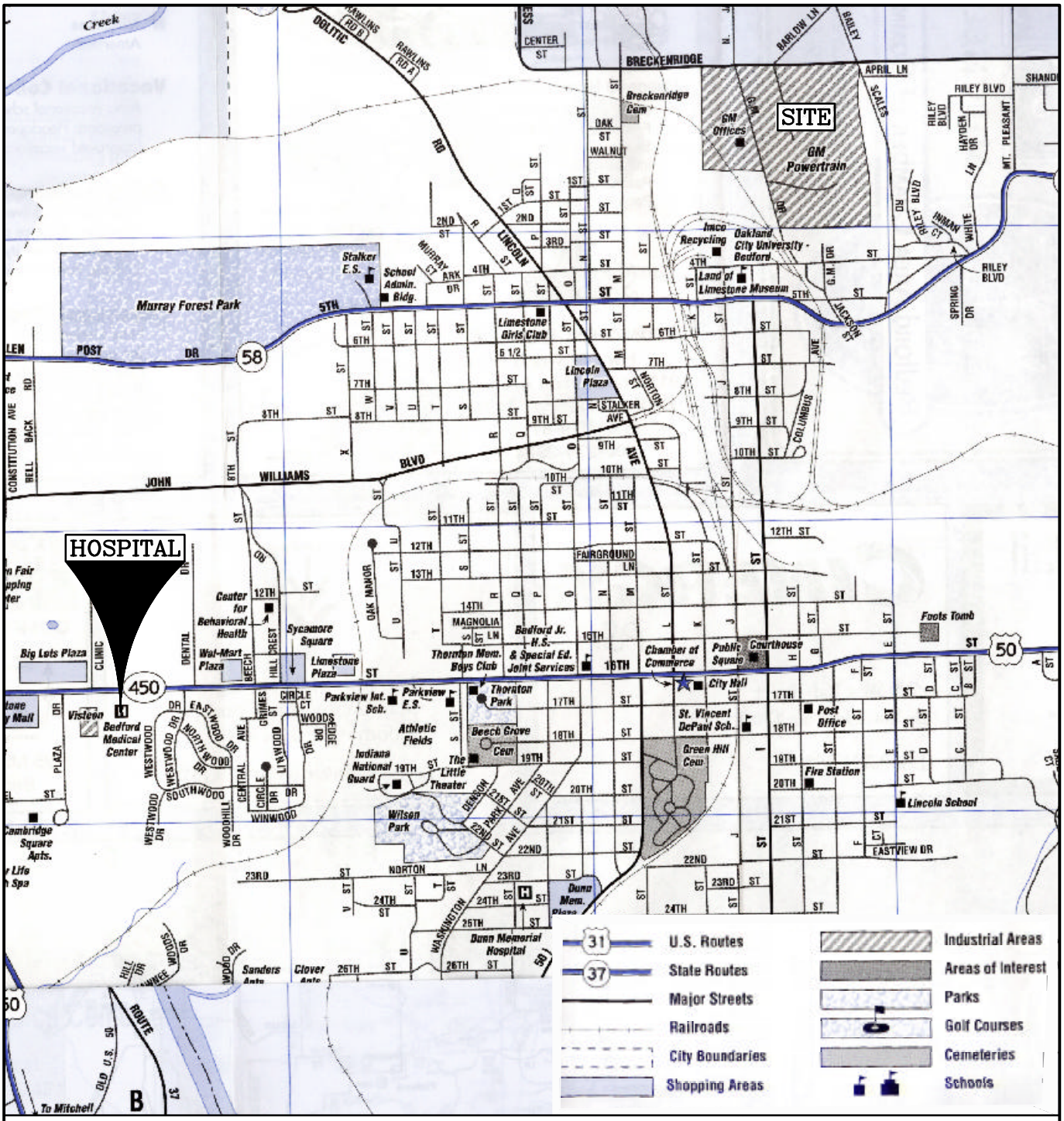
COORDINATE SYSTEM: INDIANA STATE PLANE COORDINATE (NAD 27)

ADAPTED FROM: GENERAL MOTORS POWERTRAIN
PLANT ENGINEERING DEPARTMENT
PLOT PLAN, 12/03/99

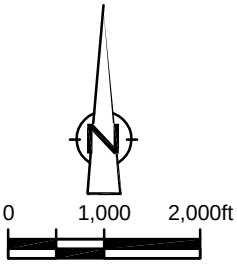
CONESTOGA-ROVERS & ASSOCIATES

Source Reference:

Project Manager:	Reviewed By:	Date:	
JAMES Mc GUIGAN	DEBBIE RUNKLE		
Scale: 1"=200'	Project N⁰: 13968-00	Report N⁰: 006	Drawing N⁰: figure 2



BASE SOURCE: HAMPTON PUBLISING CO.





DIRECTIONS

GO SOUTH ON GM DRIVE (0.6 miles)
TURN RIGHT ONTO IN-58 (0.5 miles)
TURN LEFT ONTO LINCOLN AVE. (0.7 miles)
TURN RIGHT ONTO US-50 (1.3 miles)

figure 3

HOSPITAL ROUTE MAP
GM POWERTRAIN BEDFORD FACILITY
Bedford, Indiana

13968-00(006)GN-CO003 JUL 12/2001

TABLE 1

POTENTIAL CONSTITUENTS OF CONCERN (Currently known)
GENERAL MOTORS POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Contaminant	REL	PEL	IDLH	IP (eV)	Flammability Range	Routes of Exposure	Symptoms of Exposure	Other
Polychlorinated biphenyls	0.001 mg/m^3	0.5 mg/m^3	5 mg/m^3	NL	NA	Inhalation, Ingestion, Contact	Eye irritant; chloracne; liver damage; reproductive effects	Skin Absorptive
Benzene	0.1 ppm	1 ppm	500 ppm	9.24	1.2% - 7.8%	Inhalation, Ingestion, Contact	Eye, skin, nose irritant; headache, nausea, giddiness, fatigue, anorexia, exaustion, depression	Skin Absorptive
Ethylbenzene	100 ppm	100 ppm	800 ppm	8.76	0.8% - 6.7%	Inhalation, Ingestion, Contact	Eye, skin, mucous membrane irritation; headache, dermatitus, narcosis, coma	None
Toluene	100 ppm	200 ppm	500 ppm	8.82	1.1% - 7.1%	Inhalation, Ingestion, Contact	Eye and nose irritant; fatigue, confusion, euphoria, dizziness, headache, dilated pupils, tearing, nervousness, muscle fatigue, insomnia, parasthesia, dermatitus	Skin Absorptive
Xlylene, total	100 ppm	100 ppm	900 ppm	8.44 - 8.56	0.9% - 7.0%	Inhalation, Ingestion, Contact	Eye, skin, throat, and nose irritant; dizziness, excitement, drowsiness, Incoordination, staggering, corneal vacuolization, anorexia, nausea, vomit, abdominal pain, dermatitus	Skin Absorptive
Ammonia	25 ppm	50 ppm	300 ppm	10.18	15% - 28%	Inhalation, Ingestion, Contact	Eye, nose, and throat irritant; breathing difficulty; wheezing; chest pain; pulmonary edema; pink frothy saliva; skin burns; vesiculation; frostbite	None
Fuel Oil	100 mg/m^3	NL	NL	NL	0.7% - 5%	Inhalation, Ingestion, Contact	Eye, nose, and throat irritant; burning sensation in the chest; headache; nausea; weakness; restlessness; incoordination; confusion; drowsiness; vomiting; diarrhea; dermatitis; chemical pneumonia	None
Sodium Hydroxide	2 mg/m^3	2 mg/m^3	10 mg/m^3	NA	NA	Inhalation, Ingestion, Contact	Eye, skin, mucous membrane irritation; pneumonitis; eye, skin burns; temporary loss of hair	None

Notes: NA - Not Applicable
NL - Not Listed
REL - Recommended Exposure Limit (NIOSH)
PEL - Permissible Exposure Limit (OSHA)
IDLH - Immediate Danger to Life and Health (OSHA)
IP - Ionization Potential
ppm - parts per million
mg/m^3 - milligrams per cubic meter
eV - electron volts

TABLE 2

HAZARD ANALYSIS
GENERAL MOTORS POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Field Activities	***Hazards***	***Prevention***
Monitoring Well Installation, and Soil Boring Oversight	Chemical hazards due to inhalation and dermal contact	Proper use of PPE
	Exposure to temperature extremes	Monitor for heat or cold stress
	Physical hazards associated with operation of excavation and drilling equipment	Maintain a safe distance from equipment
		Avoid overhead power lines (20 feet)
		Check for and mark underground utilities
	Physical hazards including steep grades and unstable surfaces while transporting heavy equipment	Use "buddy system" during field activities
	Biological Hazards	Proper PPE, exercising ordinary caution, use of "buddy system"
	High noise level	Use hearing protection
	Slip, trip, fall	Clean mud, snow or grease from shoes and equipment
Surface-Water and / or Stream Sampling	Hazards associated with sampling from a boat	Proper use of life jackets and / or flotation devices
		Sampling from a boat is to be performed from the bow or stern from a seated or stable position training in use of equipment Train personnel in boat and water safety
	Hazards associated with electroshocking	Exercise ordinary caution, use of "buddy system" during field activities
	Slip, trip, and fall	Exercise ordinary caution, use of "buddy system" during field activities
	Exposure to temperature extremes	Monitor for heat or cold stress
	Biological Hazards	Proper PPE, exercising ordinary caution, use of "buddy system"

