CRA

RFI WORK PLAN
REVISION 0
MAY 16, 2000

RCRA FACILITY INVESTIGATION (RFI) WORK PLAN

FORMER PEREGRINE (US) INC. COLDWATER ROAD FACILITY GENESEE TOWNSHIP, MICHIGAN

U.S. EPA ID# MIR000020743 (Peregrine ID)

Volume I - Text, Tables, Figures, Appendices A and B

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MAY 16 2000

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LIST OF ACRONYMS

AOC	Area of Interest
AST	Aboveground Storage Tank
Bgs	Below Ground Surface
CRA	Conestoga-Rovers & Associates
Delphi	Delphi Interior and Lighting Systems
DOCC	Description of Current Conditions Report
ERNS	Emergency Response Notification System
ESA	Environmental Site Assessment
20xGCC	Michigan 20xGeneric Groundwater Contact Criteria
GIHBDW	Generic Industrial Health Based Drinking Water Values
GM	General Motors Corporation
ID	Identification
IDC	Michigan Generic Industrial Direct Contact Standards
LUST	Leaking Underground Storage Tank
MCL	Maximum Contaminant Level
MDEQ	Michigan Department of Environmental Quality
MDNR	Michigan Department of Natural Resources
mgd	Million Gallons per Day
mph	Miles Per Hour
NPDES	National Pollutant Discharge Elimination System
PA	Preliminary Assessment
PA/VSI	Preliminary Assessment/Visual Site Inspection
PAOC	Potential Area of Concern
PCB	Polychlorinated Biphenyls
PNA	Polynuclear Aromatic Organic Compounds
ppb	Parts Per Billion
RCRA	Resource Conservation and Recovery Act
REALM	Remediation and Liability Management Co., Inc.
SSL	Soil Screening Level
STD	Storage Tank Division
SWMU	Solid Waste Management Unit
TCE	Trichloroethene
TPH	Total Petroleum Hydrocarbons
U.S. EPA	United States Environmental Protection Agency
UST	Underground Storage Tank
VOC	Volatile Organic Compounds
VSI	Visual Site Inspection
WWTP	On-site Wastewater Treatment Plant

1.0 INTRODUCTION

This document presents a work plan for the Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) at the Former Peregrine (US) Inc. (Peregrine) Coldwater Road Facility (Facility) in Genesee Township, Michigan (EPA ID# MIR000020743) (RFI Work Plan). This RFI Work Plan was prepared by Conestoga-Rovers & Associates (CRA) on behalf of the Remediation and Liability Management Company, Inc. (REALM), the current owner of the property.

1.1 GENERAL

The Facility is located in Genesee Township, Genesee County, Michigan, approximately two miles north of the City of Flint, Michigan, at 43° 45′ north latitude and 83° 0.5′ west longitude. The Facility is located on approximately 118.7 acres of land at G1245 East Coldwater Road. The general geographic location of the Facility is presented on Figure 1.1.

The main manufacturing building, Building 44 (approximately two million square feet), and associated buildings located at the Facility have been historically used for the manufacturing of automotive interior components. A Facility plan is presented on Figure 1.2/Plan 1. Prior to sale of the Facility to Peregrine at the end of 1996, the Facility was operated as the Delphi Interior and Lighting Systems (Delphi) Coldwater Road Plant, a division of the General Motors Corporation (GM) (U.S. EPA ID# MID005356860). The Facility sold to Peregrine was only a portion of the former property previously owned and operated by GM (which included a closed landfill located to the north of the main factory building). GM resumed ownership of the Facility in August 1999 when Peregrine ceased operations and assigned title to REALM, a wholly owned subsidiary of GM. An evaluation of the history of operations of this Facility was conducted and is summarized in the Description of Current Conditions Report (CRA, March 31, 2000) (DOCC) which was submitted to the Michigan Department of Environmental Quality - Waste Management Division (MDEQ-WMD on March 31, 2000). The DOCC listed twenty one (21) Areas of Interest (AOIs) which were identified during the review of the Facility during the preparation of the DOCC.

1.2 RFI WORK PLAN OBJECTIVES

The objectives of the RFI Work Plan are to:

- characterize the nature and extent of any potential releases of hazardous waste and/or hazardous constituents in environmental media at the Facility;
- assess potential risk to human health and the environment associated with any potential releases of hazardous waste and/or hazardous constituents;
- determine whether interim corrective measures are necessary to control current unacceptable risks, if any, to human health; and
- determine whether a corrective measures evaluation is necessary to mitigate all current and future unacceptable risks, if any, to human health and the environment.

1.3 WORK PLAN ORGANIZATION

The RFI Work Plan is consistent with U.S. EPA and MDEQ-WMD guidance. The RFI Work Plan is organized as follows:

- Section 1.0 - Introduction: This presents an introduction to, and organization of, the RFI Work Plan.
- Section 2.0 - Description of Facility: This section summarizes general conditions at the Facility, including the Facility location, historical property ownership, current and historical operations at the Facility, and current and historical waste management practices at the Facility.
- Section 3.0 - Regional Setting: This section summarizes local land use, demographics, climate, regional geology, regional hydrogeology, and regional hydrology.
- Section 4.0 - Facility Setting: This section summarizes the geology, hydrogeology, and hydrology at the Facility. In addition, this section presents the local topography of the Facility.
- Section 5.0 - RFI Scope of Work: 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 data analysis, review of exposure information, and post-investigation evaluations.

- Section 7.0 - Reporting: This section outlines the reporting requirements associated with the RFI, including the RFI Report.
- Section 8.0 - RFI Schedule: This section presents the RFI schedule.
- Section 9.0 - References: This section provides references for the documents cited in this RFI Work Plan. The background information in this RFI Work Plan has been taken from the Description of Current Conditions Report (CRA, March 31, 2000), and therefore the reference list from that report has not been reproduced.

This document also includes the following appendices:

- Appendix A - Historical Soil Boring and Monitoring Well Logs: Appendix A presents soil boring and monitoring well logs from previous investigations. Stratigraphic and well completion logs for the existing monitoring well network are also included.
- Appendix B - Project Management Plan (PMP): The PMP presents a discussion of the project team organization and the responsibilities of the project team members. The qualifications of personnel performing or directing the RFI are also included.
- Appendix C - Quality Assurance Project Plan (QAPP): The QAPP 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 corrective measure (as necessary).
- Appendix G - Field Sampling Plan (FSP): The FSP presents procedures for the collection of surface soil, subsurface soil, and groundwater samples (if required).
- Appendix H - Historical Analytical Results: Appendix H presents relevant historical analytical results for each AOI that will be investigated further as part of the RFI (as available).

1.4 INVESTIGATION SUMMARY REQUIREMENTS

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

2.0 DESCRIPTION OF FACILITY

2.1 FACILITY LOCATION

The Facility is located north of the City of Flint, in Genesee Township, Genesee County, Michigan at 43° 45' north latitude and 83° 05' west longitude. The Facility is located on approximately 118.7 acres of land at G1245 East Coldwater Road. The general geographic location of the Facility is shown on Figure 1.1. Manufacturing has historically taken place in the approximately two million square foot Building 44 and associated buildings at the Facility. The Facility is bordered to the north by an approved RCRA Part B permitted wastewater treatment sludge mono-fill landfill and a restored wetlands area owned and maintained by REALM (which is part of the original plant property owned by GM); to the east by CSX Transportation property and railroad tracks; to the south by East Coldwater Road; and to the west by Horton Street. The Facility boundaries, buildings, and support facilities are presented on Figure 1.2/Plan 1 (Plan 1 is located in the sleeve at the back of the RFI Work Plan).

2.2 PROPERTY OWNERSHIP HISTORY

GM acquired the property that the Former Peregrine Facility is located on as vacant land in 1951 from several private parties. The Buick Motor Division started construction of the plant buildings that same year. However, prior to completion, the Facility was transferred to the Ternstedt Division. The Fisher Body Division subsequently acquired the Facility in 1968. In 1985, the Fisher Body hardware plants were merged with Guide Lamp and became the Fisher Guide Division, and then in early 1989, Fisher Guide Division was merged with Inland Division to become the Inland Fisher Guide Division. The Inland Fisher Guide Division became part of Delphi Automotive Systems in early 1995 and the Facility was operated as the Delphi Interior and Lighting Systems Division Coldwater Road Plant until GM sold the Facility to Peregrine in December 1996. Peregrine ceased operations in 1998 and GM's subsidiary REALM acquired the property in August 1999. REALM is currently undertaking environmental decommissioning of the above grade structures and preparing for demolition of the industrial buildings.

2.3 CURRENT AND HISTORICAL OPERATIONS

2.3.1 CURRENT OPERATIONS

There are no industrial operations currently being conducted at the Facility. The Facility is undergoing a complete and systematic environmental decommissioning and demolition program that began during the summer of 1999 (after REALM acquired title to the Facility) and is expected to be completed in 2001. The following provides a summary of associated activities and the general time frames during which they were/will be implemented:

- Building Decommissioning Assessment (BDA) – Summer 1999;
- Collection /Removal of Chemical Containers – August/September 1999;
- Collection/Removal of Transformers and Capacitors – September-November 1999;
- Collection/Removal of Non-Structural ACM Containing Materials - December 1999 – March 2000;
- Aboveground Environmental Decommissioning – January-September 2000; and
- Building Demolition - 2000-2001.

Mobilization to the Site of a construction trailer and personnel by REALM began in August 1999. REALM is managing the Site and maintaining security through 24-hour surveillance by a contracted security firm. The Administration Building (Building 44A) has been leased to GM as a "jobs bank" personnel transition center, which is anticipated to remain after industrial portions of the buildings have been decommissioned and demolished. REALM contracted the removal of transformers and capacitors to SunOhio, Inc. during the fall of 1999. This activity has been completed. National Abatement Corporation was retained to perform abatement of non-structural asbestos (not including roofing materials and galbestos siding). Asbestos abatement began on December 8, 1999 and was completed in March 2000. Removal of the light fixtures, chillers, and miscellaneous fixtures was contracted to the MPS Group in March 2000 and is expected to be completed in May 2000. Removal of the floor block was contracted to Inland Water Pollution Control, Inc. in March 2000 and was completed in April 2000. Final cleaning of the Facility structures began in May 2000. Building demolition could begin as early as June 2000.

2.3.2 HISTORICAL INDUSTRIAL OPERATIONS (1995-1999)

The Facility was most recently used by Delphi and Peregrine (past four to five years) for the manufacture of the following automobile components:

1) window regulators;
2) door hinges;
3) door modules; and
4) seat adjusters.

The latter two processes were discontinued upon transfer of the Facility to Peregrine in December 1996. Manufacturing operations occurred in the main manufacturing building (Building 44) at the locations summarized on Figures 2.1 and 2.2 (Peregrine and Delphi, respectively). Descriptions of the manufacturing processes associated with each of the above components are summarized below:

1) **Window Regulators:** Window regulators are the mechanical devices which raise and lower the vehicle's windows. The Facility produced conventional and tape-drive regulators for W-Body and J-Body vehicles. Window regulators are comprised of both fabricated metal and motorized mechanical components. The fabricated metal components were manufactured in-house, while the motorized mechanical components were supplied by outside vendors. The in-house manufacturing processes included unrolling/straightening rolls of steel, cutting and shaping, stamping, riveting, assembly, and washing. Depending on customer specifications, certain regulator assemblies were shipped off-site to be zinc plated.

2) **Door Hinges:** The Facility produced both front and rear door hinge assemblies for W-Body and J-Body vehicles, as well as N-Body deck lift hinges. Door hinge assemblies are essentially comprised of three major components: two steel hinge joint components and a spring assembly. The two hinge joint components were manufactured in-house, while the springs were supplied by outside vendors. The in-house manufacturing processes included unrolling/straightening sheets of steel, cutting and shaping to size, stamping, assembly (both automated and hand), washing, and welding.

3) **Door Modules:** The Facility produced door modules for G-Body, K-Body, and K_{sp}-Body vehicles. Door modules are the formed steel backing plates that line

the interior of automobile doors. The door module essentially consists of a single steel plate. The entire component was manufactured in-house. The in-house manufacturing process included unrolling/straightening sheets of steel, cutting and shaping to size, stamping, and washing. This process was discontinued upon transfer of manufacturing operations to Peregrine in December 1996.

4) **Seat Adjusters:** Seat adjusters are the mechanical devices which allow adjustment of the passenger and driver seats. Seat adjuster operations at this Facility consisted of assembly operations only. All adjuster components were manufactured and supplied by outside sources. This process was moved to another Facility prior to transfer of operations to Peregrine or discontinued upon transfer of manufacturing operations to Peregrine in December 1996.

5) **Other Operations:** Some component parts were heat treated in the presence of barium salts to harden the steel. No machining, painting, heat treating, or plating was reported to take place in the Facility after Peregrine commenced operations on January 2, 1997.

Other operations at the Facility (Delphi and/or Peregrine) utilized a variety of chemicals, including oils, greases, solvents, water treatment chemicals, minor amounts of paint, and barium salts for heat treating. Wastewaters generated by Delphi during washing activities were treated on-Site prior to discharge to the local publicly owned treatment works (POTW). On-site Wastewater Treatment Plant (WWTP) operations were discontinued at the time of the property transfer to Peregrine in December 1996 and the WWTP was subsequently demolished by GM. Wastewaters generated by Peregrine during its parts stamping and manufacturing operations were subsequently managed through Wastewater Storage Tanks formerly located in the northwest corner of the main manufacturing building (Building 44). These tanks were periodically emptied by a contracted waste hauler for off-Site treatment and disposal.