TABLE 2

HAZARD ANALYSIS
GENERAL MOTORS POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Residential Well Surveying (Door-to-Door)	Slip, trip, and fall	Keep all pathways clear from obstruction
	Biological Hazards	Proper PPE, exercising ordinary caution, use of "buddy system"
	Exposure to temperature extremes	Monitor for heat or cold stress
Stream Surveying / Walking	Slip, trip, and fall	Exercise ordinary caution, use of buddy System during field activities
		In wooded locations watch footing on loose rock and debris
	Biological Hazards	Proper PPE, exercising ordinary caution, use of "buddy system"
	Exposure to temperature extremes	Monitor for heat or cold stress
Sampling Activities	Exposure to temperature extremes	Monitor for heat or cold stress
	Chemical hazards due to inhalation and dermal contact	Proper use of PPE
	Biological Hazards	Proper PPE, exercising ordinary caution, use of "buddy system"

TABLE 3

EMERGENCY CONTACTS
GENERAL MOTORS POWERTRAIN - BEDFORD PLANT
BEDFORD, INDIANA

Agency/Firm	***Emergency Telephone Number***	***Business Telephone Number***
Local Emergency Services		
Fire Department	911	
Police Department	911	
Ambulance	911	
Hospital (Regional Medical Center) 2900 16th Street Bedford, IN 47421		(812) 275-1200
National Poison Center		(800) 942-5969
National Response Center		(800) 424-8802
CRA Industrial Hygienist - Matthew Lazaric		(773) 380-9933
CRA Senior Project Engineer / Advisor - James McGuigan		(773) 380-9933
CRA Project Manager - William Steinmann		(773) 380-9933
On-Site Contacts - Michael Scalzi - Kimberly Dobosenski		(812) 279-7334 (812) 279-7404

Directions to the Hospital: **(REFER TO FIGURE 3)**	Go south on GM Drive (0.6 miles) Turn right onto IN-58 (0.5 miles) Turn left onto Lincoln Ave. (0.7 miles) Turn right onto US-50 (1.3 miles)
Total Distance:	3.1 miles
Travel Time:	7 minutes

APPENDIX A

HEALTH AND SAFETY PLAN
ACKNOWLEDGMENT FORM / VISITOR SIGNOFF FORM

July 12, 2001

APPENDIX A

HEALTH AND SAFETY PLAN ACKNOWLEDGEMENT FORM

The following employees have read and understood the attached Health and Safety Plan:

Name	***Employer***	***Date***

July 12, 2001

VISITOR REVIEW

Each visitor to the Facility pertaining to the specified SOW shall sign this form after the pre-entry briefing is completed and before being permitted access to the Facility. A copy of this signed form shall be kept at the Facility, and the original sent to the HSO.

VISITOR SIGNOFF

I have attended a pre-entry briefing outlining the specific health and safety provisions for the SOW.

I have received a copy of the Health and Safety Plan. I have read the Plan, and will comply with the provisions contained therein.

Name (printed)	Signature	Date

Witness:

Name (printed)	Signature	Date

ACTING SAFETY OFFICER:

This person has provided verification of current OSHA 40-Hour hazardous waste site worker training.

Name (printed)	Signature	Date

July 12, 2001

TRAINING ACKNOWLEDGEMENT FORM

Please Print:

NAME: _____

ADDRESS: _____

SOCIAL SECURITY NUMBER: _____

EMPLOYER: _____

JOB SITE: _____

I have attended and understood the mandatory initiation session for the above-referenced job site. This program referenced the following topics:

i) known potential hazards;

ii) level of personal protection equipment required;

iii) emergency procedures; and

iv) explanation of the basics of the HASP.

I further confirm that I have the required 40 hours of training to comply with 29 CFR 1910.120 and have a respirator for which I have been fit tested.

Signature: _____

Date: _____

APPENDIX B

RESPIRATORY PROTECTION PROGRAM

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1.0 RESPIRATORY PROTECTION

1.1 PRACTICE

The primary control of respiratory hazards shall be accomplished, whenever feasible, through the use of engineering controls, hazard substitution, revised work practices or other administrative controls. However, when such controls are not feasible, appropriate respiratory protection shall be used in accordance with the procedures established in this Standard Operating Procedure (SOP). Any deviation from the requirements set forth must have approval from an Industrial Hygienist (IH).

1.1.1 AUTHORIZATION

CRA employees requiring respiratory protection must have authorization from an IH. Office management shall notify an IH of respirator needs. The work situation will then be assessed and a respirator will be issued based on the hazard(s) the individual is exposed to. Reassessment of the hazard and the individual's needs will be repeated periodically.

1.1.2 MEDICAL SURVEILLANCE

All personnel designated to use respirators must successfully complete a physical exam as part of the medical requirements placed on persons exposed to hazardous substances and for users of respiratory protection (29 CFR 1910.134 and 1910.120). The medical exam will be repeated annually. All medical data will be reviewed by a consulting occupational physician. The physician will generate a written opinion on the suitability of the employee to wear a respirator.

1.1.3 AIR QUALITY

When air supplied respirators are used, the breathing air shall meet the Compressed Gas Association (CGA-CG7.1) Standards for Grade D breathing air or better.

1.1.4 AIR CYLINDERS

Cylinders used to supply breathing air are tested and maintained as prescribed in Shipping Container Specifications (49 CFR 178). Self-Contained Breathing Apparatus (SCBA) cylinders have approximately 2400-psi of pressure when full. Compressed air

cylinders are visually inspected annually and hydrostatically tested once every five years for steel cylinders and once every three years for composite cylinders. These test dates are stamped on the cylinder for future reference.

1.2 FIT TESTING

1.2.1 QUALITATIVE OR QUANTITATIVE FIT TESTING

Fit testing of the respirator will be conducted by an IH or other designated, trained personnel following the medical evaluation. All users of respirators must be fit tested to ensure proper protection. Only the brand and size a person is fitted for is allowed to be used in the field. DO NOT SUBSTITUTE RESPIRATORS FOR A BRAND AND SIZE THAT YOU HAVE NOT BEEN PROPERLY FITTED FOR. The fit test will be accomplished quantitatively for full face APRs. Records will be maintained by the Corporate IH with copies available in CRA field offices for audit purposes. After fit testing, the employee will be issued an authorization card. This card serves as a reference for the proper type of respirator to use as well as prima facie proof of proper medical and training clearance for regulatory purposes. The user shall have the card available at any time when on CRA business where respiratory protection is used or hazardous waste cleanup sites are being entered.

1.2.2 POSITIVE & NEGATIVE FIT TESTING

All personnel will be instructed in the proper method of testing respirator fit by use of the **Positive & Negative** pressure test. This test is to be done by the user each and every time a respirator is donned. This test is performed to help the wearer assess respirator function and find gross leaks between the face and facepiece. This positive-negative pressure test checks the presence and functioning of the respirator valves as well as leakage that may occur due to improper cartridge seal or respirator face fit.

1.2.2.1 POSITIVE PRESSURE

1) Block off the exhalation valve cover openings.

   MSA: Exhalation valve cover can be blocked with the palm of the hand.

   Scott: Exhalation valve covers have front openings as well as four small side openings. These openings are difficult to block off with the hands. However, a small piece of flexible material such as Saran wrap or latex can be used.

North: Exhalation valve cover has long narrow openings around its perimeter. These can be blocked by encircling the fingers around the valve cover.

2) The person exhales gently, creating a slight positive pressure within the facepiece. The positive pressure should be maintained for at least 10 seconds.

3) If no outward leakage is detected, the person has passed the test.

4) If leakage is detected (usually felt as a cool sensation against the skin or a loss of pressure), the respirator is either malfunctioning or a gross leak between the face and facepiece is present. The following should be done when a failure occurs: Re-don or readjust the respirator.

If the facepiece continues to lose pressure, although previous positive or negative pressure tests performed with that respirator had passed, it is probably malfunctioning. Consult Industrial Hygiene. It is also possible that there are new scars or wrinkles, beard growth, missing teeth or dentures, significant weight gain or loss, etc. to cause gross leakage into the facepiece. When such new conditions exist, reevaluation of the respirator in a test atmosphere is necessary.

1.2.2.2 NEGATIVE PRESSURE

1) Block off the respirator cartridge inlet openings.

MSA: Cartridges can be blocked with the palms of the hands or with disposable latex gloves.

Scott: Cartridges can be blocked by using the palms of the hands or with gloves.

North: Cartridges can be blocked only with gloves.

2) Inhale gently, holding the negative pressure for at least 10 seconds.

3) If no inward leakage of air is detected, the person has passed the test.

4) If leakage is detected, see 4 above.

1.2.3 EXCEPTIONS

1.2.3.1 FACIAL HAIR

Any individual with facial hair which protrudes into the sealing surface of the masks will be refused fitting. Fitting, issuance, and use will be based on clean-shaven faces only. Employees with facial hair which interferes with respirator fit will not be permitted to work where respiratory protection must be worn.

1.2.3.2 GLASSES

Employees who wear prescription glasses and must wear a full-face respirator shall be fitted with special eyeglass adapters. Contact lenses will not be permitted when worn with any type of respiratory protection.

1.3 TRAINING

Proper training of respirator users is required to insure that all respirators will provide adequate protection against respiratory hazards and so that the user will understand the device's limitations. The training will include the following elements:

1) An explanation of the nature of respiratory hazards and what may happen if the respirator is not used properly.
2) A description of what engineering and administrative controls may be utilized to reduce the effects of the respiratory hazard and why respirators are required.
3) An explanation of the various types of respirators and why specific types have been selected.
4) A discussion of the function, capabilities, and limitations of respirator cartridges.
5) Instruction in the inspection, fit, and maintenance of the respirator.
6) Instructions in recognizing and handling emergency situations.