2.3.3 HISTORICAL OPERATIONS (PRIOR TO 1995)

Prior to the manufacture of automobile components by Delphi and Peregrine, historical activities at the Facility consisted of a variety of other automotive parts related operations. The most significant of these historical operations was the former plating operations, for which there were reported to be 13 chrome plating lines and 2 zinc barrel plating lines. The generalized location of the former platers are presented on Figure 2.3 (based upon historical drawings that are available for the Facility). Other known

historical processes included die casting, solvent-based adhesive/gluing and painting operations. Descriptions of the pre-1995 historical operations at the Facility are summarized below:

1) **Plating Operations:** There are reported to have been 15 plating lines (see Figure 2.3) in operation at some point in time during the almost 30 years of manufacturing by the Fisher Body Division and two subsequent merged divisions. The following list identifies some of the chrome platers, and their approximate removal dates:

   - The #9 Chrome Plater was located between Columns D and G and Columns 23 and 24 and was removed in 1983.
   - Two chrome platers located between Columns H and M and Columns 31 and 33 were removed in 1983.
   - The #10 Chrome Plater was located between Columns D and G and Columns 22 and 23 and was removed in 1973.
   - The #5 Chrome Plater was located at Column K-25 and was removed in 1971.
   - Two other chrome platers, located between Columns E and G and Columns 33 and 35, were removed in 1971.
   - Two other chrome platers, located at Columns K-34 & K-35, were removed in 1969 and 1973.
   - A half-size automated chrome plater located at Column L-30 was removed in 1969
   - A manual still plater (with hoist) located at Column J-22, as well as a small still plater known as the North Still, were removed in 1967.

   The above plating lines had associated sumps and an associated series of aboveground storage tanks (ASTs) in the basement of the main manufacturing building which made up the Plating Solution Filtration/Treatment Area. The filtration and treatment area allowed the plating solutions to be processed for re-use.

   There were also two zinc plating lines in operation concurrently with many of the chrome platers. One zinc plating unit was located near Column K-8 and was removed in 1979. An inactive zinc barrel plating unit which was located between Columns K-1 and K-2 was removed by GM in 1997. The locations of the platers are presented on Figure 2.3.

In addition, die casting operations were historically conducted at the Facility within the northeast quadrant of Building 44 (see Figure 2.3).

2) **Headliner Operations:** The headliner adhesive and chemical aboveground storage tanks are located east of the main manufacturing building and remain on-Site. There were eight stainless steel storage tanks (Tank #6 has been removed), approximately 8,000 gallons each, associated with the former headliner activities. These tanks historically contained solvent-based adhesives. More recently, the tanks contained water based adhesives.

3) **Plastisol Related Activities:** A former underground storage (UST), located east of the main manufacturing building (near Column S-31), was reportedly used for plastisol related activities between approximately 1953 and 1984. Plastisol was a rubber-type coating placed on the plating racks in order to separate the racks themselves from the plating operation. The Plastisol UST was simply used as temporary storage whenever the interior dip tank required cleaning. The tank was removed and closed by GM in 1997.

4) **Underground Storage Tanks:** There are no known USTs currently present at the Facility. Based on available information, all former USTs have been removed. Two 2,000-gallon gasoline and two 2,000-gallon waste oil tanks were removed in November 1990 and a letter of closure was received from the Michigan Department of Natural Resources (MDNR). A 1952 design drawing of the Facility identified a tank farm located near the current electrical substation area. However, based upon interviews with former Delphi personnel, no tanks were ever installed at this location (due to a change in projected use of the Facility).

5) **Other Operations:** It is believed that some metal parts were historically degreased with chlorinated solvents within the western portion of the former tool room.

2.4 CURRENT AND HISTORICAL WASTE MANAGEMENT PRACTICES AND REGULATORY HISTORY

Wastes were generated at the Facility since the start of production in 1952. These wastes were primarily generated in plating operations, press room operations, and maintenance operations within Building 44 and handled by the former WWTP and lagoons. The Facility's non-production support areas (e.g., powerhouse) also generated some wastes. The waste management activities associated with the operation of the Facility are

summarized in this section, including the Facility's waste generation, treatment, storage, and disposal practices.

Building 66 contained the Peregrine 90 day Hazardous Materials Storage Area. Hazardous waste is no longer stored at this location, as operations at the Facility ceased in late 1998, at which time the Hazardous Materials Storage Area was demolished. No stored hazardous waste related to manufacturing activities exists at the Facility. The Building 66 Hazardous Materials Storage Area (AOI 5) is discussed further in Section 7.5 of the DOCC. The Former Drum Storage Area (AOI 1), a permitted waste storage area for hazardous waste, ceased operation in September 1994 and is discussed further in Section 7.1 of the DOCC.

2.4.1 WASTE GENERATION

1) **Former Plating Operations:** Plating operations historically took place in Building 44 from approximately 1953 through 1987. There were 13 chrome plating lines and 2 zinc plating lines. A number of these plating lines had collection sumps associated with them. Spent plating solutions were generated which variously contained chrome, zinc, copper, acid, alkali, and cyanide. Plating solutions were recycled through the plating system which included a variety of solution regeneration activities which occurred at the filtration and treatment area located in the basement. Spent plating solutions were pumped to the WWTP for treatment. The resultant wastewater treatment plant sludges were stored in associated sludge drying beds. The sludge drying beds were active until June 1987 when cessation of plating operations took place, and discharge from the WWTP was re-routed to the sanitary sewer system. The Former Plating Areas (AOI 9) are discussed in Section 7.9 of the DOCC. As part of the closure activities of the drying beds, a permitted monofill landfill was constructed and closed. The lagoons, drying beds, and landfill were closed under a separate State of Michigan lead regulatory program and that no further corrective action is required for the landfill (other than ongoing operation and maintenance activities).

2) **Former Press Room Operations:** The metal stamping operations generated wastewater containing oil, grease, suspended solids, and scrap metal. The metal drawing lubricants used in more recent years were made from saturated animal fats rather than petroleum based lubricating oils.

3) **Former Maintenance Operations:** Maintenance cleaning operations historically generated spent solvents from parts washers. The Maintenance Paint Booth and small part marking operations generated waste paint and paint related materials. The Maintenance Paint Booth had not been used in recent years (Peregrine/Delphi time frame). Other solid wastes from maintenance operations included batteries, fluorescent lights, and light ballasts.

4) **Support Areas:** Other wastes were generated by the Facility powerhouse, including boiler blowdown and fly ash. The PA/VSI identified the Former Coal Storage Area as an AOC (AOI 11). According to file material and Facility personnel, the Former Coal Storage Area was unlined.

5) **Other:** The Former Coal Storage Area (AOI 11) is discussed further in Section 7.11 of the DOCC. Another AOC identified by U.S. EPA in the PA/VSI was the 11 former empty caustic tanks (AOI 12) staged immediately west of the Former Drum Storage Area (AOI 1) adjacent to the north side of Building 44. The caustic was used to clean sheet metal prior to coating. The related coating operations took place on the second floor of Building 44. The Former Caustic Tanks (AOI 12) are discussed further in Section 7.12 of the DOCC.

Peregrine submitted a Notification of Regulated Waste Activity to MDEQ, dated December 23, 1996, prior to acquiring the former Delphi Facility. Peregrine identified their waste streams as being D001, D002, D006, D007, D008, D009, D018, D039, D040, F005, F006, U070, 003D, and 039L in a letter to MDEQ dated January 23, 1997. An additional notification was submitted on April 11, 1997 to supply the additional waste streams D011, D019, F001, P098, P104, 006L, and 021L.

2.4.2 WASTE STORAGE

Historically, metal scrap and waste metal drawing lubricant generated during the parts stamping operations in the press room were collected in a below grade system consisting of a series of conveyor trench systems and sumps. These materials were then recycled through oil filters. Wastes from the oil filters were conveyed to the WWTP through the Overhead Waste Pipes (AOI 10). This system was located adjacent to the parts stamping machines. The conveyor systems were used to transport the scrap metal and waste lubricant to one of the 11 collection locations in Building 44. These conveyor systems and sumps in Building 44 are collectively referred to as the Former Scrap Metal Management System (AOI 7) and are discussed further in Section 7.7 of the DOCC.

During Peregrine operations, waste metal drawing lubricant from the collection pits, together with wastewater and runoff from the floor drains, was pumped through Overhead Waste Pipes (AOI 10) to temporary storage in the Former Wastewater Storage Tanks (AOI 6) located in the northwest corner of Building 44 (see Section 7.6 and 7.10 of the DOCC). Scrap metal collected in the sumps was transported by carts to the Former Scrap Metal Storage Area (AOI 8) located in Building 63. The Former Scrap Metal Storage Area is discussed further in Section 7.8 of the DOCC. During historical operations at the Facility, some of the waste lubricating oils generated from operations were stored in two 2,000-gallon USTs which were located outside the east wall of Building 44 (see Figure 1.2). The former USTs (AOI 3) are discussed further in Section 7.3 of the DOCC.

Former Satellite Accumulation Areas (AOI 4) were located at several points in Building 44 for temporary storage of wastes. The wastes included the following:

- non-hazardous waste including plant trash;
- used aerosol paint cans and used batteries in the General Stores area, located in the basement of Building 44,
- waste paint and related materials in the Maintenance Paint Booth,
- spent heat furnace salt generated in the Former Tool Room,
- accumulation of emergency light batteries and polychlorinated biphenyls (PCB)/non-PCB lighting ballasts in the Electric Repair Shop, and
- accumulated spent solvent in the Machine Repair Shop.

The Former Satellite Accumulation Areas are discussed further in Section 7.4 of the DOCC. All of the hazardous wastes from the Former Satellite Accumulation Areas were transported to the Hazardous Materials Storage Area (AOI 5) prior to off-Site treatment or disposal. The Hazardous Materials Storage Area, which is discussed further in Section 7.5 of the DOCC, was operated as a less than 90-day generator storage area.

The Former Waste Pile Pad (AOI 2) is located north of Building 63 and was used for sorting non-hazardous solid wastes, such as trash from bins from various locations on the Facility and from trash cans in the parking lot. A trench drain collected spills and rainwater from the Former Waste Pile Pad which drained to the Former Wastewater Storage Tanks. The Former Waste Pile Pad is discussed further in Section 7.2 of the DOCC.

2.4.3 WASTE DISPOSAL

The following is a summary of the Facility's waste disposal practices based on a review of available file information and information provided by past Facility employees.

Spent plating solutions generated in Building 44 were treated in the former WWTP located immediately to the north of the Facility. The subsequent sludges were deposited in adjacent drying beds and subsequently landfilled. The landfilled areas located to the north of the Facility are regulated under an approved and completed RCRA Closure Plan and Consent Order. Other than the sludges from the treatment of spent plating solutions, all other wastes were sent off Site to licensed facilities for treatment or disposal.

2.4.4 PAST WASTE SPILLS

The Emergency Response Notification System (ERNS) database identified spill notifications during the years 1987-1999. The following summary is believed to be a comprehensive listing for the Facility for the period from 1987 to 1999:

Spill Date	*Company Name (As listed in Database)*	*Chemical/Compound of Concern*	*Estimated Quantity Released*
10/31/90	GM - Inland Fisher Guide Division	Metal Working oil	100 gallons
06/13/90	GM - Inland Fisher Guide Division	Water soluble oil discharged into Hughes Drain	400 gallons
06/27/89	GM - Inland Fisher Guide Division	Water soluble oil discharged into Hughes Drain	Estimated at 1,200 gallons

REALM is currently conducting a supplemental review of these spills and will supplement the DOCC and RFI Work Plan, as required.

2.4.5 REGULATORY HISTORY

GM filed a Notification of Hazardous Waste Activity Form for the Inland Fisher Guide Coldwater Road Facility with the U.S. EPA on August 12, 1980, pursuant to Section 3010 of RCRA, wherein GM identified itself as a generator of hazardous waste, and an owner/operator of a treatment, storage, and disposal facility. The U.S. EPA Identification (ID) Number which had been used for the entire property including the adjoining landfill was MID005356860. Currently, this identification number only applies to the landfill portion. GM filed a Part A Hazardous Waste Permit Application for the Facility on November 19, 1980, and amended it on August 10, 1984. The treatment, storage, and disposal operations were subject to the interim status requirements of R299.9601 and 40 CFR Part 265. A Closure Plan addressing these areas was subsequently submitted to the Michigan Department of Natural Resources (MDNR) in September 1988. A report entitled "Closure Plan and Post-Closure Plan (Volumes I and II), August 1989, O'Brien and Gere Engineers, Inc." was submitted to the MDNR, (now MDEQ) and approved in accordance with 40 CFR Part 265.112(d)(4) on September 7, 1989 (pursuant to a Consent Order). The only areas of the Facility included in this DOCC Report that was part of the referenced Closure Plan are the Former Drum Storage Area (AOI 1) and Former Waste Pile Pad (AOI 2). REALM has submitted a Work Plan to complete the additional closure sampling of the Former Drum Storage Area as requested by MDEQ. The current identification number for the Former Peregrine Facility is MIR000020743.

The Facility currently has an active Storm Water General Permit Certificate of Coverage No. MIR11L010, as issued to Peregrine, which has been requested for transfer to REALM. This storm water permit replaced GM's National Pollutant Discharge Elimination System (NPDES) Permit No. M10025194 that was terminated on November 15, 1995.

The Facility has a Sewer Use Permit for sanitary discharge to the WWTP owned by the City of Flint. Under this permit, the Facility has two municipal water sanitary service accounts: 60-52-3791-007 and 60-52-3840-040.

Finally, the Facility has a "Renewable Operating Permit" for air. It should be noted, however, that there are no current operations occurring at the Facility with air emissions.

3.0 REGIONAL SETTING

3.1 LAND USE

The Facility is bordered on the north by REALM property (i.e., the closed landfill) on the south by East Coldwater Road, on the west by Horton Street, and on the east by CSX Transportation property and railroad tracks. The Facility is currently zoned and was historically utilized for industrial activities since 1952.

The property to the north contains the permitted wastewater treatment sludge mono-fill landfill, former WWTP (demolished by GM in 1999), and a restored wetlands area owned and maintained by REALM. Stanley Road runs east-west immediately north of this adjacent REALM property.

The property immediately south of East Coldwater Road is a mixture of light industrial, commercial, and residential property.

The property to the west across Horton Street is primarily residential. In addition, Interstate Highway I-475 curves through the area west of Horton Street from an east-west path to the north-south direction.

The property to the east of the Facility contains a railroad right-of-way that is the property of CSX Transportation. The railway tracks within this right-of-way run in a north northwest/south southeast direction.

3.2 WATER SUPPLY

The water supply at the Facility has been provided by the City of Flint and the Beecher Metropolitan District System. The City of Flint provides water service to the former manufacturing area (Building 44) and Beecher Metropolitan District provides water services to the Administration Building (Building 44A).