1.4 MAINTENANCE AND CARE OF RESPIRATORS

A program for the maintenance and care of respirators will include the following:

1) Inspection for defects;
2) Cleaning and disinfecting;

3) Repair/miscellaneous maintenance; and

4) Storage.

1.4.1 INSPECTION FOR DEFECTS

Employees shall inspect their respirators before each use. A respirator that is not routinely used but is kept ready for emergency use shall be inspected after each use and at least monthly to ensure that it is in satisfactory condition. SCBA shall be inspected monthly if kept for emergency response (i.e., long duration site remediation).

Inspection shall include a check of the tightness of the connections and the condition of the face piece, headbands, valves, breathing tubes and canisters. Rubber or elastic parts shall be inspected for pliability and signs of deterioration. Any respirator with worn or defective parts will be immediately taken out of service.

Before and after each use, respirators will be inspected for the following:

1) Tightness of connections and condition of the facepiece;
2) The headstraps or head harness should be examined for: breaks, loss of elasticity, broken or malfunctioning buckles and attachments, and excessively worn head-harness serrations that might permit slippage;
3) Valves and valve seats;
4) Connecting tube and canisters, air or oxygen cylinders;
5) Rubber or elastomer parts for pliability and deterioration; and
6) Regulators, fittings and gauges.

The following contains inspection information for various types of respirators.

Air Purifying Respirators

1) Check rubber facepiece for dirt, pliability of rubber, deterioration and cracks, tears, holes or distortion from improper storage.
2) Check straps for breaks, tears, loss of elasticity, broken snaps and proper tightness.
3) Check valves (exhalation and inhalation) for holes, warpage, cracks, etc. After removing its cover, the exhalation valve should be examined for: foreign material, distortion, defective or missing valve cover, or improper installation of valve into the valve seat.

4) Check filters or cartridges for dents, corrosion, etc. (loose or missing gaskets, improperly seated cartridges).

5) Check for cracked or badly scratched lenses in full facepieces; incorrectly mounted lens, broken or missing mounting clips.

Atmosphere-Supplying Respirators

1) Check appropriate items above.

2) Check air supply system for breaks or kinks in supply hoses and detachable coupling attachments.

3) Follow manufacturer's recommendations for the specific equipment.

4) Check air supply level and warning devices.

1.4.2 CLEANING AND DISINFECTING

The respirator must be washed after each day's use. If the respirator is shared it must be disinfected according to the manufacturer's instructions. Organic solvents of any kind must not be used for cleaning. Air purifying filters must not be wetted.

1.4.2.1 CLEANING

1) Remove any filters, cartridges or canisters and, if required by the manufacturer, straps and speaking diaphragms from the facepiece. Remove regulators on airline or SCBA.

2) Wash respirator parts excluding cartridges and canisters in warm (not to exceed 140°F), soapy water or in a product specifically designed by respirator manufacturers for this purpose. A plastic bristle hand brush may be helpful in removing dirt from respirator parts.

3) Rinse all parts thoroughly in warm water.

4) Air dry all parts.

5) Reassemble the respirator and insert new cartridges if needed.

6) Place the respirator in a plastic bag or container and seal it for storage. The respirator facepiece should be stored in its normal position so as not to distort the elastomer.

1.4.2.2 DISINFECTION

1) Disinfection should be done with a cleaner/disinfection agent purchased from the respirator vendor. If that material is not available the following NIOSH procedures can be followed:

   a) Immerse the respirator body for two minutes in a 50 ppm chlorine solution (about 2 ml bleach to 1 liter of water). Rinse thoroughly in clean water and air dry.

   b) Immerse the respirator body for two minutes in an aqueous solution of iodine (add 0.8 ml of iodine in 1 liter water). The iodine is about 7% ammonium and potassium iodide, 45% alcohol and 48% water. Rinse thoroughly in clean water and air dry.

2) Immersion times have to be limited to minimize damage to the respirator. The solutions can age rubber and rust metal parts.

   NOTE: The air-purifying elements must be removed from the respirator prior to cleaning and sanitizing the respirator. Never allow the air-purifying elements to come in contact with water or cleaning/sanitizing solution.

1.4.3 STORAGE

Respirators must be stored to protect them from contamination and mechanical damage at all times when not in use. New, cleaned or reconditioned respirators are to be kept in a clean, sealed plastic bag or container, stored in a normal position. The plastic bag should be labeled with the users name. A suitable cabinet or drawer should be used to protect respirators and supplies from dirt, extremes of temperature or bright sunlight. They are not to be left in vehicles or on perimeter fences, in change sheds, etc.

APPENDIX C

HEAT STRESS AND COLD STRESS PROCEDURES

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1.0 COLD STRESS

1.1 OVERVIEW

Fatal exposures to cold have been reported in employees failing to escape from low environmental air temperatures or from immersion in low temperature water. Hypothermia, a condition in which the body's deep core temperature falls significantly below 98.6 degrees Fahrenheit (°F), can be life threatening. A drop in core temperature to 95°F or lower must be prevented.

Air temperature is not sufficient to determine the cold hazard of the work environment. The wind-chill must be considered as it contributes to the effective temperature. The body's physiologic defense against cold includes constriction of the blood vessels, inhibition of the sweat glands to prevent loss of heat via evaporation, glucose production, and involuntary shivering to produce heat by rapid muscle contraction.

The frequency of accidents increases with cold temperature exposures as the body's nerve impulses slow down, individuals react sluggishly and numb extremities make for increased clumsiness. Additional safety hazards include ice, snow blindness, reflections from snow, and possible skin burns from contact with cold metal.

There are certain predisposing factors that make an individual more susceptible to cold stress. It is the responsibility of the project team members to inform the Health and Safety Officer to monitor an individual, if necessary, or use other means of preventing/reducing the individual's likelihood of experiencing a cold related illness or disorder.

1.2 PREDISPOSING FACTORS

Predisposing factors that will increase an individual's susceptibility to cold stress are listed below:

- Dehydration: The use of diuretics and/or alcohol, or diarrhea can cause dehydration. Dehydration reduces blood circulation to the extremities.
- Fatigue During Physical Activity: Exhaustion reduces the body's ability to constrict blood vessels resulting in the blood circulation occurring closer to the surface of the skin and the rapid loss of body heat.
- Age: Some older and very young individuals may have an impaired ability to sense cold.

- Alcohol Consumption: Alcohol dilates the blood vessels near the skin surface resulting in excessive body heat loss.
- Sedative Drugs: Sedatives may interfere with the transmission of impulses to the brain, thereby interfering with the body's physiological defense against cold. Some prescription drugs may react the same way.
- Poor Circulation: Vasoconstriction of peripheral vessels reduces blood flow to the skin surface.
- Heavy Work Load: Heavy work loads generate metabolic heat and make an individual perspire even in extremely cold environments. If perspiration is absorbed by the individual's clothing and is in contact with the skin, cooling of the body will occur.
- The Use of PPE: PPE usage which traps sweat inside the PPE may increase an individual's susceptibility to cold stress.
- Lack of Acclimatization: Acclimatization, the gradual introduction of workers into a cold environment, allows the body to physiologically adjust to cold working conditions.
- History of Cold Injury: Previous injury from cold exposures may result in increased cold sensitivity.

1.3 PREVENTION OF COLD STRESS

There are a variety of measures that can be implemented to prevent or reduce the likelihood of employees developing cold related ailments and disorders. These include acclimatization, fluid and electrolyte replenishment, eating a well balanced diet, wearing warm clothing, the provision of shelter from the cold, thermal insulation of metal surfaces, adjusting work schedules, and employee education.

- Acclimatization: Acclimatization is the gradual introduction of workers into the cold environment to allow their bodies to physiologically adjust to cold working conditions. However, the physiologic changes are usually minor and require repeated uncomfortably cold exposures to induce them.
- Fluid and Electrolyte Replenishment: Cold, dry air can cause employees to lose significant amounts of water through the skin and lungs. Dehydration affects the flow of blood to the extremities and increases the risk of cold injury. Warm, sweet, caffeine-free, non-alcoholic drinks and soup are good sources to replenish body fluids.

- Eating a Well-Balanced Diet: Restricted diets including low salt diets can deprive the body of elements needed to withstand cold stress. Eat high energy foods throughout the day.
- Warm Clothing: It is beneficial to maintain air space between the body and outer layers of clothing in order to retain body heat. However, the insulating effect provided by such air spaces is lost when the skin or clothing is wet.

The parts of the body most important to keep warm are the feet, hands, head and face. As much as 40 percent of body heat can be lost when the head is exposed.

Recommended clothing includes:

- Inner layers (t-shirts, shorts, socks) should be of a thin, thermal insulating material.
- Wool or thermal trousers. Denim is not a good protective fabric.

- Felt-lined, rubber-bottomed, leather-upper boots with a removable felt insole is preferred. Change socks when wet.
- Wool shirts/sweaters should be worn over inner layer.
- A wool cap is good head protection. Use a liner under a hard hat.
- Mittens are better insulators than gloves.
- Face masks or scarves are good protection against wind.
- Tyvek/poly-coated Tyvek provides good wind protection.
- Wear loose fitting clothing, especially footwear.
- Carry extra clothing in your vehicle.
- Shelters with heaters should be provided for the employees' rest periods if possible. Sitting in a heated vehicle is a viable option. Care should be taken that the exhaust is not blocked and that windows are partially open to provide ventilation.
- At temperatures of 30°F (-1°C) or lower, cover metal tool handles with thermal insulating material if possible.
- Schedule work during the warmest part of the day if possible, rotate personnel and adjust the work/rest schedule to enable employees to recover from the effects of cold stress.

1.3.1 EMPLOYEE EDUCATION

Employees have already been trained to recognize and treat the effects of cold stress during their 40-hour training. Signs, symptoms, and treatment of cold stress should be reviewed in project safety meetings where applicable. The buddy system will help in preventing cold stress once the employees are trained to recognize the signs and symptoms of cold stress.

1.3.2 COLD STRESS PREVENTION GUIDELINES

It may not be practically feasible to implement all the above prevention measures. Follow the guidelines given below when the ambient air temperature is -5°F (-20°C) or lower:

- Contact the Project Manager (PM) or the industrial hygienest to determine if the project team should be working in such temperatures;
- Dress warm;
- Replenish fluids and electrolytes at regular intervals;
- Provide shelter from the cold; and
- Adjust work/rest schedules.

1.3.3 ADJUST WORK-REST SCHEDULES

Follow the work/rest schedule on Table C.1. It is based on the cooling power of air that is a function of wind speed and ambient air temperature.

1.4 FIRST-AID TREATMENT GUIDELINES

The following describes symptoms of different stages in cold stress and the related first-aid treatment guidelines.

1.4.1 FROSTBITE

Stages

Incipient (frost nip)	May be painless. Tips of ears, nose, cheeks, fingers, toes, chin affected. Skin blanched white.
Superficial	Affects skin/tissue just beneath skin; turns purple as it thaws. Skin is firm, waxy; tissue beneath is soft, numb.
Deep	Tissue beneath skin is solid, waxy, white with purplish tinge. Entire tissue depth is affected.

First-Aid

Incipient	Warm by applying firm pressure - blow warm breath on spot or submerge in warm water (102°F to 110°F) (39°C to 43°C). Do not rub the area.
Superficial	Provide dry coverage, steady warmth; submerge in warm water.
Deep	Hospital care is needed. Do not thaw frostbitten part if needed to walk on. Do not thaw if there is danger of refreezing. Apply dry clothing over frostbite. Submerge in water; do not rub.

1.4.2 GENERAL HYPOTHERMIA

Stages

- Shivering
- Indifference
- Decreased Consciousness
- Unconsciousness
- Death

Symptoms

- Muscle Tension
- Uncontrollable Shivering
- Glassy Stare
- Decreased Muscle Function
- Speech Distortion
- Blue, Puffy Skin
- Slow Pulse
- Shallow Breathing
- Coordination Loss
- Stumbling
- Forgetfulness
- Freezing Extremities
- Dilated Pupils
- Fatigue

Emergency Response

- Keep person dry; replace wet clothing;
- Apply external heat to both sides of patient using available heat sources, including other bodies;
- Give warm liquids - not coffee or alcohol - after shivering stops and if conscious;
- Handle gently;
- Transport to medical facility as soon as possible; and
- If more than 30 minutes from a medical facility, warm person with other bodies.

2.0 HEAT STRESS

2.1 OVERVIEW

Heat induced occupational illnesses, injuries and reduced productivity occur in situations in which the total heat load (environmental plus metabolic) exceeds the body's capacities to maintain normal body functions without excessive strain. Heat stress is the sum of the heat generated in the body plus the heat gained from the environment minus the heat lost from the body to the environment. The body's response to heat stress is called heat strain. The level of heat stress at which excessive heat strain will result depends on the heat tolerance of the individual. Certain predisposing factors may reduce an individual's ability to tolerate heat stress.

Using PPE may put a hazardous waste worker at an increased risk of developing heat stress. Health effects may range from heat rash or heat fatigue to serious illness or death. Heat stress is caused by a number of interacting factors, including environmental conditions such as temperature and relative humidity, protective clothing which limits natural heat loss through perspiration, workload and the individual characteristics of the worker.

It is the responsibility of the project team members to inform the HSO or industrial hygienest if any of the predisposing factors listed below apply to them. This enables the HSO to monitor the individual if necessary, or use other means of preventing/reducing the individual's likelihood of experiencing a heat related illness or disorder.

2.2 PREDISPOSING

Predisposing factors that will increase the individual's susceptibility to heat stress are listed below:

- Lack of Physical Fitness: Such individuals experience more physiological strain including a higher heart rate, a higher body temperature, less efficient sweating and slightly higher oxygen consumption as compared to fit individuals.
- Obesity: Overweight individuals produce more heat per unit surface area than thin individuals and have a lowered ability to dissipate heat.
- Age: Older individuals may have a decreased ability to cope with heat stress.
- Dehydration: Dehydrated individuals will have a decreased ability to cool the body by sweating. Diarrhea can cause dehydration.

- Alcohol, Medications and Drug Use: Alcohol consumption may dehydrate individuals and certain medications/drugs may act as diuretics. Hence, the individual may have a decreased ability to lose heat by sweating.
- Infection, Sunburn, Illness and Certain Chronic Diseases: These factors may interfere with the body's normal mechanisms to lose heat.
- Heart Conditions or Circulatory Problems: Heat stress may place an additional strain on the heart and circulatory system that could harm the individual as well as decrease the individual's physiologic response.
- Low Salt Diet: Could affect the individual's electrolyte balance.
- Pregnancy
- Previous History of Heat Stroke or Heat Exhaustion: May increase the individual's susceptibility to heat stress.
- Heavy Work Load: Will generate metabolic heat thereby increasing the heat stress placed on the individual
- The Use of PPE Over Light Summer Clothing: This will decrease the ability of an individual to lose heat by sweating, as evaporative cooling can no longer occur.
- Lack of Acclimatization: Acclimatization is the gradual introduction of workers into a hot environment to allow their bodies to physiologically adjust to hot working conditions. Acclimatized individuals generally have lower heart rates and lower body temperatures. In addition, they sweat sooner and more profusely and even have more dilute sweat (thereby losing less electrolytes) than non-acclimatized individuals.

2.3 PREVENTION OF HEAT STRESS

There are a variety of measures that can be implemented to prevent or reduce the likelihood of employees developing heat stress related disorders. These include fluid and electrolyte replenishment, the provision of shelter from the sun and heat, work schedule adjustment, the use of cooling devices, acclimatization, heat stress monitoring and employee education, as discussed below:

- Fluid and Electrolyte Replenishment: Personnel should drink about 16 ounces of water before starting work and drink water at every break. To encourage water consumption, cool water and disposable cups should be made available. The normal thirst mechanism is not sensitive enough to ensure that enough water will be drunk to replace lost sweat. When heavy sweating occurs, personnel should be encouraged to drink more. Replacing body fluids with Gatorade is an

option. It is advisable to have Gatorade available if the air temperature is 70°F (21°C) or more and the workers are performing tasks with a moderate to heavy work load in chemical resistant clothing.

- Shelter From the Sun and Heat: Air-conditioned (if possible) or shaded areas should be made available for rest periods. Sitting in an air-conditioned truck is an acceptable option.
- Work Schedule Adjustment: Scheduling work for early mornings and/or late afternoons will avoid the hottest parts of the day and reduce the heat stress placed on personnel. Rotation of personnel will help reduce overexertion of workers and adjusting the work-rest schedule will help personnel recover from the effects of heat stress periodically.
- Use of Cooling Devices: The use of cooling devices like field showers, hose-down areas or cooling vests should be considered for project tasks that involve heavy work loads in chemical resistant clothing.
- Acclimatization: Acclimatization is the gradual introduction of workers into a hot environment to allow their body to physiologically adjust to hot working conditions. Acclimatized individuals generally have lower heart rates and lower body temperatures. In addition, they sweat sooner and more profusely and even have more dilute sweat (thereby losing less electrolytes) than non-acclimatized individuals.
- Heat Stress Monitoring: Monitoring hot environments for potential heat stress should be initiated when the ambient air temperature is in excess of 70°F (21°C). There are several ways to monitor heat stress: measuring heart rate, oral temperature, loss of body weight, and the Wet Bulb Globe Temperature using a Reuter-Stokes or Quest Electronics heat stress monitor.
- Employee Education: Workers have already been trained to recognize and treat the effects of heat stress during the 40-hour training course. Signs, symptoms, and treatment of heat stress should be discussed in safety meetings. The buddy system will help in preventing heat stress once the employees are trained to recognize the signs and symptoms of heat stress.

2.3.1 PREVENTION PRACTICES

It may not be practically feasible to implement all of the above prevention measures. The following has been developed as a field guide for use in actual field situations.

Ambient air temperature is 70°F (21°C) or more:

- Replenish fluids and electrolytes. Drink cool (50°F to 60°F/10°C to 15°C) fluids hourly. The fluids should be caffeine-free and non-alcoholic. Do not wait until you are thirsty. Your normal thirst mechanism is not sufficient to overcome the effects of dehydration. If you feel thirsty, you are already becoming dehydrated.

- Provide shelter from the sun and heat.

Ambient air temperature is 70°F (21°C) or more and chemical-resistant clothing is being used:

- Same as above;
- Adjust work schedules if feasible; and
- Initiate heat stress monitoring and/or the use of cooling devices.

2.3.2 HEAT STRESS MONITORING

Heat stress monitoring may be performed by monitoring the heart rate. Heart rate should be measured at the beginning of the work shift, at regular intervals and at the start of each rest period.