3.3 DEMOGRAPHICS

The Facility is located in the Genesee Township, Genesee County, Michigan. The closest and largest municipality is the City of Flint located directly south and west of the

Facility. The City of Flint has a population of approximately 134,800 with a median age of 31.2 years for men and 33.3 years for women (Flint Chamber of Commerce - www.accessflint.com). The population racial mix is 49.6 percent white, 47.9 percent black, 0.5 percent native American, 0.5 percent Asian, and 1.4 percent other races. The City of Flint has 2.7 percent of the population of Hispanic origin (U.S. Bureau of Census). The Urbanized Area of Flint has a population of 326,452 and the Genesee County population is 430,459 (U.S. Bureau of Census). Genesee County has several businesses directly and indirectly tied to the automotive industry. According to the U.S. Bureau of Census, GM is the largest employer in the area. Other major areas of employment include education, health care, banking, and retail.

3.4 CLIMATE

Lake Michigan and Lake Huron have a somewhat moderating effect on the climate of Genesee County. Meteorological data from the National Climatic Data Center are summarized as follows:

Temperature (°F)	*January*	*July*
Normal Daily Maximum	28.7	81.5
Normal Daily Mean	21.5	70.6
Normal Daily Minimum	14.2	59.7

The record high and low temperatures are 101°F and -25°F, respectively.

Precipitation (Inches)	*January*	*July*
Normal	1.39	2.71

Total annual precipitation is 30.28 inches. Average snowfall is 45.0 inches per year.

Wind Speed (MPH)	*January*	*July*
Average	11.8	8.3
Maximum	45.0	41.0

Annual average wind speed is 10.2 miles per hour (mph) and the highest measured wind was 81 mph. The prevailing wind direction is from the west.

3.5 REGIONAL GEOLOGY

3.5.1 OVERBURDEN

The bedrock of Genesee County is covered with glacial deposits. These glacial materials generally thicken from the northwest corner to the southeast corner of the County. Glacial deposits in the northwest corner range between 100 and 150 feet, and increase to 150 to 200 feet in thickness toward the middle of the County. In some small areas in the southeast corner of the County, the glacial cover ranges between 200 and 400 feet in thickness.

Geologic evidence south of Genesee County indicates four major advances of glacial ice in the past two million years (during the Pleistocene period). These glacial advances and the interglacial periods between them left complex deposits of till and stratified drift. Because each subsequent advance reworked deposits from previous advances, and because the deposits were derived from the same source, it is difficult to distinguish deposits from each separate advance.

The Huron-Erie and Saginaw lobes of the most recent Wisconsinan glaciation carried clay, silt, sand, gravel, and boulders into what is now Genesee County. These materials are deposited in many forms such as till plains, lake plains, moraines, outwash, beaches, bars, and deltas.

The surficial deposits in the vicinity of the Facility consist of urban land and soils from the Conover-Brookston and Celina-Conover-Miami, based on information contained in the Soil Survey of Genesee County, Michigan prepared by the United States Department of Agriculture and the Soil Conservation Service. In general, these soil associations are characterized as level to gently sloping, somewhat poorly drained to well drained loams, that have a clay loam subsoil. In combination, the Conover-Brookston and Celina-Conover-Miami soil associations occupy 56 percent of the surficial soils throughout Genesee County.

Figure 3.1 presents the Regional Glacial Drift Surface Deposition information for the area around Flint. Figure 3.2 presents the Regional Glacial Drift Thickness information for the same area.

3.5.2 BEDROCK

The bedrock of Genesee County is composed of evaporites, carbonates, and clastics that are Paleozoic in age, more specifically, Mississippian and Pennsylvanian. These carbonates, clastics, and evaporates were formed in the Michigan Basin which had developed by late Silurian time.

Three specific bedrock formations can be found beneath the glacial overburden of Genesee County. The lowest unit is the Marshall Formation, which underlies the whole of Genesee County. This formation is Mississippian in age and ranges in thickness from 100 to 150 feet. The Marshall Formation is fine to coarsely grained, gray and white sandstone with some conglomerate, shale, and dolomite beds.

The Michigan Formation (Mississippian) lies on top of the Marshall Formation, and is between 50 and 210 feet thick. The Michigan Formation is predominantly composed of gray shale with some blue or green shale, and intermittent thick beds of gypsum and dolomite.

The uppermost bedrock formation in Genesee County is the Saginaw Formation (Pennsylvanian). The Saginaw Formation is composed of very fine to coarse, sub-rounded to rounded sandstone and is light gray or brownish gray to grayish brown in color. Crossbedding can often be seen in the Saginaw Formation sandstone. If crossbedding is observed, it is common to see alternating bands of lighter coarse-grained quartz and darker fine-grained quartz. Sandy shale, shale, coal, and limestone are also found in the Saginaw Formation. The shale is frequently thinly bedded, fissile, micaceous, firm to very hard, and gray to dark gray in color.

Figure 3.3 presents the Regional Bedrock Geology information for the area around Flint. Figure 3.4 presents the Regional Bedrock Topography information for the same area.

3.6 REGIONAL HYDROGEOLOGY

Both the glacial overburden and the bedrock in Genesee County have water bearing geologic units. The glacial overburden consists of mainly low permeability tills that are poor sources of groundwater, but some lenses of sand and gravel may provide adequate volumes of water for domestic use. The sandstone beds of the Saginaw and Marshall Formations are the best and most commonly used water bearing hydrogeologic units in Genesee County. This is because of the high yields that can be obtained and because bedrock wells in these formations are well protected by overlying clays and are easily maintained.

Rainfall and subsequent infiltration is the primary source of recharge for the water bearing units in the glacial overburden. These water bearing units are generally hydraulically connected to the surface waters in Genesee County. As a result, dry weather flow for most streams and rivers is supported by groundwater discharge. Further, water levels and groundwater flow directions in the shallow glacial water bearing units are influenced by local drainage areas such as lakes, creeks, and rivers, which act as points of discharge.

The glacial drift water bearing units may also be locally connected hydraulically to the bedrock water bearing zones. Rainfall is the main source of recharge and the upland areas in the southern part of the County act as a regional recharge source. The flow direction in the bedrock conforms to a regional pattern and generally moves from recharge areas toward the Flint River, which acts as a general point of groundwater discharge.

The types of material in Genesee County, and their formation are relevant to the potential for aquifer contamination. According to the Hydrogeologic Atlas of Michigan map "Aquifer Vulnerability to Surface Soil Contamination in Michigan", dated 1982, the majority of Genesee County contains moderately or slowly permeable soils over low sensitivity drift material. The Hydrogeologic Atlas of Michigan map "Quaternary Geology of Southern Michigan (upper 1-10 meters)", dated 1982, shows that these low permeability soils are predominantly comprised of medium textured tills from end moraines or till plains, lacustrine clays, and lacustrine silts. As a result, groundwater in glacial overburden is generally of limited extent and usability. In many locations, groundwater may be considered to be present as "groundwater not in an aquifer".

Some unconsolidated water bearing units along the Flint River and in the northwest corner of Genesee County are more vulnerable to contamination according to the Hydrogeologic Atlas of Michigan aquifer vulnerability map. The Hydrogeologic Atlas of Michigan quaternary geology map indicates that these areas are primarily glacial outwash sands and gravels, postglacial alluvium, or lacustrine sands and gravels. These units would generally be more capable of producing significant yields of groundwater.

Figure 3.5 presents the Regional Glacial Drift Aquifer Characterization information for the area around Flint. Figure 3.6 presents the Regional Groundwater Flow directions for lower peninsula of Michigan.

3.7 REGIONAL HYDROLOGY

The Flint River runs approximately forty miles across Genesee County and drains the majority of the County. The southern halves of Argentine Township and Fenton Township drain to the Shiawassee River. The average stream flows recorded at the Flint River gaging station near Flint was 362 million gallons per day (mgd) and 231 mgd at the Flint River gaging station near Genesee.

The Flint River has seven main tributaries: Swartz Creek, Thread River, Kearsley Creek, Butternut Creek, Brent Run, Pine Run, and Armstrong Creek. The Misteguay drainage system and Swartz Creek drain the southwest portion of Genesee County. The Thread River and Kearsley Creek drain the southeast portion of the County. The northeast portion of the County is drained by Butternut Creek. Armstrong Creek, Pine Run, and Brent Run drain the western areas of the County.

Figure 3.7 presents the Regional River Drainage Basins for the area around Flint.

4.0 FACILITY SETTING

4.1 LOCAL TOPOGRAPHY

The local topography of the Facility is presented on Figure 1.1. The Facility is located on a terminal moraine which is a local topographic high in an area that is generally flat. There are no rivers, streams, surface water bodies, or creeks located within the Facility's property boundaries. Surface water drainage at the Facility is toward a low area, which occupies the northwest corner of the adjacent REALM property, to the north of the Facility.

4.2 GEOLOGY

The subsurface geology in the vicinity of the Facility consists of unconsolidated Pleistocene glacial sediments overlying sandstone bedrock. These sediments consist primarily of a series of tills (clay/silt/sand/gravel mixture), with occasional intervening outwash layers. The outwash deposits are more permeable silt and sand layers of limited thickness and lateral extent within the clay till unit. Boring logs from investigations on the adjacent landfill property to the north of the Facility indicate that the near surface material is generally comprised of silty sand and silty clay underlain by a relatively impervious clay layer which is up to 50 feet thick. This clay is underlain by a sand/silty sand outwash layer. Below this unit is another impervious clay till layer, approximately 30 to 75 feet in thickness, which directly overlies bedrock. The bedrock is part of the Saginaw Formation of Pennsylvania age, which consists primarily of sandstone, shale, coal, and limestone.

4.3 HYDROGEOLOGY

The overburden glacial deposits contain one potential water bearing zone (overburden water bearing zone), as well as a discontinuous perched water table zone found near the ground surface (upper 10 to 15 feet). These two zones are separated from each other and from the upper bedrock water bearing zone by thick glacial clay till aquitards. The overburden water bearing zone consists of a sand/silty sand unit that is located below a clay aquitard at a depth of approximately 50 feet below ground surface (bgs). This unit rests on a continuous unit of silty clay glacial till which extends to bedrock. Figure 4.1 presents a typical geologic cross-section for the area beneath the Facility.

Vertical water movement from the perched water table zone and overburden water bearing zone is severely restricted by the underlying glacial clay till aquitards. Differences in the static water levels between the perched water table zone and the overburden water bearing zone confirm that the intervening clay till unit serves as an effective aquitard.

Figure 4.2 presents the locations of the boreholes and monitoring wells which have been installed at the Facility. A summary of completion details for the boreholes and monitoring wells is presented in Table 4.1. Figure 4.2 also presents the locations of cross-sections A-A' and B-B'. Cross-sections A-A' and B-B' are presented on Figures 4.3 and 4.4, respectively. Figures 4.3 and 4.4 indicate that a discontinuous perched water layer was found at only some borehole and monitoring well locations near surface. In general, clay was found at a very shallow depth.

The perched water table flow direction (if present) is controlled by topography. Available water levels collected within this zone are presented on Figure 4.5. The overburden water bearing zone is protected from any potential impacts within the discontinuous perched water table zone by the glacial clay till aquitard which exists beneath the surface soils. Groundwater flow in the overburden water bearing zone is expected to be westerly or northerly (TechLaw, 1998). The regional groundwater flow direction within the bedrock is discussed in Section 3.6.

Available borehole and monitoring well logs (see Appendix A) indicate domestic wells in the area are completed in the overburden water bearing zone (between the two clay till aquitards) and in the upper bedrock water bearing zone.

4.3.1 GROUNDWATER USE

With the exception of some private wells, the Beecher Metropolitan District provides the water supply for the City of Flint from five well fields. Four of the well fields are completed within the overburden water bearing zone at depths of approximately 100 to 140 feet bgs and are located approximately one mile north of the Facility. The fifth well field is completed within the bedrock aquifer at a depth of 275 to 300 feet bgs.

The nearest active residential or domestic wells are located on Stanley Road approximately two-thirds of a mile north of the Facility. Perched water table flow

beneath the Facility (see Figure 4.5) is discontinuous. The domestic wells are completed in the overburden water bearing zone (between the two clay till aquitards) and in the upper bedrock water bearing zone. Sampling by the Genesee County Health Department found the Stanley Road wells to be uncontaminated in 1990/1991.

4.4 HYDROLOGY

The nearest surface water body is Mott Lake, approximately two miles east of the Facility. Mott Lake was created in 1971 with the construction of a 30-foot high dam across the Flint River. The 500-acre lake is used solely for recreational activities, such as fishing, boating, and swimming.

5.0 RFI SCOPE OF WORK

The Scope of Work for the RFI is comprised of the following tasks:

Task 1: Topographic Survey
Task 2: Groundwater Use Inventory
Task 3: Phase I Characterization of Areas of Interest
Task 4: RFI Reporting

The purpose of sampling activities will be to determine whether a significant release of hazardous waste and/or hazardous constituents has occurred at certain AOIs and, if so, to characterize the exposure concentration so that risk-based decisions on the need for corrective measures can be made. Sampling activities will generally be completed as outlined below and in Tasks 1 through 4, although methods will be varied as necessary to accommodate the specific features of each AOI.

The sampling strategy for the RFI is based upon the significant knowledge and database which has been developed for the Facility through previous investigations. The investigations were conducted at the Facility as part of the property transfer from Delphi to Peregrine in December 1996. These investigations have provided information regarding the SWMUs and AOCs identified by TechLaw during the subsequent PA/VSI as well as other AOIs identified in the DOCC Report (March 31, 2000) by REALM. Additional details from these previous environmental investigations are included with the discussion of each AOI presented in Sections 7.0 and 8.0 of the DOCC report. The analytical results from the previous investigations are presented in Appendix H.

The strategy for the RFI investigation is also based on the current approaches being utilized by MDEQ-WMD related to corrective actions at industrial properties. The pertinent constraints which guide the appropriate application of the Act 451, Part 201 industrial standards are as follows:

1. the Facility is zoned and utilized for industrial purposes;
2. appropriate security measures are currently in place to restrict Facility access;
3. future land use plans contemplate the continued industrial use of the Facility;
4. the Facility is serviced by a municipal water supply;

5. the overburden and bedrock water bearing zones, which are locally used as water supplies off Site, are protected by significant overlying confining clay till aquitards; and

6. the perched water table zone is shallow (<25 feet bgs) and discontinuous and would be generally prohibited from use as a potable water (regardless of potential yield) supply by Michigan Department of Public Health (MDPH) rules.