1) If the heart rate is <110 beats per minute (bpm), personnel may continue the current work/rest schedule.
2) If the heart rate is >110 bpm, take a 10 minute break. Monitor heart rate at the end of the rest period. If not <110 bpm, rest until the heart rate is <110 bpm. Reduce the current work time between breaks by approximately one hour. If the next scheduled monitoring session shows a heart rate of >110 bpm once again, reduce the work time between breaks by one hour.

2.4 HEAT STRESS FIRST AID

2.4.1 HEAT CRAMPS

Cause: Excessive water loss/electrolyte imbalance.

Symptoms	First-Aid Guidelines
Muscular pain in arms, legs, abdomen Faintness, dizziness, exhaustion Normal temp, cool moist skin person	Administer sips of Gatorade (1/2 glass every 15 minutes) Do not massage cramping muscles Relax

2.4.2 HEAT EXHAUSTION

Cause: Large amount of water loss; blood circulation diminishes.

Symptoms	First-Aid Guidelines
Moist, clammy, skin, usually pale Dilated pupils Weak, dizzy, nauseous, headache Normal or low body temperature	Move to a cool place Apply cold, wet compresses to skin Raise feet 8 to 12 inches Administer sips of Gatorade (1/2 glass every 15 minutes) Get medical attention

2.4.3 HEAT STROKE

Cause: Body overheats; temperature rises; no sweating occurs

Symptoms	First-Aid Guidelines
No sweating occurs	Get emergency medical assistance ASAP
Dry, hot skin, usually red	Remove from sunlight
Constricted pupils	Wet down body with cool water or rubbing alcohol
Hot body temperature (105°F to 110°F/40.5°C to 43.5°C)	Elevate head/shoulders
Strong, rapid pulse	Wrap in wet, cold wrapping
Unconsciousness may occur	Once cooled to 102°F (38.9°C), stop cooling measures
Muscular twitching	

TABLE C.1

WORK/REST SCHEDULE
FOR AN EIGHT HOUR WORK SHIFT

Air Temperature with Sunny Sky		*Work/Break Schedule (Minutes)*				
°F	*°C*	*No Wind*	*5 mph Wind*	*10 mph Wind*	*15 mph Wind*	*20 mph Wind*
-05 to -09	-20 to -23	110 / 10	110 / 10	75 / 10	55 / 10	40 / 10
-10 to -14	-23 to -26	110 / 10	75 / 10	55 / 10	40 / 10	30 / 10
-15 to -19	-26 to -28	75 / 10	55 / 10	40 / 10	30 / 10	Stop
-20 to -24	-29 to -31	55 / 10	40 / 10	30 / 10	Stop	Stop
-25 to -29	-32 to -34	40 / 10	30 / 10	Stop	Stop	Stop
-30 to -34	-35 to -37	30 / 10	Stop	Stop	Stop	Stop
-35 & Below	-38 & Below	Stop	Stop	Stop	Stop	Stop

Note:

This table addresses the health hazards related to cold weather work. The practicality of working under a work / rest schedule, together with the ability of the necessary equipment to function properly in cold weather, may be more restrictive than the health hazards and also need to be considered.

The cold stress schedule applies to light or sedentary work activities. Light to moderate work activities can be moved down one level, moderate to heavy work activities can be moved down two levels if workers are acclimated, have proper protective clothing and show no signs of cold stress.

0 - 5 mph Wind:	Light flag moves.
5 mph Wind:	Raises newspaper sheet.
10 mph Wind:	Light flag fully extended.
20 mph Wind:	Blowing and drifting snow.

JUNE 6, 2001

COMMUNITY RELATIONS PLAN

GM POWERTRAIN BEDFORD FACILITY
105 GM DRIVE
BEDFORD, INDIANA

U.S. EPA ID# IND 006036099

Prepared for:
GENERAL MOTORS CORPORATION

Prepared by:
Conestoga-Rovers & Associates

651 Colby Drive
Waterloo, Ontario
Canada N2V 1C2

Office: (519) 884-0510
Fax: (519) 884-0525

web: http:\\www.CRAworld.com

JUNE 6, 2001
REF. NO. 13968 (4)

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LIST OF ACRONYMS / SHORT FORMS

CRA	Conestoga-Rovers & Associates, Inc.
CRP	Community Relations Plan
ENCORE	Environmental Corporate Remediation Co. Inc.
GM	General Motors Corporation
IDEM	Indiana Department of Environmental Management
RCRA	Resource Conservation and Recovery Act
Facility	GM Powertrain-Bedford Plant
U.S. EPA	United States Environmental Protection Agency

1.0 INTRODUCTION

This Community Relations Plan (CRP) has been prepared for the General Motors Corporation (GM) Powertrain-Bedford Plant (Facility), located at 105 GM Drive, Bedford, Indiana as part of Corrective Action activities at the Facility. In March 2001, GM and Region 5 of the United States Environmental Protection Agency (U.S. EPA) signed a RCRA Voluntary Corrective Action Agreement (Appendix A) to conduct Corrective Action at the Facility. RCRA was enacted by Congress in 1976 to address the safe management and disposal of solid or hazardous waste. Corrective Action is the process of investigating and cleaning up, if necessary, areas of a facility that may have been affected by the management of hazardous waste and hazardous constituents. All facilities that ever applied for a permit to treat, store, or dispose of hazardous waste are subject to this kind of investigation.

The Facility was originally developed in the early 1900's to support limestone milling operations. In 1942, the operation was developed into an Allison Aircraft aluminum engine foundry. After World War II, the Facility remained an aluminum foundry, casting automotive parts as part of various GM divisions, including Delco-Remy, Allison, Fabricast, Central Foundry, and Powertrain. The Facility is still in operation today, producing aluminum casting products such as transmission castings, engine blocks, and pistons. The Facility covers 152.5 acres, and has approximately one million square feet of operating floor space. The Facility location is presented on Figure 1.1.

This Community Relations Plan (CRP) has been prepared to describe how GM will keep the community informed during the Corrective Action process, as well as the methods by which the community can participate in the process. GM is committed to informing and listening to community members throughout the process.

1.1 PROJECT TEAM

The Facility will be evaluated and remediated (where necessary) under the RCRA Voluntary Corrective Action Agreement between U.S. EPA and GM and GM will work closely with U.S. EPA during all phases of the project. GM's team includes highly committed staff and consultants with expertise in environmental studies, human health and ecological risk assessment, engineering, and community relations. Experienced staff at the Facility will be closely involved with the Corrective Action project. The Corrective Action Project Team is presented in Figure 1.2.

2.0 COMMUNITY RELATIONS PROGRAM ACTIVITIES

2.1 COMMUNITY RELATIONS GOALS

GM will conduct community relations in accordance with the RCRA Voluntary Corrective Action Agreement for the Facility.

The goals of the CRP are as follows:

1. establish an open working relationship with the community;
2. keep the public informed as the project progresses and as investigative results are received;
3. provide a mechanism for disseminating information on a routine, as well as a non-routine, basis in response to individual requests; and
4. provide a mechanism for the public to provide feedback and input to GM.

2.2 CRP IMPLEMENTATION

2.2.1 SCOPE

Implementation of the CRP will provide a mechanism for dissemination of information from GM to the community and to receive input from the community during the course of the Corrective Action project. This section presents the activities necessary for implementation of the various community relation goals identified in Section 2.1.

2.2.2 PUBLIC INFORMATION REPOSITORY

An information repository will be established at the Bedford Public Library located at 1323 K Street, Bedford, Indiana. The library is within approximately five miles of the Facility. The phone number for the Bedford Public Library is (812) 275-4471.

The information repository will provide public access to final copies of work plans, technical reports, and project fact sheets.

2.2.3 PROJECT FACT SHEETS

GM will prepare project fact sheets to be distributed to the public. Copies of these Fact Sheets will be available at the Library and at City Hall. The fact sheets will briefly describe the history of the Facility, activities planned or being performed at the Facility and describe the status of the project.

Project Fact Sheet 1 (May 2001) is presented in Appendix B.

2.2.4 POINT OF CONTACT

GM has designated a point of contact to provide the public with an individual who can be contacted directly to provide accurate information on the status of the Corrective Action project and receive input from the community. The GM contact is:

Ms. Kathy Bommarito
(248) 857-6386
kathy.bommarito@gm.com

2.2.5 RCRA VOLUNTARY CORRECTIVE ACTION CONTACT LIST

GM will develop a contact list that will be used to keep parties informed about activities related to the Corrective Action project and will include elected and appointed officials, and the local media. If community members would like to be included on this contact list, they may telephone the designated point of contact identified in Section 2.2.4.

2.2.6 PUBLIC COMMENT

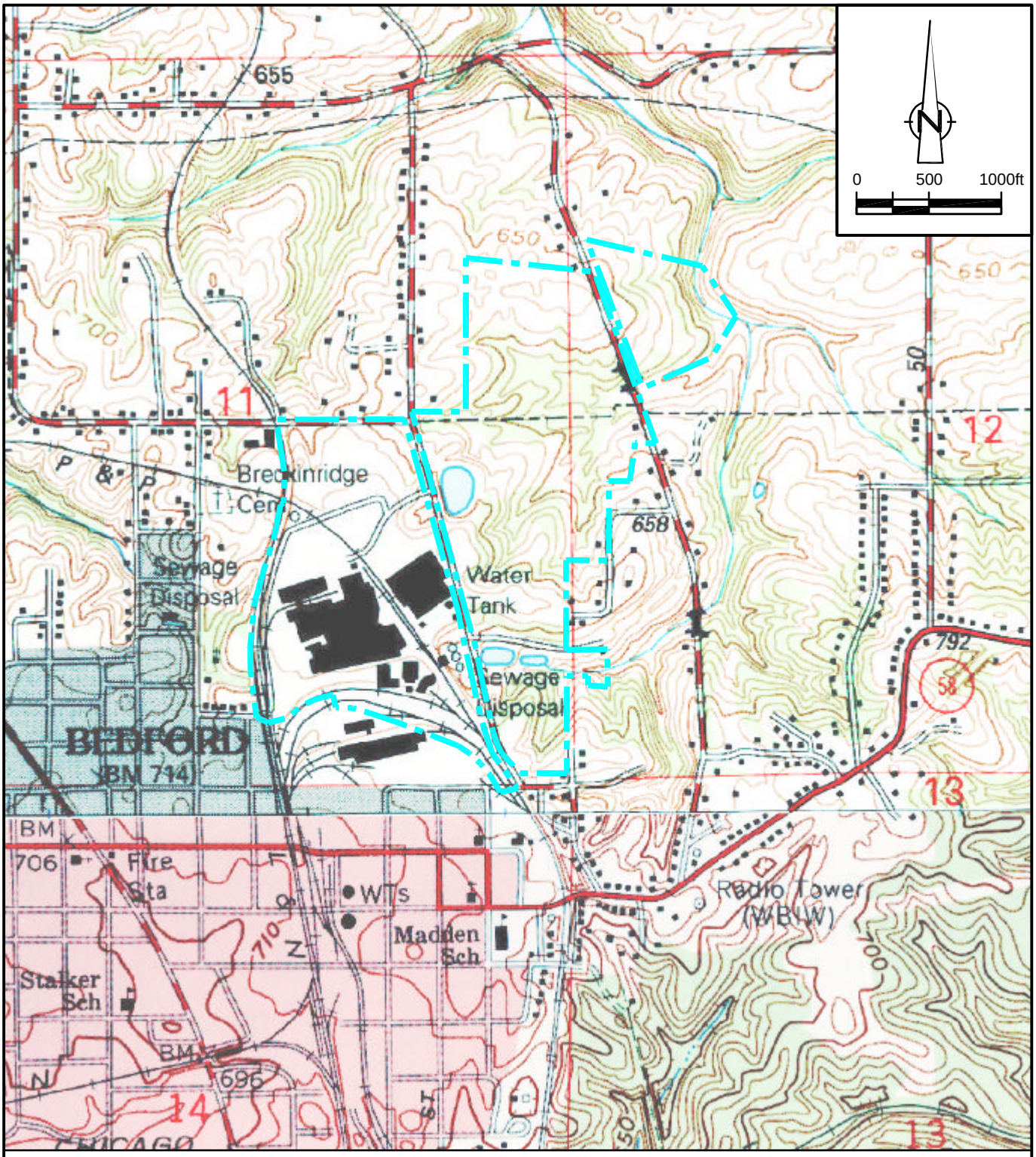
Once the Facility investigation is complete, GM will prepare a Corrective Measures Study (CMS) that defines and evaluates potential cleanup options and recommends a cleanup plan. U.S. EPA will review the CMS and propose the final cleanup plan for the Facility. U.S. EPA will provide for public comment on the proposed plan and then make a final decision based on the comments received.

2.2.7 ADDITIONAL COMMUNITY SUPPORT

This CRP is intended to be flexible and to adapt to the Corrective Action process and community interests. GM will, as necessary, respond to community comments or concerns as they are expressed and may evaluate the use of additional community relations tools during the project, if appropriate.

3.0 SUMMARY

This CRP was developed for this project based upon the project activities that will be conducted under the RCRA Voluntary Corrective Action Agreement for the Facility. This CRP will be revised, as necessary, to keep the community informed about, and involved in, the RCRA Voluntary Corrective Action process. The community relations activities are summarized in Table 3.1.



BASE SOURCE: USGS 7.5 MINUTE TOPOGRAPHIC QUADRANGLE;
BARTLETTSVILLE, INDIANA 1994
BEDFORD EAST, INDIANA 1978
BEDFORD WEST, INDIANA 1993
OOLITIC, INDIANA 1987

LEGEND

FACILITY BOUNDARY



figure 1.1

FACILITY LOCATION
GM POWERTRAIN BEDFORD FACILITY
Bedford, Indiana

13968-00(004)GN-WA002 JUN 19/2001

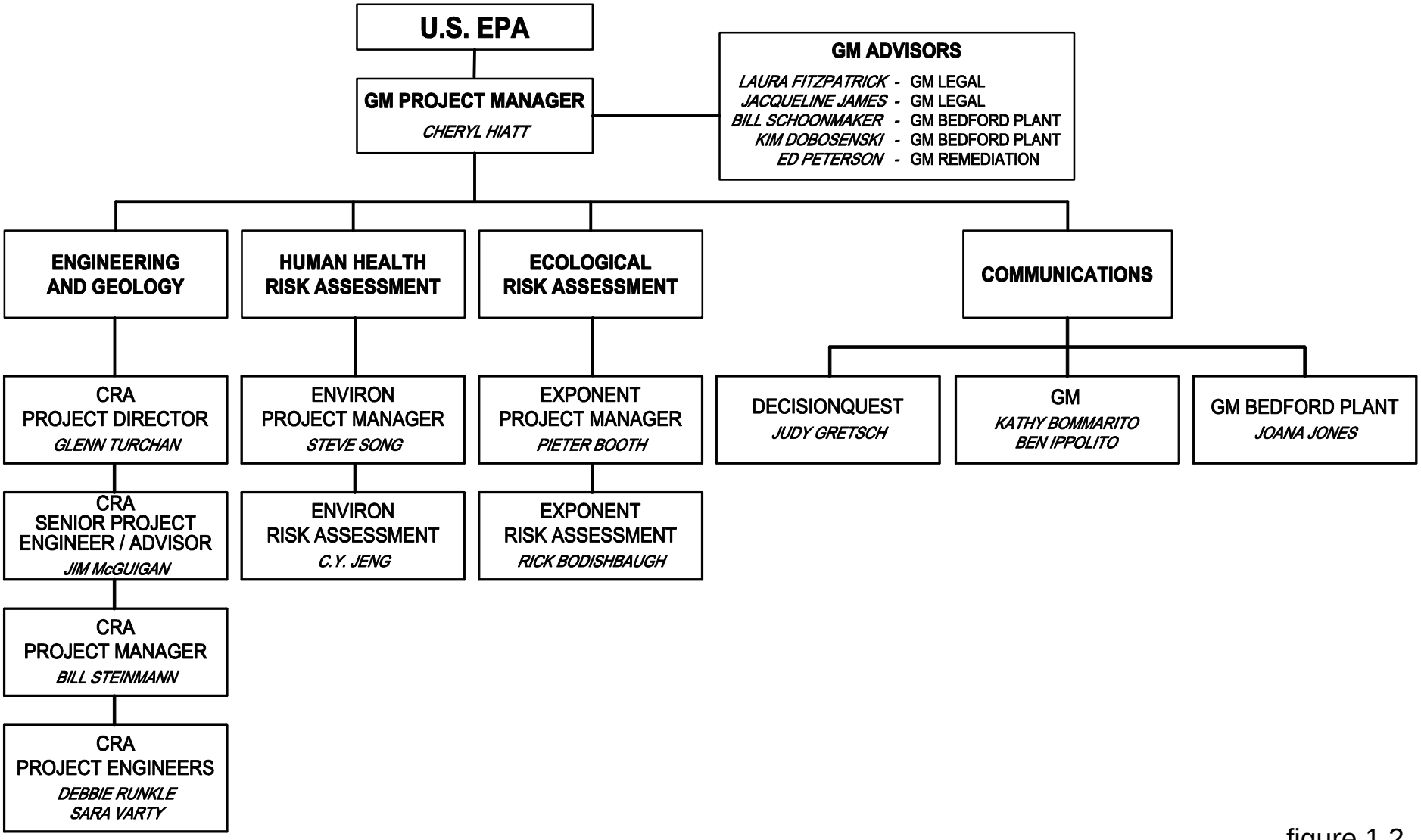


figure 1.2

PROJECT TEAM ORGANIZATION
GM POWERTRAIN BEDFORD FACILITY
Bedford, Indiana

TABLE 3.1

SUMMARY OF POTENTIAL COMMUNITY RELATIONS ACTIVITIES
GM POWERTRAIN BEDFORD FACILITY
BEDFORD, MICHIGAN

1. Establish Public Information Repository

Objective: To provide the community with access to comprehensive information, reports, and data produced throughout the RCRA Voluntary Corrective Action project at the Facility.

Action: An information repository will be established at a local public library.

Discussion: The repository will include items such as technical reports, work plans, and project fact sheets. In general, all pertinent project information will be copied to the repository (final work products only).

2. Prepare and Distribute Project Fact Sheets

Objective: To inform the public of RCRA Voluntary Corrective Action activities at the Facility.

Action: Project fact sheets will be issued periodically.

Discussion: Project fact sheets can be an effective method of providing information to the public.

3. Designate a Point of Contact

Objective: To provide the public with a point of contact who can provide accurate information regarding the status of the RCRA Voluntary Corrective Action project.

Action: The point of contact is:

GM Communications
Ms. Kathy Bommarito
(248) 857-6386
kathy.bommarito@gm.com

Discussion: The contact person will coordinate and direct responses to inquiries. When appropriate, technical personnel will assist in providing responses.

TABLE 3.1

SUMMARY OF POTENTIAL COMMUNITY RELATIONS ACTIVITIES
GM POWERTRAIN BEDFORD FACILITY
BEDFORD, MICHIGAN

4. Prepare and Maintain a Contact List

Objective: To facilitate communication with interested community members.