Based upon the factors summarized above, the Act 451, Part 201 generic industrial direct contact criteria for both soil (IDC) and the perched water table zone groundwater (GCC) are applicable for use as screening levels for the Facility. A more Facility-specific (or AOI-specific) risk based (i.e., limited industrial) approach may be utilized in the future to more accurately identify potential risks which may exist at the Facility (as required).

The data collected during four studies conducted during 1996 and 1997 are summarized in Tables H.1 through H.5 (for soil, groundwater, TCLP, PCB, and wipe results, respectively) and compared to current Act 451, Part 201 generic industrial standards. The generic industrial criteria used for comparison of the data in Tables H.1-H.5 are summarized as follows:

Soil

1. Statewide Default Background Levels
2. Industrial Drinking Water Protection Criteria (IDWP)
3. Industrial Direct Contact Criteria (IDC)
4. Soil Volatilization to Indoor Air Inhalation Criteria
5. Infinite Source Soil Inhalation Criteria
6. Particulate Soil Inhalation Criteria

Water

1. Groundwater Contact Criteria (GCC)
2. Industrial Drinking Water Criteria (IDWC)
3. Industrial Groundwater Volatilization to Indoor Air Inhalation Criteria

A review of the data presented in Appendix H is summarized on Figure 5.1 and has been used in development of the sampling strategy summarized in Table 5.1. Proposed

RFI sampling locations are presented on Figure 5.2/Plan 2. Exact sample locations will be determined based on the actual field conditions encountered. Surface soil, subsurface soil, background soil, and groundwater sampling strategies are further discussed below.

A. SURFACE SOIL SAMPLING

A surface soil sample will be collected at each of the borehole locations presented on Figure 5.2/Plan 2. These samples will be collected from the 0 to 2 feet bgs interval. Sampling and analytical protocols are presented in the QAPP and FSP, respectively.

B. SUBSURFACE SOIL SAMPLING

Subsurface soil samples will be collected at selected borehole locations presented on Figure 5.2/Plan 2. The subsurface soil sample will be collected at the interval exhibiting the greatest degree of staining or positive response on the PID. If no staining or PID response is encountered, a subsurface sample may not be collected. All boreholes will be installed to a depth of approximately 5 to 10 feet into the native clay till horizon (as directed by the CRA field representative). Sampling and analytical protocols are presented in the QAPP and FSP, respectively.

C. BACKGROUND SOIL SAMPLING

To allow evaluation of the soil sample results from the AOIs and to obtain Facility-wide stratigraphic information, background soil samples will be collected from the native soils. A total of four to eight samples will be collected from areas at the Facility with no history of activities that may have caused contamination. The approximate location of these samples is presented on Figure 5.2/Plan 2. The distance between the sample locations was maximized in order to obtain a representative background data set. The four background soil boreholes will be drilled to a total depth of approximately 30 feet. Background soil samples will be analyzed for grain size, vertical hydraulic conductivity, and TAL inorganics. Sampling and analytical protocols are presented in the QAPP and FSP, respectively.

In addition, REALM will also evaluate the use of background inorganic levels defined for the landfill closure completed for the property to the north of the Facility, to determine if any of that information may be applicable to the Facility.

D. GROUNDWATER SAMPLING

Based on historical groundwater data, certain existing monitoring wells will be re-sampled during Task 3. The locations of these previously existing monitoring wells are presented on Figure 5.2/Plan 2 and summarized in Table 5.1.

A perched water table zone may exist in isolated locations of the near surface (upper 5 to 10 feet) at the Facility. A significant groundwater aquifer may be present in this perched water table zone if groundwater is identified to be continuous over the Facility and the formation is capable of producing a significant yield (e.g. 5 feet of porous media, 2 feet saturated, continuous horizontally). The exact location and number of monitoring wells, if any, will be determined after the completion of Task 3. The scope of the groundwater investigation will be refined based upon a review of the MDEQ-WMD guidance currently under preparation for assessment of "groundwater not in an aquifer" (as identified in the REALM/MDEQ-WMD meeting on April 12, 2000) and an assessment of the presence of significant soil contamination based upon the soil analytical results developed during Task 3. Sampling and analytical protocols are presented in the QAPP and FSP, respectively.

5.1 TASK 1: TOPOGRAPHIC SURVEY

A topographic survey will be completed to support tracking of all Corrective Action activities, including the RFI investigation. This task will include completing aerial photographs and "ground truthing" to establish a new base plan for the Facility.

5.2 TASK 2: GROUNDWATER USE INVENTORY

An updated groundwater use inventory will be completed to identify all groundwater users in a vicinity of approximately 1 mile of the Facility.

5.3 TASK 3: PHASE I CHARACTERIZATION OF AREAS OF INTEREST

The following subsections outline the proposed investigation, if any, at each AOI and provides descriptions of the current conditions of the AOIs at the Facility. Figure 1.2

presents the locations of these AOIs. The locations of the proposed boreholes are presented on Figure 5.2/Plan 2 and summarized in Table 5.1.

5.3.1 AOI 1 – FORMER DRUM STORAGE AREA

The Former Drum Storage Area is located north of, and immediately adjacent to, the main manufacturing facility (Building 44). The Former Drum Storage Area consisted of a fenced concrete pad with dimensions of 42 feet by 72 feet, with a capacity to store 648 standard 55-gallon drums.

A 4-inch high perimeter concrete curb surrounding the Former Drum Storage Area provided secondary containment. A sump associated with the Former Drum Storage Area is located in the southwest corner of the pad under a small enclosure and consists of two concrete vaults, each 8 feet in depth. The location of the Former Drum Storage Area is presented on Figure 1.2/Plan 1.

GM submitted a "Draft Final Closure Certification Package" for the Former Drum Storage Area to MDEQ in October 1998. In response to a letter received from MDEQ regarding inadequate clean closure of the Former Drum Storage Area, REALM has recently submitted a Work Plan to MDEQ that proposes additional sampling to satisfy the questions identified by MDEQ. Therefore, sampling activities for the Former Drum Storage Area will not be addressed as part of the Facility RFI.

5.3.2 AOI 2 – FORMER WASTE PILE PAD

The Former Waste Pile Pad (AOI 2) was located east of Building 44 and approximately 100 feet north-northeast of Building 63. The area consisted of a concrete pad measuring 60 feet by 40 feet, with an underground drainage trench connected to an adjacent collection sump which discharged to the former WWTP (reconnected to former Wastewater Storage Tanks (AOI 6) during Peregrine operations). This drainage trench is oriented north/south along the center of the long axis of the pad. The unit has a 1.5-foot high curb on the east side and the concrete floor was sloped to direct runoff to the underground drainage system. The location of the Former Waste Pile Pad is presented on Figure 1.2/Plan 1.

The Former Waste Pile Pad was determined by GM to be clean (Weston, 1998), as verified by a program of rinse water sampling and analyses performed following removal of contaminated soils and sludge as specified in the approved Closure Plan. Subsequent use of this area by Peregrine did not involve hazardous materials. There were no specified future monitoring requirements in the "Draft Final Closure Certification Package" for the Former Waste Pile Pad, dated October 1998.

5.3.2.1 PROPOSED SOIL INVESTIGATION

The purpose of the proposed soil investigation is to determine whether a release of hazardous waste and/or hazardous constituents has occurred from the Former Waste Pile Pad and, if so, to characterize the exposure concentration so that risk-based decisions on the need for corrective measures can be made. Historical soil data from this area is limited to one sample from one location with no exceedances of the IDWP or IDC criteria.

Three borehole locations in the Former Waste Pile Pad will be installed for soil sampling (see Figure 5.2/Plan 2). The boreholes will be sampled continuously to a depth of approximately 5 feet into the native clay till soils (a depth of approximately 10 to 20 feet). At each borehole location, one sample of the surface soil will be collected at the 0 to 2 foot bgs interval as discussed in Section 5.0. The subsurface soil sample exhibiting the most staining or highest PID response will also be submitted for chemical analysis as discussed in Section 5.0. If no staining or PID response is encountered, a subsurface sample may not be collected.

The soil samples (and associated QA/QC samples) will be analyzed for TCL VOCs, TCL SVOCs, TCL PCBs, and TAL inorganics (including both total and hexavalent chromium).

5.3.2.2 PROPOSED GROUNDWATER INVESTIGATION

Previous investigations showed an exceedance of the IDWC for trichloroethene (TCE) in one of the two samples collected at an existing monitoring well, MW-13. As part of the proposed groundwater investigation, a sample will be collected from MW-13 and analyzed for VOCs.

5.3.3 AOI 3 - FORMER USTs

Two former USTs were located outside along the eastern wall of Building 44 near building grid S-10. Each tank had a capacity of 2,000 gallons. The location of the Former USTs is presented on Figure 1.2/Plan 1.

No additional investigation is required. These two Former USTs are identified as being removed on the Michigan UST-DMS Facility and Tank Data Listing, dated April 27, 1999, and as being closed on November 9, 1990 on the Genesee County LUST Site List, dated October 11, 1999 (As requested, documentation of the UST closures provided to MDEQ-WMD through separate correspondence).

5.3.4 AOI 4 – FORMER SATELLITE ACCUMULATION AREAS

Former Satellite Accumulation Areas existed throughout Building 44. Each area consisted predominantly of 55-gallon drum(s) used for the temporary storage of hazardous wastes.

No investigation is required. Previous studies performed at the Facility in 1996/1997 did not identify the Former Satellite Accumulation Areas as a potential area of concern (refer to DOCC for further information). The Former Satellite Accumulation Areas no longer exist at the Facility.

5.3.5 AOI 5 – FORMER HAZARDOUS MATERIALS STORAGE AREA

The Former Hazardous Materials Storage Area was located in the northwest corner of the Hazardous Materials Storage Shed (former Building 66). Access to the specific area within the building was through a gate in the fence that was kept locked when not in use. Building 66 no longer exists (the shed has been removed). The Former Hazardous Materials Storage Area is empty and no longer fenced, but is surrounded by a 6-inch high concrete curb that provides approximately 2,500 gallons of containment volume. The location of the Former Hazardous Materials Storage Area is presented on Figure 1.2/Plan 1.

No investigation is necessary. The U.S. EPA identified this area to have low potential for concern. The Former Hazardous Materials Storage Area building has been demolished with no evidence of releases from the area (refer to DOCC for further information).

5.3.6 AOI 6 – FORMER WASTEWATER STORAGE TANKS

The Former Wastewater Storage Tanks (AOI 6) were located indoors in the northwest corner of Building 44 after December 1996. The Former Wastewater Storage Tanks included four aboveground 20,000-gallon steel frac tanks that were used to accumulate wastewater, metal drawing lubricants, and other non-hazardous liquid waste streams from the Facility. The tanks were underlain by the concrete floor of the building and could be readily inspected. The location of the Former Wastewater Storage Tanks is presented on Figure 1.2/Plan 1.

Previous studies performed at the Facility in 1996/1997 did not identify the Former Wastewater Storage Tanks as a potential area of concern since the tanks had only been installed by Peregrine after December 1996 and there had been no report or evidence of a release. As such, no sampling was specifically performed in the vicinity of this AOI.

All remaining tanks were removed from the Facility during the auction held by Peregrine in 1999 prior to leaving the Facility. As a result, no investigation is required (refer to DOCC for further information).

5.3.7 AOI 7 – FORMER SCRAP METAL MANAGEMENT SYSTEM

The non-hazardous Former Scrap Metal Management System was located primarily within the eastern half of the first floor of Building 44, associated with the die cast operations, plating machines, and the press room pits. It consisted of a series of conveyors within trenches and associated collection sumps located adjacent to the various machinery.

Previous studies performed at the Facility in 1996/1997 observed oil staining on the floor in the vicinity of the Former Scrap Metal Management System. Ten soil samples and one groundwater sample have been collected from the area. These sampling activities identified no exceedances of the IDWP in the soil, but barium was detected in

the groundwater at PFW-10 at a level exceeding the IDWC. No exceedances of the IDC or GCC were reported for soil or groundwater in any of the collected samples. Additional sampling will be performed and addressed as part of the investigation of the Former Press Room (AOI #19).

5.3.8 AOI 8 – FORMER SCRAP METAL STORAGE AREA

The Former Scrap Metal Storage Area was located indoors in the northwest-central portion of Building 63. It was comprised of a series of five in-ground concrete storage bins measuring approximately 8 feet by 10 feet by 10 feet deep. The Former Scrap Metal Storage Area location is presented on Figure 1.2/Plan 1.

The five concrete storage bins have been filled with gravel and covered with concrete. No documentation is available to demonstrate that integrity testing was performed prior to filling by Peregrine.

5.3.8.1 PROPOSED SOIL INVESTIGATION

The purpose of the proposed soil investigation is to determine whether a release of hazardous waste and/or hazardous constituents has occurred from the Former Scrap Metal Storage Area and, if so, to characterize the exposure concentration so that risk-based decisions on the need for corrective measures can be made. Historically, five soil samples have been collected from different locations in the Former Scrap Metal Storage Area. An exceedance of the IDWP for arsenic was encountered at PFB-7.

Five borehole locations in the Former Scrap Metal Storage Area will be selected for soil sampling (see Figure 5.2/Plan 2). The boreholes will be completed either through or immediately adjacent to the former scrap metal collection bins. The boreholes will be sampled continuously to a depth of approximately 5 feet into the native clay till soils.

The soil samples will be collected in a manner consistent with the protocols discussed for AOI #2 (and associated QA/QC samples) will be analyzed for TCL VOCs, TCL SVOCs, TCL PCBs, and TAL inorganics (including both total and hexavalent chromium).

5.3.9 AOI 9 - FORMER PLATING AREAS

The Former Plating Areas comprised three separate areas in Building 44 (see Figure 1.2/2.3, Plan 1). The zinc barrel plating area was located toward the southeast end of the first floor, the chrome plating area was located toward the north end of the first floor, and the plating solution recycling area was located in the basement.