Action: The contact list will be developed initially through research and informal meetings with Facility neighbors and elected and appointed officials. The contact list will be periodically updated.

Discussion: An established contact list helps to ensure that information reaches interested community members.

5. Public Comment

Objective: To provide for public comment on U.S. EPA's proposed final cleanup plan for the Facility.

Action: U.S. EPA will provide for public comment on its proposed final cleanup plan for the Facility.

Discussion: U.S. EPA encourages public involvement during the RCRA Voluntary Corrective Action process.

6. Provide Community Support

Objective: To release accurate information, as needed.

Action: Press releases and public meetings will be conducted on an as-needed basis. Additional Facility-specific community support will be determined, as required.

Discussion: Press releases would be sent to the appropriate local media and the public would be notified in advance of public meetings or other community support activities.

APPENDIX A

VOLUNTARY RCRA CORRECTIVE ACTION AGREEMENT

Worldwide Facilities Group
Environmental & Regulatory Support
Remediation Team

March 20, 2001

Mr. Robert Springer, Director
Waste Pesticides and Toxics Division
Region 5 U.S. Environmental Protection Agency
77 W. Jackson Blvd (DW-8J)
Chicago, Illinois 60604-3590

RE: Return of Fully Executed Bedford Voluntary Corrective Action Agreement
Bedford, Indiana

Dear Mr. Springer:

Enclosed is one fully executed copy of the Voluntary Corrective Action Agreement (signed by U.S. EPA on March 16, 2001) for the General Motors Powertrain Bedford Plant Site (U.S. EPA ID# IND006036099), located in Bedford, Indiana. Figure 1 is being sent under separate cover. Please note that General Motors recognizes the need to protect human health and the environment and will not wait until the end of the project to address those issues. GM intends to take interim measures, where practicable, during the process to address problems as they are identified and defined.

Please call Cheryl Hiatt at 313-556-9032 or Jean Caufield at 313-556-0845 if you require any further information.

Sincerely,

William J. McFarland

William J. McFarland
Director Remediation
Worldwide Facilities Group
General Motors Corporation

Enclosure

pc: Peter Ramanauskas, U.S. EPA
Hak Cho, U.S. EPA
Gerald Phillips, U.S. EPA
Jean Caufield, GM WFG
Cheryl Hiatt, GM WFG
Michelle Fisher, GM Legal
Laura Fitzpatrick, GM Legal
Bill Schoonmaker/Kim Dobosenski, GM Bedford

485 West Milwaukee Avenue • Mail Code 482-310-004 • Detroit, MI 48202-3220 • FAX (313) 556-0803

Voluntary Corrective Action Agreement
Between
The United States Environmental Protection Agency and
General Motors Corporation
For GM Powertrain Group - Bedford Plant, Bedford

I. Purpose

The United States Environmental Protection Agency (U.S. EPA) and General Motors Corporation (GM), collectively referred to as the Parties, establish this Agreement to enable GM to work independently and voluntarily to investigate, and as necessary stabilize and remediate releases of hazardous wastes or hazardous constituents at or from the GM Powertrain Group-Bedford Plant, more particularly delineated in attached Figure 1, (the "Site") located in Bedford, Indiana that may present an unacceptable risk to human health or the environment. The Parties believe that GM will appropriately, efficiently and effectively investigate and, as necessary, remediate the Site on an accelerated basis by following the procedures and guidelines in this Agreement.

II. Background

The Site (U.S. EPA ID# IND006036099) is located at 105 GM Drive, Bedford (Lawrence County), Indiana, 47421. The Bedford Plant produces aluminum casting products, such as transmission cases, pistons, and engine blocks. Major aluminum production processes include die casting and permanent molding.

The Site is bounded to the south by the Canadian and Pacific Railway, Imco, and residential property, to the east by undeveloped land and residential areas, to the north by residential and undeveloped areas, and to the west by former railway, residential, and industrial property.

The Site was originally developed in the early 1900s as limestone milling operations. In 1942, the operation became an Allison Aircraft aluminum engine foundry. After World War II, the Site remained an aluminum foundry, casting automotive parts as part of various General Motors divisions, including Delco-Remy, Allison, Fabricast, Central Foundry, and Powertrain. The Bedford Plant is still in operation today. In 1977, GM purchased a piece of property from the railroad, south of the original Plant, that had formerly included the railroad roundhouse. This property was leased by Mica Metals (a dross reclaimer of aluminum) until 1997. Other parcels, mostly undeveloped, have also been purchased over the years to the north and east of the original property boundaries. The Site today includes 152.5 acres (see attached site map in Figure 1), with approximately one million square feet of floor space.

U.S. EPA and GM expect that GM will investigate, and as necessary remediate, all releases of hazardous wastes or constituents at or from the Site under the guidelines established in this Voluntary Corrective Action Agreement.

III. Definitions

Unless otherwise specified herein, terms used in this Agreement which are defined in RCRA or in regulations promulgated under RCRA will have the definitions given to them in RCRA or in such regulations.

IV. Project Manager

U.S. EPA and GM will each designate a Project Manager and notify each other in writing of the Project Manager selected within 14 days of the effective date of this Agreement. Each Project Manager will be responsible for overseeing the implementation of this Agreement. The parties will provide prompt written notice whenever they change Project Managers.

V. Work to be Performed

GM agrees to perform the actions specified in this section, in the manner and by the dates specified herein. GM will perform the work undertaken pursuant to this Agreement in compliance with RCRA and other applicable Federal and State laws and their implementing regulations, and consistent with all relevant U.S. EPA guidance documents as appropriate to the Site. This guidance includes, but is not limited to, the Documentation of Environmental Indicator Determination Guidance, and relevant portions of the Model Scopes of Work for RCRA Corrective Action and of U.S. EPA's Risk Assessment Guidance for Superfund.

1. GM will complete activities necessary to identify and define the nature and extent of releases of hazardous waste and/or hazardous constituents at or from the Site. These activities include:

   a. Provide to U.S. EPA, within 120 days after the effective date of this Agreement, a brief Current Conditions Report covering all areas of the Site. The Current Conditions Report will include any recent sampling data and a summary of the historic operations, remedial efforts, and physical setting of the Site. The Current Conditions Report, in total, will describe, at a minimum, conditions at the locations identified in the Indiana Department of Environmental Management (IDEM) Preliminary Assessment, dated April 30, 1985 and any past or present locations at the Site for which GM knows of past treatment, storage, or disposal of hazardous waste or hazardous constituents.

   b. Perform phased investigations, at a minimum, at the locations identified in the Current Conditions Report as needing further investigation to identify the nature and extent of any releases of hazardous waste and/or hazardous constituents at

or from the Site which may pose an unacceptable risk to human health or the environment, and provide a report to U.S. EPA. The report will also describe the nature and extent of any releases of hazardous waste and/or hazardous constituents at or from the Site which do not pose an unacceptable risk to human health or the environment, and provide the basis for those conclusions, including an evaluation of the risks. The report may be prepared in phases to provide timely support for the demonstrations described in Section V.2, below, and for the determinations and proposal described in Section V.3, below. If investigation determines that acceptable risks to human health or the environment are exceeded, GM will determine the need for interim measures based on a professional evaluation of the data and will notify the U.S. EPA of the planned course of action.

c. GM may choose to proceed with remedial actions to limit site investigation or risk assessment activities in order to complete the work as defined in Sections V.2 and V.3 below.

2. GM will demonstrate by the dates indicated, through submitting an Environmental Indicators Report and by performing any other necessary activities, consistent with this Section, that:

a. By January 30, 2005, all current human exposures to contamination at or from the Site are under control. That is, for all media known or reasonably suspected to be contaminated with hazardous wastes or hazardous constituents above risk-based levels, for which there are complete pathways between contamination and human receptors, significant or unacceptable exposures do not exist.

b. By April 30, 2007, migration of contaminated groundwater at or from the Site is stabilized. That is, the migration of all groundwater known or reasonably suspected to be contaminated with hazardous wastes or hazardous constituents above acceptable levels is stabilized to remain within any existing areas of contamination as defined by monitoring locations designated at the time of the demonstration. In addition, any discharge of groundwater to surface water is either insignificant or shown to be currently acceptable according to an appropriate interim assessment. Monitoring and measurement data must be collected in the future as necessary to verify that migration of any contaminated groundwater is stabilized.

c. In order to prepare for and provide the demonstrations required by Sections V.2.a and V.2.b., above, GM will:

i. Determine appropriate risk screening criteria under current use scenarios and provide the basis and justification for the use of these criteria.

ii. Determine any current unacceptable risks to human health and the environment and describe why other identified risks are acceptable.

iii. Control any unacceptable current human exposures that are identified. This may include performing any corrective actions or other response measures ("Corrective Measures") necessary to control current human exposures to contamination to within acceptable risk levels.

iv. Stabilize the migration of contaminated groundwater. This may include implementing any Corrective Measures necessary to stabilize the migration of contaminated groundwater.

v. Conduct groundwater monitoring to confirm that any contaminated groundwater remains within the original area of contamination.

vi. Prepare a report, either prior to or as part of the Environmental Indicators Report, that provides a description and justification for any interim actions performed to meet the requirements of this Section, including sampling documentation, construction completion documentation, and/or confirmatory sampling results.

3. Final Corrective Measures:

a. GM will propose to U.S. EPA by December 31, 2007, final Corrective Measures necessary to protect human health and the environment from all current and future unacceptable risks due to releases of hazardous waste or hazardous constituents at or from the Site (the "Final Corrective Measures Proposal"). The proposal will describe all Corrective Measures implemented at the Site since the effective date of this Agreement. It will also include a description of all other final Corrective Measures evaluated by GM, a detailed explanation of why the proposed final Corrective Measures were preferred by GM, and cost estimates for the final Corrective Measures evaluated. The proposal will also include a detailed schedule to construct and implement the final Corrective Measures and to submit a Final Remedy Construction Completion Report. This schedule will provide that as much of the initial construction work as practicable will be completed within one year after U.S. EPA selects the final Corrective Measures and that all final Corrective Measures will be completed within a reasonable period of time to protect human health and the environment.

b. As part of developing its proposals, GM must propose appropriate risk screening criteria, cleanup objectives, and points of compliance under current and reasonably expected future land use scenarios and provide the basis and justification for these decisions.

c. U.S. EPA may request supplemental information from GM if it determines that the proposals and supporting information do not provide an adequate basis to select final Corrective Measures that will protect human health and the environment from the release of hazardous waste or hazardous constituents at or

from the Site. GM will provide such supplemental information in a timely manner as directed in writing by U.S. EPA.

d. U.S. EPA will provide the public with an opportunity to review and comment on its proposed final Corrective Measures, including a detailed description and justification for the proposals (the "Statement of Basis"). Following the public comment period, U.S. EPA will select the final Corrective Measures and provide notification of its decision and rationale in a "Final Decision and Response to Comments ("Final Decision").

e. If GM agrees with U.S. EPA's selection of final Corrective Measures, GM will implement the final Corrective Measures selected in U.S. EPA's Final Decision according to the schedule therein.

4. Reporting and other requirements:

a. GM will establish a publicly accessible repository for information regarding site activities and conduct public outreach and involvement activities in accordance with U.S. EPA's RCRA public participation guidance, as appropriate for the Site.

b. GM will provide quarterly progress reports to U.S. EPA by the 15th day of the month following a quarter. The report will list work performed to date, data collected, problems encountered, project schedule, and percent project completed. The parties will communicate frequently and in good faith to assure successful completion of the requirements of this Agreement, and will meet on at least a semi-annual basis to discuss the work proposed and performed under this Agreement.

c. GM will provide a Final Remedy Construction Completion Report documenting all work that it has performed pursuant to the schedule in U.S. EPA's Final Decision.

d. If ongoing monitoring or operation and maintenance is required after construction of the selected final Corrective Measures, GM will include an operations and maintenance plan in the Final Remedy Construction Completion Report. GM will revise and resubmit the Report in response to U.S. EPA's written comments, if any, by the dates U.S. EPA specifies. Upon U.S. EPA's written approval, GM will implement the approved operation and maintenance plan according to the schedule and provisions contained therein.

e. Any risk assessments conducted by GM must estimate human health and ecological risk under reasonable maximum exposure for both current and reasonably expected future land use scenarios. Risk assessments will be conducted in accordance with the Risk Assessment Guidance for Superfund (RAGS) or other appropriate U.S. EPA guidance. GM will use appropriate, conservative screening values when screening to determine whether further investigation is required. Appropriate screening values may include those

derived from Federal Maximum Contaminant Levels, U.S. EPA Region 9 Preliminary Remediation Goals, U.S. EPA Region 5 Ecological Screening Levels, U.S. EPA Region 5 Risk Based Screening Levels, U.S. EPA Region 3 Risk Based Concentration Table, or RAGS.

f. All sampling and analysis conducted under this Agreement will be performed in accordance with the Region 5 RCRA Quality Assurance Project Plan Policy (April 1998) as appropriate for the Site, and be sufficient to identify and characterize the nature and extent of all releases. U.S. EPA may audit laboratories selected by GM or require GM to purchase and have analyzed any Performance Evaluation (PE) samples selected by U.S. EPA which are compounds of concern. GM will notify U.S. EPA in writing at least 14 days before beginning each separate phase of field work performed under this Agreement. At the request of U.S. EPA, GM will provide or allow U.S. EPA or its authorized representative to take split or duplicate samples of all samples collected by GM under this Agreement.

VI. Record Preservation

GM will retain, during the pendency of this Agreement and for at least six (6) years after termination of the entire Agreement, all data and all final documents now in its possession or control or which come into its possession or control which relate to this Agreement. GM will notify U.S. EPA in writing 90 days before destroying any such records, and provide U.S. EPA the opportunity to take possession of any such non-privileged documents. GM's notice will refer to the effective date, caption, and docket number of this Agreement and will be addressed to:

Director
Waste, Pesticides and Toxics Division
U.S. EPA, Region 5 (D-8J)
77 W. Jackson Blvd.
Chicago, IL 60604-3590

GM will also promptly provide U.S. EPA's Project Manager a copy of any such notice.

GM further agrees that within 30 days of retaining or employing any agent, consultant, or contractor ("Agents") to carry out the terms of this Agreement, GM will enter into an agreement with the Agents to provide GM a copy of all data and final non-privileged documents produced under this Agreement.

GM agrees that it will not assert any privilege claim concerning any data developed to prepare any reports or conduct any investigations or other actions taken under the Agreement.

VII. Modification, Termination and Satisfaction

This Agreement may be modified by written, mutual agreement of the Parties. The Project Managers may agree in writing to extend any deadline in this Agreement. Either Party may unilaterally terminate this Agreement upon written notice to the other Party. U.S. EPA's participation in this Agreement is subject to the Anti-Deficiency Act, 31 USC § 1341.

GM may request that U.S. EPA issue a determination that GM has met the corrective action cleanup objectives for the Site or a portion of the Site. GM will submit documentation that it has achieved the objectives and U.S. EPA will respond in writing indicating whether GM has completed RCRA corrective action. If U.S. EPA agrees that RCRA corrective action is complete it will issue a "No Further Action" determination for all or a portion of the Site.

The provisions of the Agreement will be satisfied upon GM's and U.S. EPA's execution of an "Acknowledgment of Termination and Agreement on Record Preservation and Reservation of Rights", consistent with U.S. EPA's Model Scope of Work. GM's execution of the Acknowledgment will affirm its continuing obligation to preserve all records as required by Section VI, to maintain any necessary institutional controls or other long term measures, and to recognize the Parties' reservation of rights as required in Section VIII.

VIII. Reservation of Rights

The Parties reserve any and all rights, remedies, authorities or defenses that they respectively have under law. Nothing in this Agreement limits or affects the authority or ability of either Party to take any action authorized by law. Nothing in this Agreement creates any legal rights, claims or defenses in either Party or by or for any third Party. Nothing in this Agreement relieves GM from complying with applicable federal, state and local laws.

This Agreement does not limit or affect the rights of the Parties against any Third Party, nor does it limit the rights of Third Parties. The Parties agree that this Agreement does not constitute any decision on pre-authorization of funds under §111(a)(2) of CERCLA.

IX. Effective Date

This Agreement is effective on the date the last Party signs.

DATE: 3-20-01 BY: William J McFarland

William J. McFarland, Director
Remediation
Worldwide Facilities Group
General Motors Corporation

DATE: March 16, 2001 BY: Robert Springer

Robert Springer, Director
Waste, Pesticides and Toxics Division
U.S. Environmental Protection Agency
Region 5

APPENDIX B

PROJECT FACT SHEET

VOLUNTARY RCRA CORRECTIVE ACTION
GM POWERTRAIN BEDFORD FACILITY
BEDFORD, INDIANA
May 24, 2001

This is the first Project Fact Sheet that has been prepared to provide the community with an introduction to activities to be performed at the General Motors Corporation (GM) Bedford facility.

The facility is located at 105 GM Drive in Bedford, Indiana, on approximately 150 acres. The facility currently produces aluminum casting products, such as transmission casings, pistons, and engine blocks, within approximately one million square feet of operating floor space.

The Bedford facility was originally constructed in the early 1900's as a limestone milling operation. The U.S. Government purchased the mill in 1942, converting the facility to an aluminum aircraft engine foundry, which was operated by GM. GM purchased the property after World War II and has continued to operate the facility as an aluminum foundry as part of various GM operating divisions.

GM is currently initiating a Resource Conservation and Recovery Act (RCRA) Corrective Action program at the facility pursuant to a Voluntary Corrective Action Agreement between the U.S. EPA and GM, which became effective in March 2001. Corrective Action is required for all facilities who applied for a permit to store hazardous waste and is basically an evaluation of whether plant waste management operations have impacted the environment. The Agreement states that GM will work with U.S. EPA voluntarily to identify whether releases of hazardous constituents have occurred at or from the Bedford facility, define the nature and extent of potential releases, and complete cleanups, as needed.

Project Fact Sheets will be issued periodically during the implementation of the Corrective Action program to keep the community informed of ongoing activities at the facility.



March 27, 2001 Aerial Photograph

project fact sheet

The RCRA Corrective Action at the facility will include completion of the following activities:

1. Current Conditions Report (CCR)
   - *Report summarizing available information on the plant.*
   - *GM is currently completing the CCR*

2. RCRA Facility Investigation (RFI)
   - *Investigates open environmental issues identified in the CCR.*

3. Interim Cleanup Activities (as needed)

4. Final Cleanup Activities (as needed)

The CCR will be sent to U.S. EPA and the Indiana Department of Environmental Management (IDEM) upon completion in May and will be available for public review in a local information repository (to be established).

A toll free number will be established as a point of contact for the public.

Please direct any questions about this project to Ms. Kathy Bommarito of GM by phone at (248)857-6386 or by e-mail at kathy.bommarito@gm.com.

ENCORE