The Former Plating Areas are currently vacant and inactive. The platers were all decommissioned through cleaning/ rinsing and removed from the Facility. The pits, sumps, and trenches associated with the plater removals were backfilled with sand and covered with concrete, except for the former zinc barrel plater (near the south end of Building 44) for which the foundations still exist.

5.3.9.1 PROPOSED SOIL INVESTIGATION

The purpose of the proposed soil investigation in the Former Plating Areas is to supplement existing data that indicates the presence of hazardous constituents in the soil beneath the building floor.

Thirty borehole locations in the Former Plating Areas will be selected for soil sampling (see Figure 5.2/Plan 2). The boreholes will be completed through the floor. The boreholes will be sampled continuously to a depth of approximately 5 feet into the native clay till soils.

The soil samples will be collected in a manner consistent with the protocols discussed for AOI #2 (and associated QA/QC samples) will be analyzed for TCL VOCs and TAL inorganics (including both total and hexavalent chromium). Two samples will be collected at AOI #19 and analyzed for the Appendix IX parameter list as modified for this investigation.

5.3.10 AOI 10 - OVERHEAD WASTE PIPES

The Overhead Waste Pipes were located in Building 44. The overhead waste pipes included separate lines for spent plating solutions and other process waste streams. Plan 1 presents the location of the main branches of the Overhead Waste Pipes.

No additional investigation is required. Previous studies performed at the Facility in 1996/1997 concluded that the Overhead Waste Pipes were not a specific area of concern. Nevertheless, five samples of residues were collected from the open ends of pipes in the area and analyzed by TCLP. No exceedances of regulatory limits were encountered. All remaining process pipes are being properly decommissioned as part of ongoing decommissioning and demolition activities.

5.3.11 AOI 11 – FORMER COAL STORAGE AREA

The Former Coal Storage Area was comprised of a relatively large area located to the north of the powerhouse (Building 45). The location of the Former Coal Storage Area is presented on Figure 1.2/Plan 1.

The Former Coal Storage Area is currently inactive, and residual coal will be removed from the area as part of the demolition efforts being performed by REALM.

5.3.11.1 PROPOSED SOIL INVESTIGATION

The purpose of the proposed soil investigation is to determine whether a release of hazardous waste and/or hazardous constituents has occurred from the Former Coal Storage Area and, if so, to characterize the exposure concentration so that risk-based decisions on the need for corrective measures can be made.

Five borehole locations in the Former Coal Storage Area will be selected for soil sampling (see Figure 5.2/Plan 2). The boreholes will be sampled continuously to a depth of approximately 5 feet into the native clay till soils.

The soil samples will be collected in a manner consistent with the protocols discussed for AOI #2 (and associated QA/QC samples) will be analyzed for TCL SVOCs, TCL PCBs, and TAL inorganics (including both total and hexavalent chromium).

5.3.12 AOI 12 –FORMER CAUSTIC TANKS

Eleven aboveground steel tanks, each approximately 4 feet by 6 feet by 6 feet, are located immediately west of the Former Drum Storage Area (AOI 1) along the north side of

Building 44. All of the tanks are located on asphalt and are covered. The tanks were used to collect caustic cleaning solutions from a former metal cleaning process. The locations of the Former Caustic Tanks are presented on Figure 1.2/Plan 1.

The Former Caustic Tanks are currently inactive. These tanks will be properly decommissioned as part of ongoing decommissioning and demolition activities at the Facility.

5.3.12.1 PROPOSED SOIL INVESTIGATION

The purpose of the proposed soil investigation is to determine whether a release of hazardous waste and/or hazardous constituents has occurred from the Former Caustic Tanks and, if so, to characterize the exposure concentration so that risk-based decisions on the need for corrective measures can be made.

Two borehole locations in the Former Caustic Tanks area will be selected for soil sampling (see Figure 5.2/Plan 2). The boreholes will be sampled continuously to a depth of approximately 5 feet into the native clay till soils.

The soil samples will be collected in a manner consistent with the protocols discussed for AOI #2 (and associated QA/QC samples) will be analyzed for pH, TCL VOCs, and TAL inorganics (including both total and hexavalent chromium).

5.3.13 AOI 13 – FORMER HEADLINER ADHESIVE TANKS

The Former Headliner Adhesive Tanks (AOI 13) are located in a tank farm east of Building 44. There were eight stainless steel tanks, each about 30 feet high by 8 feet in diameter (approximately 8,000-gallon capacity), with secondary containment (provided by an approximate 2-foot concrete curb). The location of the Former Headliner Adhesive Tanks is presented on Figure 1.2.

During the removal of Tank #6 and the piping associated with the tanks, Tank #6 (known as the green adhesive tank) was found to contain a small quantity of solidified material. The material in the piping was sampled and found to be characteristically hazardous based on toxicity (benzene) and flammability. GM completed the removal of the green adhesive tank and associated piping in 1997. The other seven Former

Headliner Adhesive Tanks were found to be clean and remain at this AOI. The remaining tanks will be properly decommissioned and removed as part of the ongoing decommissioning and demolition activities.

5.3.13.1 PROPOSED SOIL INVESTIGATION

The purpose of the proposed soil investigation is to determine whether a release of hazardous waste and/or hazardous constituents has occurred from the Former Headliner Adhesive Tanks and, if so, to characterize the exposure concentration so that risk-based decisions on the need for corrective measures can be made.

Two borehole locations in the Former Headliner Adhesive Tanks area will be selected for soil sampling (see Figure 5.2/Plan 2). The boreholes will be sampled continuously to a depth of approximately 5 feet into the native clay till soils.

The soil samples will be collected in a manner consistent with the protocols discussed for AOI #2 (and associated QA/QC samples) will be analyzed for TCL VOCs.

5.3.14 AOI 14 - FORMER INCINERATOR

The Former Incinerator was located east of the powerhouse (Building 45). It was demolished in the 1980s. The approximate location of the Former Incinerator is presented on Figure 1.2/Plan 1.

The Former Incinerator area is currently vacant and inactive.

5.3.14.1 PROPOSED SOIL INVESTIGATION

The purpose of the proposed soil investigation is to determine whether a release of hazardous waste and/or hazardous constituents has occurred from the Former Incinerator and, if so, to characterize the exposure concentration so that risk-based decisions on the need for corrective measures can be made.

Two borehole locations in the Former Incinerator area will be selected for soil sampling (see Figure 5.2/Plan 2). The boreholes will be sampled continuously to a depth of approximately 5 feet into the native clay till soils.

The soil samples will be collected in a manner consistent with the protocols discussed for AOI #2 (and associated QA/QC samples) will be analyzed for TCL SVOCs, TCL PCBs, and TAL inorganics (including both total and hexavalent chromium).

5.3.15 AOI 15 - OTHER FORMER USTs

In addition to the two Former USTs identified as AOI 3, three Other Former UST areas were located at the Facility. These comprised two gasoline tanks, each of 2,000 gallon capacity, and a tank associated with the former plastisol operations. One of the gasoline tanks was located north of Building 44 and the other to the west of the Facility, toward the south end of Building 44 near the shipping dock. The plastisol tank was located on the east side of Building 44 in the vicinity of column S-31. The location of these three Other Former USTs is presented on Figure 1.2/Plan 1.

No investigation is required. The two gasoline USTs are identified as being removed and closed on the Michigan UST-DMS Facility and Tank Data Listing, dated April 27, 1999. The plastisol tank is also identified as being removed on the Michigan UST-DMS Facility and Tank Data Listing, dated April 27, 1999 (As requested, documentation of the UST closures will be provided to MDEQ-WMD through separate correspondence).

5.3.16 AOI 16 - FORMER POTENTIAL TANK FARM

One of the earlier environmental investigations performed at the Facility identified a Former Potential Tank Farm located approximately 300 feet east of the north end of Building 63 (based upon a Facility design drawing). The tank farm was initially designed to contain USTs associated with a potential jet fuel operation but the tanks were never installed according to interviews with former employees (due to a change in the projected use of the Facility). The location of the Potential Former Tank Farm (AOI 16) is presented on Figure 1.2.

No additional investigation is required. The area of the Former Potential Tank Farm is currently vacant.

5.3.17 AOI 17 - SUBSTATION DRAINAGE SUMP

The Substation Drainage Sump is located to the northeast of Building 63. The electrical substation area contained several large transformers. Stormwater runoff from the uncovered substation area collects in the sump located in the basement of the adjacent substation building and was historically pumped to the Former WWTP. The location of the Substation Drainage Sump is presented on Figure 1.2.

The area will become inactive and vacant as the decommissioning and demolition activities are completed.

5.3.17.1 PROPOSED SOIL INVESTIGATION

The purpose of the proposed soil investigation is to determine whether a release of hazardous waste and/or hazardous constituents has occurred from the Substation Drainage Sump and, if so, to characterize the exposure concentration so that risk-based decisions on the need for corrective measures can be made.

Two borehole locations in the Substation Drainage Sump area will be selected for soil sampling (see Figure 5.2/Plan 2). The boreholes will be sampled continuously to a depth of approximately 5 feet into the native clay till soils.

The soil samples will be collected in a manner consistent with the protocols discussed for AOI #2 (and associated QA/QC samples) will be analyzed for TCL SVOCs and TCL PCBs.

5.3.18 AOI 18 – FORMER INACTIVE EQUIPMENT RESERVE YARD

The Former Inactive Equipment Reserve Yard (IERY) was located east of Building 63 and was used to store dies and obsolete equipment. The location of the Former IERY is presented on Figure 1.2/Plan 1.

The area is currently vacant and inactive.

5.3.18.1 PROPOSED SOIL INVESTIGATION

The purpose of the proposed soil investigation is to determine whether a release of hazardous waste and/or hazardous constituents has occurred from the Former IERY and, if so, to characterize the exposure concentration so that risk-based decisions on the need for corrective measures can be made.

Four borehole locations in the Former IERY will be selected for soil sampling (see Figure 5.2/Plan 2). The boreholes will be sampled continuously to a depth of approximately 5 feet into the native clay till soils.

The soil samples will be collected in a manner consistent with the protocols discussed for AOI #2 (and associated QA/QC samples) will be analyzed for TCL VOCs, TCL SVOCs, TCL PCBs, and TAL inorganics (including both total and hexavalent chromium).

5.3.18.2 PROPOSED GROUNDWATER INVESTIGATION

Historically, an exceedance of the IDWC for lead was encountered in the groundwater at existing monitoring well, MW-3. As part of the proposed groundwater investigation, a sample will be collected from MW-3 and analyzed for filtered lead.

5.3.19 AOI 19 - FORMER PRESS ROOM

The Building 44 Former Press Room pits have been indirectly discussed under AOI 7 and AOI 8. However, AOI 19 addresses these and other miscellaneous pits and sumps in the Former Press Room.

Many of the sumps associated with the press room pits were filled with gravel/concrete by Peregrine prior to leaving the Facility.

5.3.19.1 PROPOSED SOIL INVESTIGATION

The purpose of the proposed soil investigation is to determine whether a release of hazardous waste and/or hazardous constituents has occurred from the Former Press Room and, if so, to characterize the exposure concentration so that risk-based decisions on the need for corrective measures can be made.

Eight borehole locations in the Former Press Room area will be selected for soil sampling (see Figure 5.2/Plan 2). The boreholes will be sampled continuously to a depth of approximately 5 feet into the native clay till soils.

The soil samples will be collected in a manner consistent with the protocols discussed for AOI #2 (and associated QA/QC samples) will be analyzed for TCL VOCs, TCL SVOCs, TCL PCBs, and TAL inorganics (including both total and hexavalent chromium). One sample will be collected at AOI #19 and analyzed for Appendix IX the parameter list as modified for this investigation.

5.3.20 AOI 20 – FORMER TOOL ROOM

The Former Tool Room on the first floor of Building 44 previously contained a small solvent-based degreaser, an air wash unit, and die wash station.

The Former Tool Room air wash unit was cleaned by GM and has been removed from the Site. The remaining portions of the Former Tool Room area are generally vacant and will undergo decommissioning, including the existing die wash station.

5.3.20.1 PROPOSED SOIL INVESTIGATION

The purpose of the proposed soil investigation is to determine whether a release of hazardous waste and/or hazardous constituents has occurred from the Former Tool Room and, if so, to characterize the exposure concentration so that risk-based decisions on the need for corrective measures can be made.

Four borehole locations in the Former Tool Room area will be selected for soil sampling (see Figure 5.2/Plan 2). The boreholes will be sampled continuously to a depth of approximately 5 feet into the native clay till soils.

The soil samples will be collected in a manner consistent with the protocols discussed for AOI #2 (and associated QA/QC samples) will be analyzed for TCL VOCs, TCL SVOCs, TCL PCBs, and TAL inorganics (including both total and hexavalent chromium). One sample will be collected at AOI #20 and analyzed for the Appendix IX parameter list as modified for this investigation.

5.3.21 AOI 21 – POWERHOUSE FUEL OIL STORAGE TANKS

Four aboveground fuel oil storage tanks with secondary containment were formerly located north of the Powerhouse. Three of the tanks (250-gallon each) have previously been removed. The location of the remaining tank is presented on Plan 1.

5.3.21.1 PROPOSED SOIL INVESTIGATION

The purpose of the proposed soil investigation is to determine whether a release of hazardous waste and/or hazardous constituents has occurred from the Powerhouse Fuel Oil Storage Tanks and, if so, to characterize the exposure concentration so that risk-based decisions on the need for corrective measures can be made.

Two borehole locations in the Powerhouse Fuel Oil Storage Tanks area will be selected for soil sampling (see Figure 5.2/Plan 2). The boreholes will be sampled continuously to a depth of approximately 5 feet into the native clay till soils.

The soil samples will be collected in a manner consistent with the protocols discussed for AOI #2 (and associated QA/QC samples) will be analyzed for TCL VOCs, TCL SVOCs, and TAL inorganics (including both total and hexavalent chromium).

5.3.22 NON-SPECIFIC SAMPLING LOCATIONS

Sampling during previous investigations identified groundwater exceedances of published criteria at 3 existing monitoring well locations that are not specific to any particular AOI location. Exceedances of the IDWC for both lead and TCE were encountered in groundwater samples collected at MW-4. As part of the RFI, a groundwater sample will be collected from MW-4 and analyzed for TCL VOCs and

filtered lead. Exceedances of the IDWC for lead were also encountered in groundwater samples collected at MW-10 and PFW-9. Both of these wells will be resampled and analyzed for filtered lead. The locations of these monitoring wells are presented on Figure 5.2/Plan 2.

5.4 TASK 4: RFI REPORTING

The RFI Report will be prepared as discussed in Section 7.1.

6.0 DATA EVALUATION

6.1 OBJECTIVES

The data evaluation process will ensure that the investigation data are sufficient in quality and quantity to:

- characterize the nature and extent of any releases of hazardous waste and/or hazardous constituents at or from the Site;
- assess the potential risk to human health and the environment from any releases; and
- support the RCRA corrective action environmental indicators determinations.

The data analysis will be conducted on each data point that is generated during the RFI. Each new data point will be compared to historical sample results to ensure that the new data point fits with conclusions of earlier data. If new data do not fit with earlier conclusions, both the earlier conclusions and the new data will be reviewed to determine the reasons for the discrepancy.

6.2 DATA VALIDATION AND ANALYSIS

The data will be validated as discussed in the QAPP (Appendix C) to ensure that the data are useable. The data will be received in electronic form from the laboratory, where possible, and a database will be developed to facilitate data review and calculations.

Cross-checking between various data sets will be performed to ensure that the data are consistent and correct. This may include comparison between RFI sample results from the same AOI, comparison between RFI and historical sample results in the same area, and concurrent evaluation of chemical and physical data.

6.3 EXPOSURE ASSESSMENT

The chemical data will be evaluated to identify the nature and extent of contamination, if any, associated with each AOI. The exposure concentration(s) for each AOI will be estimated from the RFI data and compared to screening levels to determine the necessity for the completion of AOI-specific evaluations.

The physical characteristics of the Facility, including topography, hydrology, geology, hydrogeology, geology, and ecology, will be evaluated in conjunction with the chemical data to identify chemical fate and transport mechanisms. This information will be used to assess the impact, if any, of the AOI on various environmental media at the Facility, both individually and as a whole.

The human health risk assessment will be conducted for each AOI using reasonable current and future land use scenarios. The ecological risk assessment will be conducted on a Facility-wide basis taking into account habitat restrictions. The human health and ecological risk assessments will be completed in a manner consistent with applicable guidance, including Risk Assessment Guidance for Superfund (RAGS).

6.4 POST-INVESTIGATION EVALUATION

A post-investigation evaluation will be conducted to:

- define the nature and extent of contamination, if any, associated with each AOI and at the Facility as a whole;
- define the risk associated with each AOI considering the exposure assessment discussed in Section 6.3;
- evaluate the need for corrective measures, if any, at each AOI and for the Facility as a whole to allow a final environmental indicators determination;
- if corrective measures are required, determine whether sufficient data are available to evaluate corrective measures technologies and support selection of a corrective measure; and
- identify additional data collection activities, if any, required to support the environmental indicators determination or corrective measures evaluation.

The data evaluation will be presented in the RFI Report.

6.5 POTENTIAL CORRECTIVE MEASURES TECHNOLOGIES

The following sections present potential corrective measures technologies to be considered for the Facility, as necessary, to protect human health and the environment.

The final list of corrective measures technologies will be presented and evaluated in the Corrective Measures Study Work Plan (CMS Work Plan), and will utilize the results of the RFI.

6.5.1 NO ACTION

The "No Action" alternative allows the Facility to exist as it is without the implementation of any corrective measures. The "No Action" alternative may incorporate long-term environmental monitoring at the Facility to confirm that the future conditions do not deteriorate significantly from current conditions.

Hazardous constituents would remain under the "No Action" alternative and no measures would be implemented to treat, remove, or contain source areas. A contingency plan would be developed and implemented dependent on the monitoring results.

6.5.2 LIMITED ACTION

The "Limited Action" alternative involves the implementation of institutional controls and property access restrictions to reduce potential human exposure to hazardous constituents.

Institutional controls would involve the placement of a restriction within the deed (or other institutional controls such as zoning or building code changes) to the property preventing the property owner from conducting certain activities that may lead to unacceptable exposures (e.g., excavation, residential use). This action would also warn prospective future purchasers of the past history and condition of the property.

Property access restrictions may include upgrading the existing security fence, constructing additional security fencing, or instituting other controls to minimize access to the Facility by unauthorized personnel.

Hazardous constituents would remain under the "Limited Action" alternative. However, the potential for unacceptable future human or ecological exposure would be reduced.

6.5.3 CONTAINMENT

The "Containment" alternative involves the use of structural controls to contain or otherwise restrict the mobility and migration of hazardous constituents. Potential containment technologies applicable to the Facility include one or a combination of the following:

- capping; and
- property re-development.

Capping would involve the construction of a low permeability cover over the contaminated soil at the Facility. The purpose of the cover is to prevent direct contact and reduce infiltration of precipitation through the soil and subsequent percolation of potentially contaminated water into the subsurface. In addition, capping provides long-term protection against erosion and subsequent transport of hazardous constituents.

The property is currently being marketed for possible re-development. Re-development of the property would include the construction of buildings, roadways, parking areas, and landscaping which all could be designed as effective containment components.

Hazardous constituents would remain under the "Containment" alternative. However, the potential for unacceptable future human or ecological exposure would be significantly reduced or eliminated. The "Containment" alternative may also involve consolidation of contaminated materials in a centralized location where they can be effectively managed.

6.5.4 TREATMENT

The "Treatment" alternative involves the use of treatment technologies to remove or destroy hazardous constituents from environmental media. Numerous treatment technologies are available, which may use physical, chemical, or biological treatment methods. Either in situ or ex situ treatment technologies may be employed. Ex situ treatment technologies may be employed both on or off the Facility. The treatment technologies to be evaluated would be selected depending on the chemical(s) to be

removed or destroyed, the environmental media to be treated, and various physical characteristics of the media.

Hazardous constituents would be removed or destroyed under the "Treatment" alternative, and therefore, the potential for unacceptable future human or ecological exposure would be eliminated after treatment was complete. The "Treatment" alternative may also involve consolidation of contaminated materials in a centralized location where they can be effectively treated and managed.

6.5.5 REMOVAL

The "Removal" alternative involves the removal of contaminated material from the Facility for permanent disposal.

Hazardous constituents would be removed, and therefore, the potential for unacceptable future human or ecological exposure at the Facility would be eliminated.

7.0 REPORTING

The following submittals will be completed:

- The Description of Current Conditions Report has already been submitted;
- RFI Work Plan; and
- RFI Report.

7.1 RFI REPORT

The RFI Report will be developed after investigative activities have been completed to 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, and geology;
- 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 corrective 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 CMS Work Plan. 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.

8.0 RFI SCHEDULE

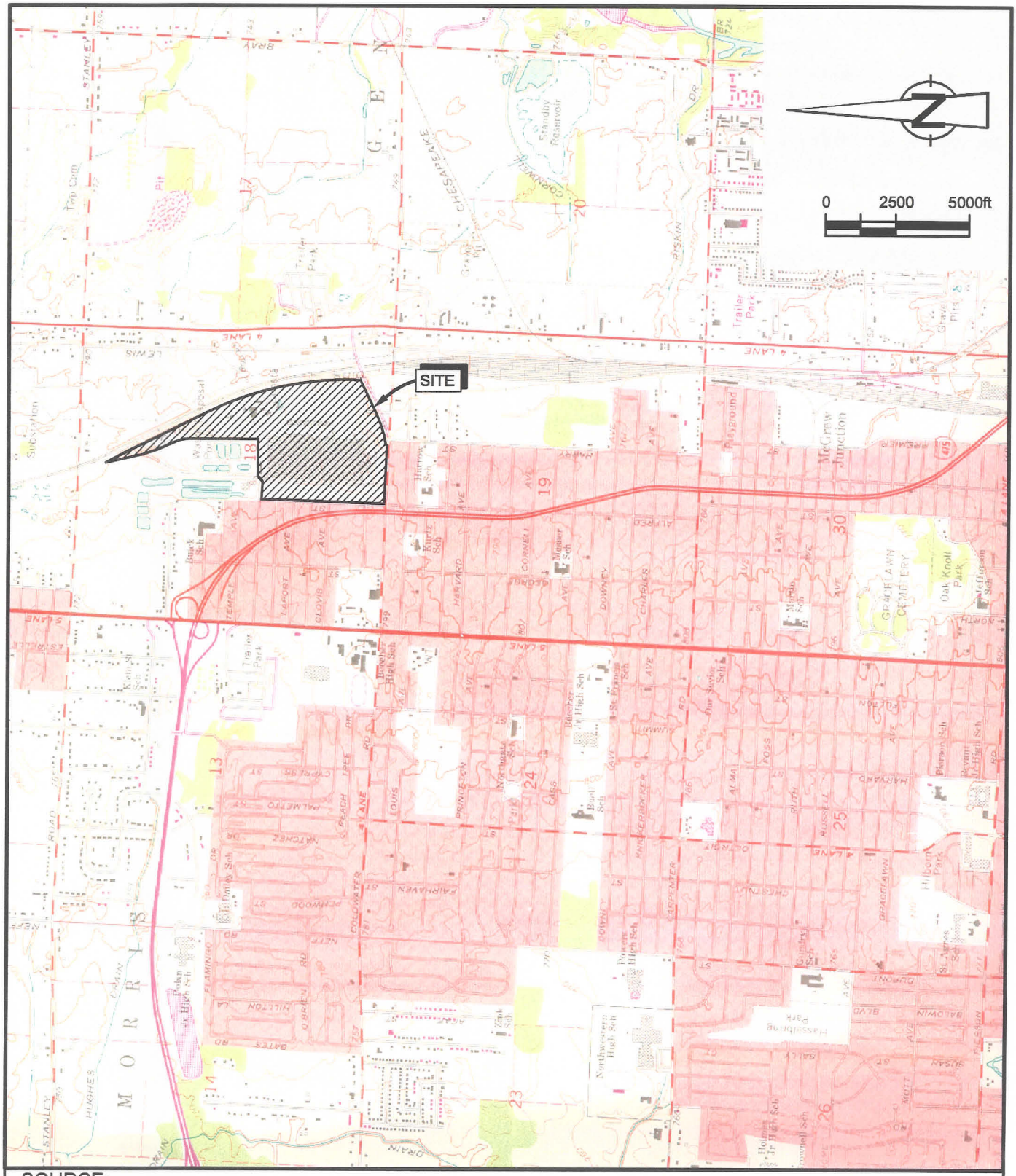
The RFI Work Plan will be implemented in a manner consistent with the schedule presented on Figure 8.1.

The schedule may be affected by access issues (e.g., property re-development opportunities), weather conditions, and the necessity to obtain approvals or reviews from regulatory agencies. If changes to the schedule occur, MDEQ-WMD will be notified.

9.0 REFERENCES

1) "Interim Final RCRA Facility Investigation (RFI) Guidance", EPA 530/SW-89-031; Waste Management Division, Office of Solid Waste, U.S. EPA; May 1989.

2) "Description of Current Conditions Report", CRA, March 31, 2000.



SOURCE:

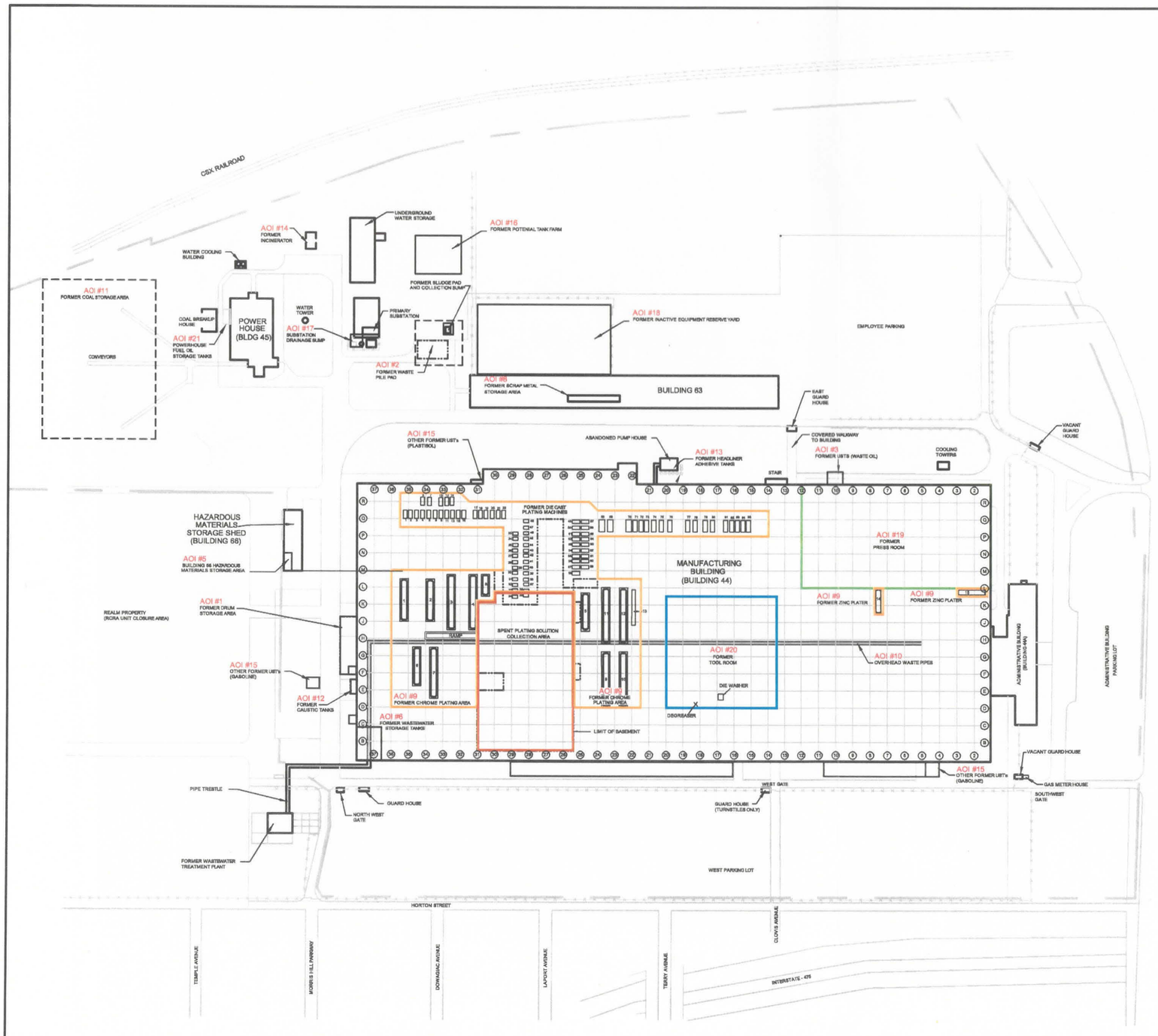
USGS FLINT NORTH QUADRANGLE
MICHIGAN-GENESEE CO.
MAPS, PHOTOREVISED 1975.

figure 1.1

FACILITY LOCATION
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA006 MAY 15/2000



0 100 300ft

LEGEND

PROPERTY BOUNDARY

FENCE LINE

AOI DESCRIPTION

AOI #1	FORMER DRUM STORAGE AREA
AOI #2	FORMER WASTE PILE PAD
AOI #3	FORMER USTs
AOI #4	FORMER SATELLITE ACCUMULATION AREAS
AOI #5	FORMER HAZARDOUS MATERIALS STORAGE AREA
AOI #6	FORMER WASTEWATER STORAGE TANKS
AOI #7	FORMER SCRAP METAL MANAGEMENT SYSTEM
AOI #8	FORMER SCRAP METAL STORAGE AREA
AOI #9	FORMER PLATING AREAS
AOI #10	OVERHEAD WASTE PIPES
AOI #11	FORMER COAL STORAGE AREA
AOI #12	FORMER CAUSTIC TANKS
AOI #13	FORMER HEADLINER ADHESIVE TANKS
AOI #14	FORMER INCINERATOR
AOI #15	OTHER FORMER USTs
AOI #16	FORMER POTENTIAL TANK FARM
AOI #17	SUBSTATION DRAINAGE SUMP
AOI #18	FORMER INACTIVE EQUIPMENT RESERVE YARD
AOI #19	FORMER PRESS ROOM
AOI #20	FORMER TOOL ROOM
AOI #21	POWERHOUSE FUEL OIL STORAGE TANKS

NOTE:
THERE IS NO SPECIFIC LOCATION FOR AOI#4 AND AOI #7.

NOTES

1. THIS DRAWING IS FOR REFERENCE ONLY AND IS NEITHER COMPLETE NOR TO EXACTING SCALE.
2. DRAWING No. 1795W BUICK MOTOR DIVISION SHEET No. 1M WAS USED AS A REFERENCE DRAWING.
3. LOCATIONS OF GENERAL MOTORS RCRA UNIT CLOSURE MONITORING WELLS (i.e. B-WELLS) ARE APPROXIMATE

figure 1.2

FACILITY PLAN AND AOI LOCATIONS
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA007 MAY 15/2000

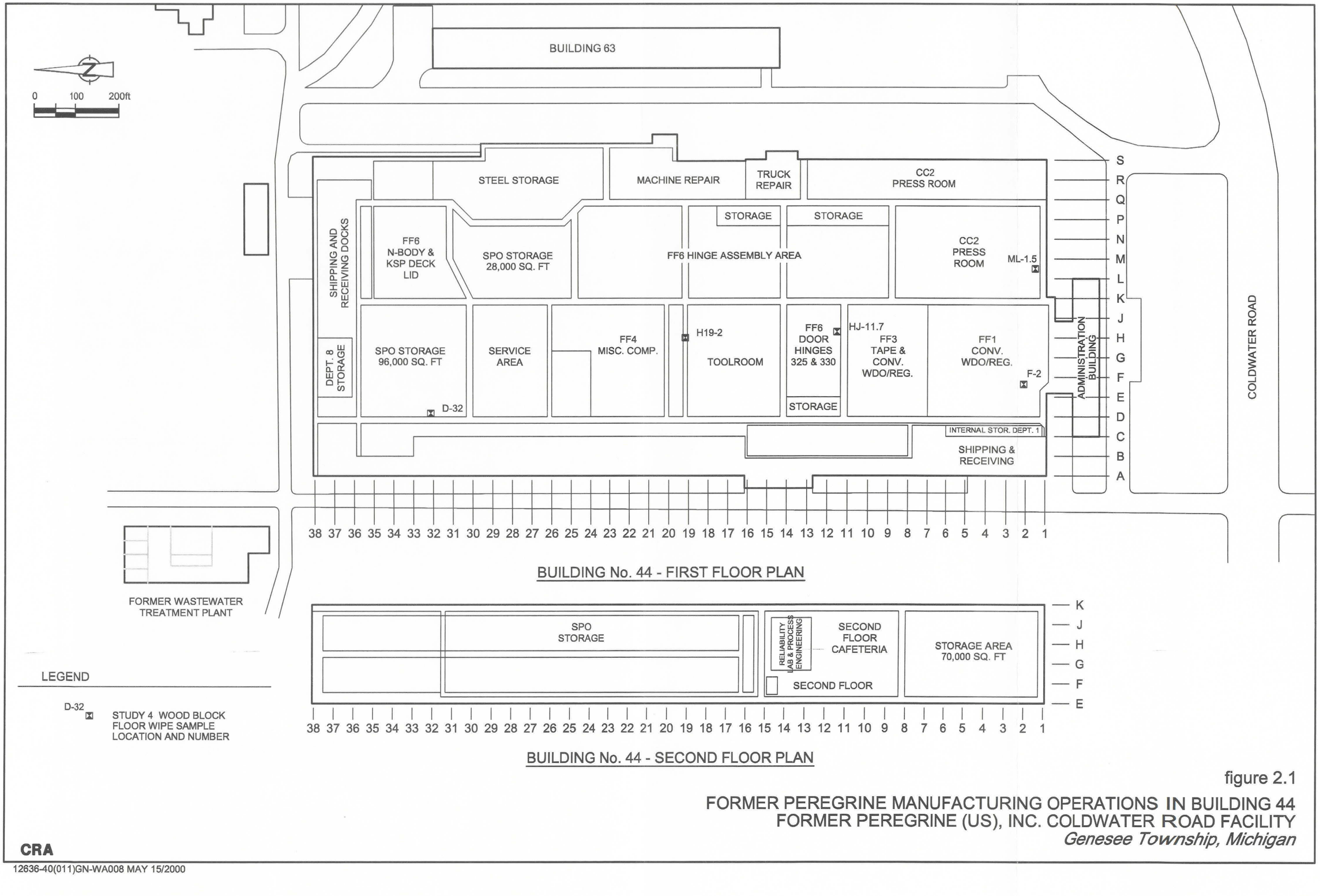
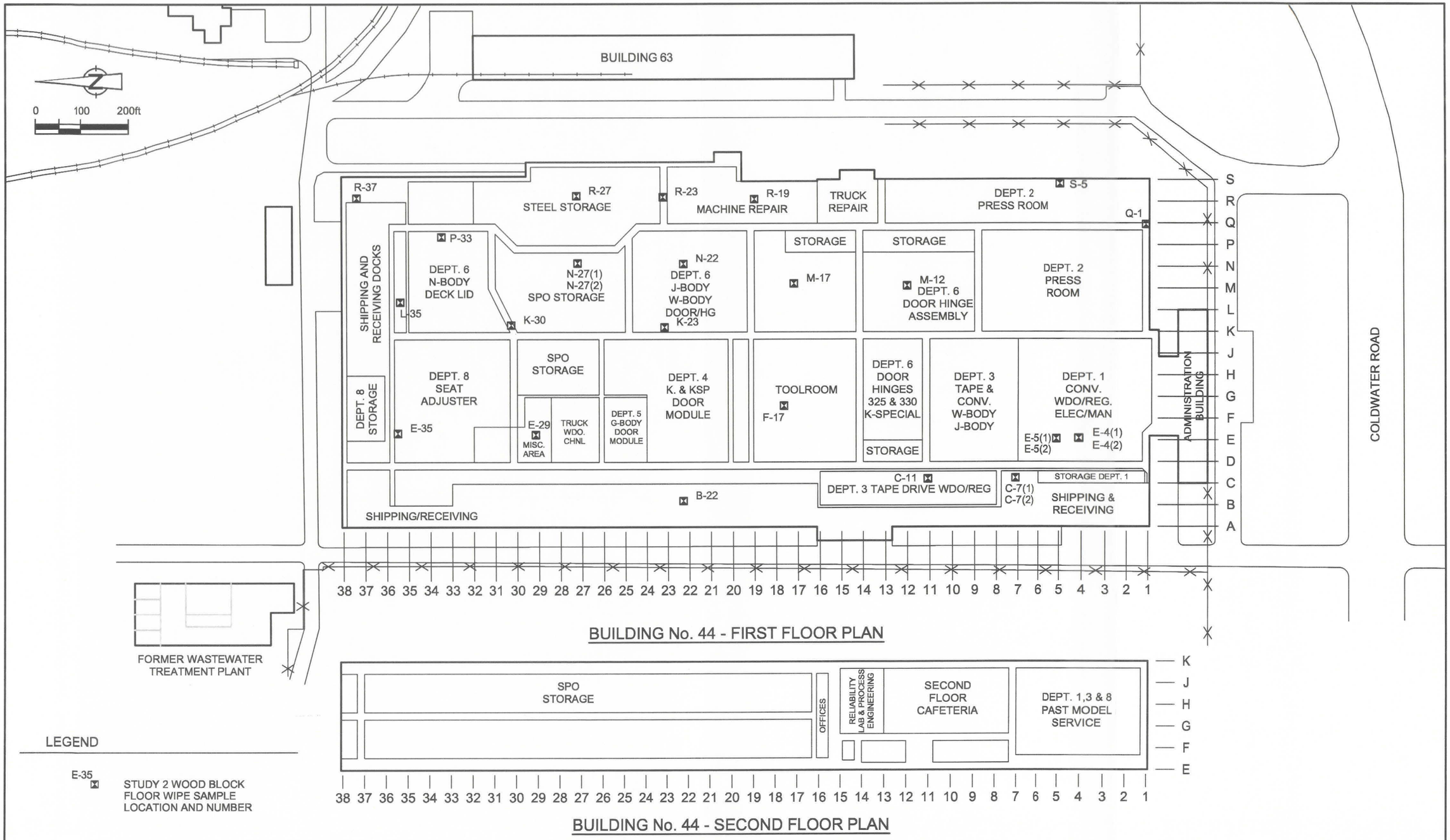


figure 2.1

FORMER PEREGRINE MANUFACTURING OPERATIONS IN BUILDING 44
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA008 MAY 15/2000



BUILDING No. 44 - FIRST FLOOR PLAN

BUILDING No. 44 - SECOND FLOOR PLAN

LEGEND

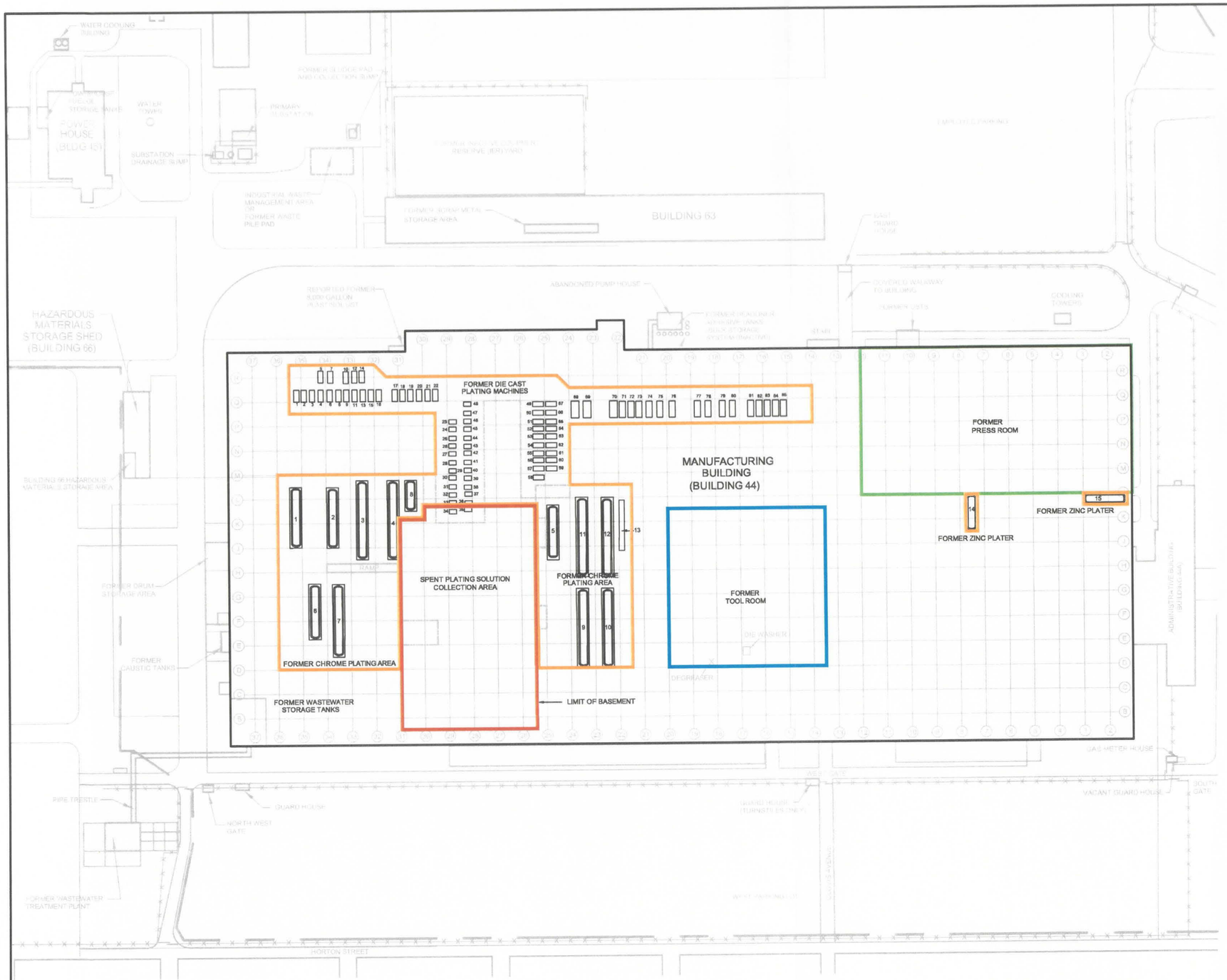
E-35 STUDY 2 WOOD BLOCK FLOOR WIPE SAMPLE LOCATION AND NUMBER

figure 2.2

FORMER DELPHI MANUFACTURING OPERATIONS IN BUILDING 44
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA009 MAY 15/2000



LEGEND

PROPERTY BOUNDARY

FENCE LINE

DESCRIPTION OF FORMER PLATING MACHINES

1. HANSON VAN WINKLE PLATING MACHINE #1
2. HANSON VAN WINKLE CHROME PLATER #2
3. STEVENS COPPER NICKEL CHROME PLATING MACHINE #3
4. STEVENS COPPER NICKEL CHROME PLATING MACHINE #4
5. HANSON VAN WINKLE PLATING MACHINE #5
6. #6 NICKEL PLATING MACHINE
7. #7 COPPER PLATING MACHINE
8. UDYLITE JUNIOR #8
9. UDYLITE #9 DUAL NICKEL DULA CHROME PLATER
10. UDYLITE #10 DUAL NICKEL DULA CHROME PLATER
11. HANSON VAN WINKLE PLATER #11
12. HANSON VAN WINKLE PLATER #12
13. CINCINNATI PARTS WASHER (STILL PLATER #13)
14. STEVENS BARREL ZINC PLATER
15. INACTIVE ZINC BARREL PLATING UNIT

NOTES

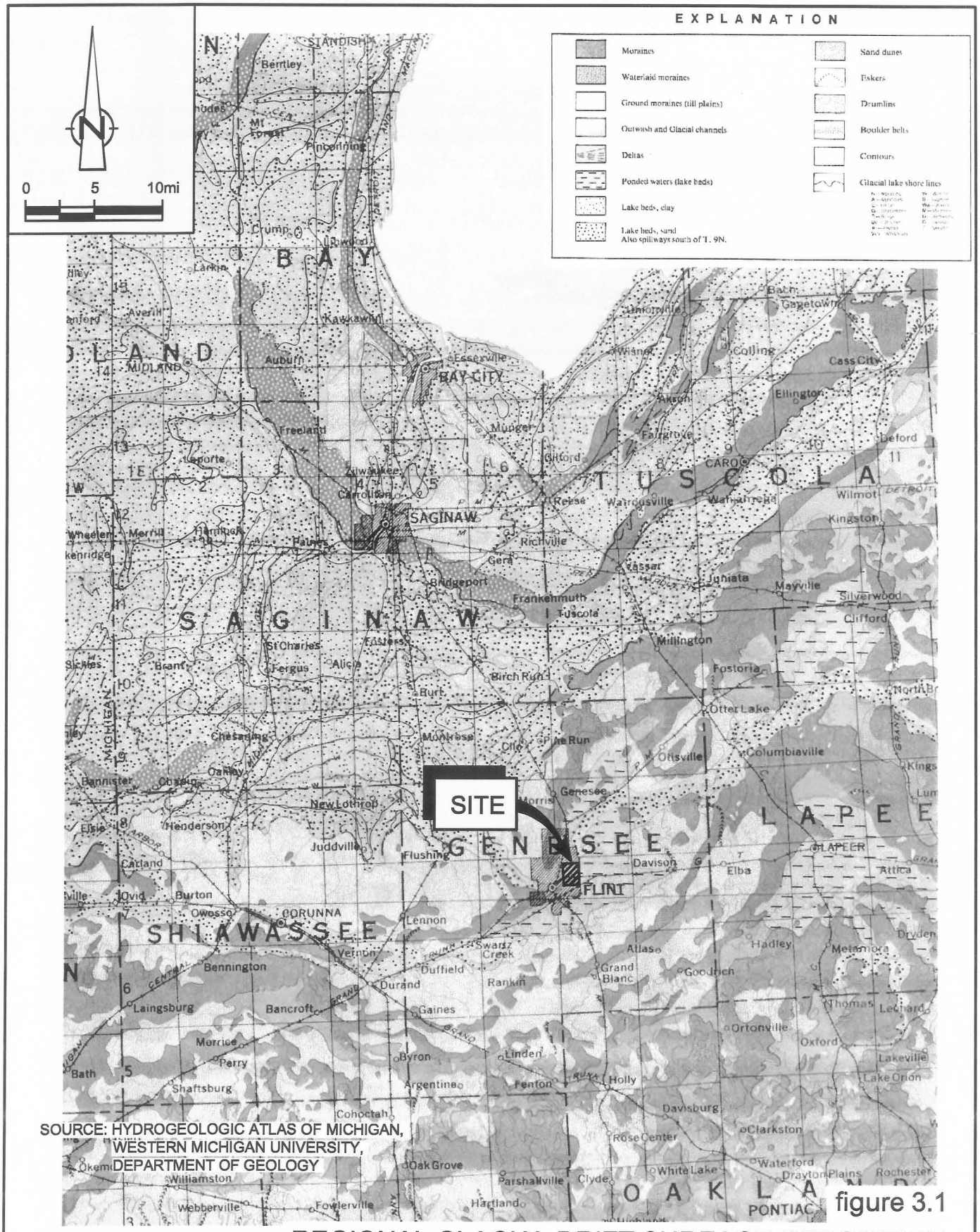
1. THIS DRAWING IS FOR REFERENCE ONLY AND IS NEITHER COMPLETE NOR TO EXACTING SCALE.
2. DRAWING No. 1795W BUICK MOTOR DIVISION SHEET No. 1M WAS USED AS A REFERENCE DRAWING.
3. LOCATIONS OF GENERAL MOTORS RCRA UNIT CLOSURE MONITORING WELLS (i.e. B-WELLS) ARE APPROXIMATE

figure 2.3

FORMER PLATING AND DIE CAST OPERATION AREAS
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA010 MAY 15/2000



SOURCE: HYDROGEOLOGIC ATLAS OF MICHIGAN, WESTERN MICHIGAN UNIVERSITY, DEPARTMENT OF GEOLOGY

figure 3.1

REGIONAL GLACIAL DRIFT SURFACE DEPOSITION
FORMER PEREGRINE (US) INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA011 MAY 15/2000

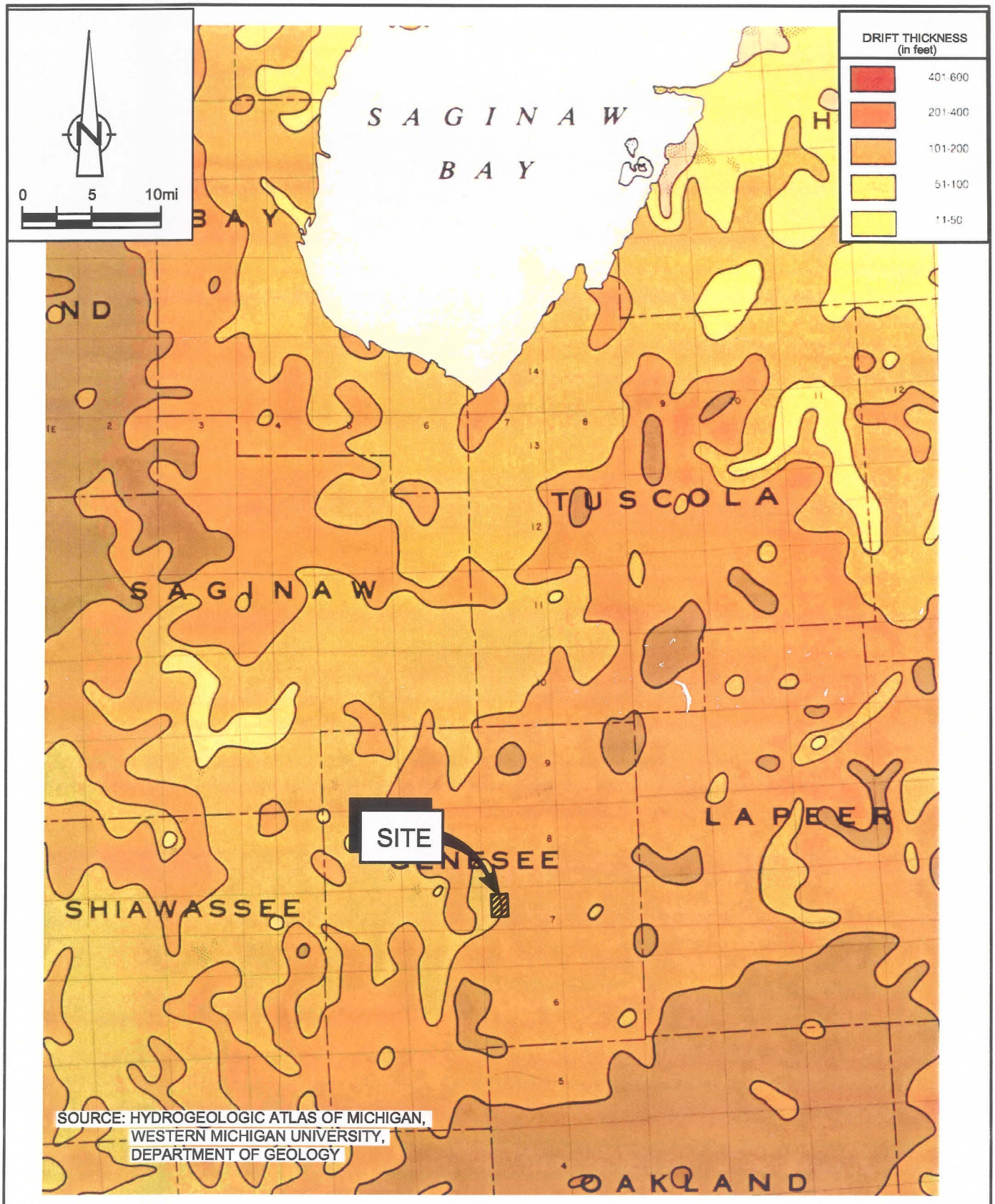
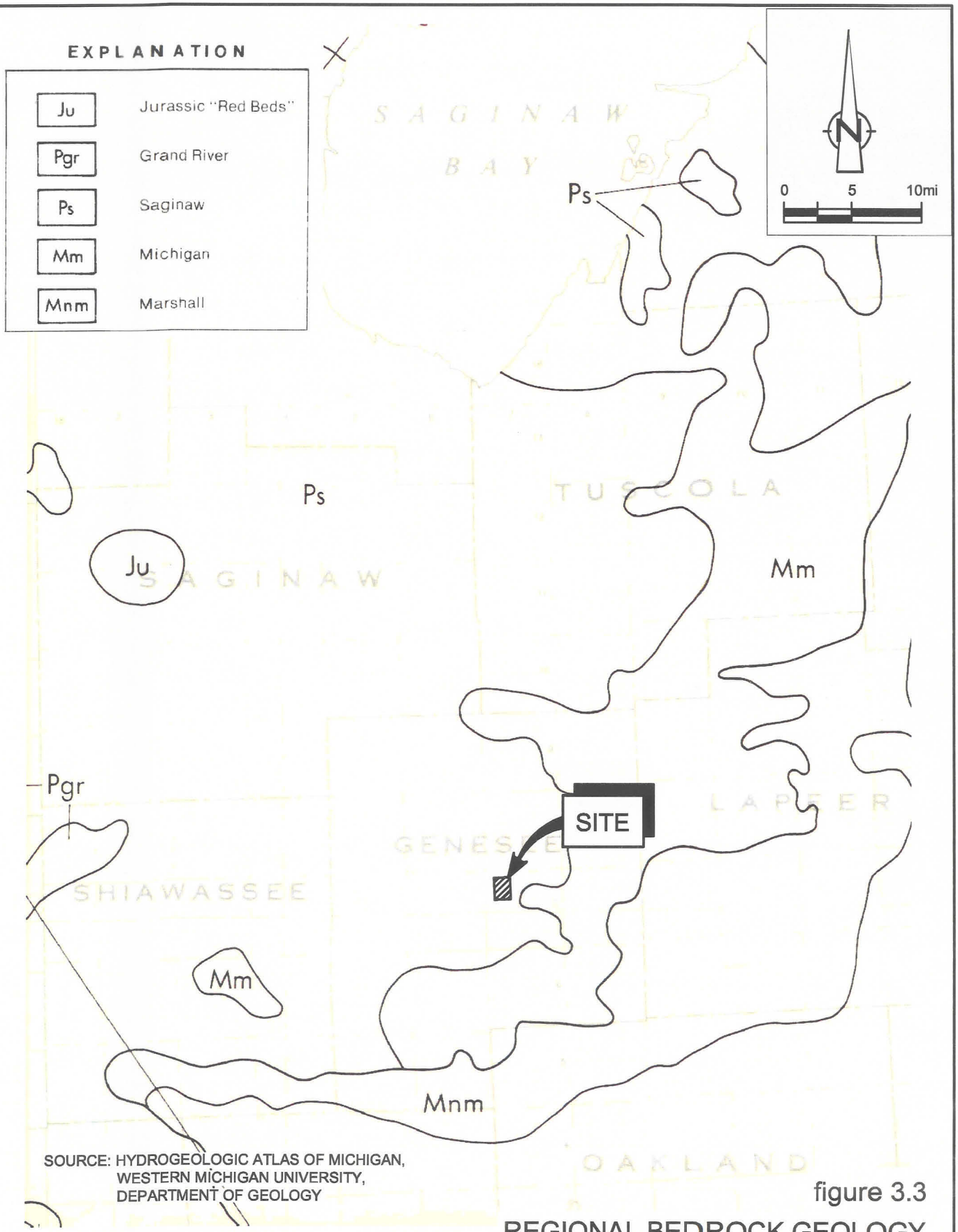


figure 3.2

REGIONAL GLACIAL DRIFT THICKNESS
FORMER PEREGRINE (U.S.) INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA012 MAY 15/2000



SOURCE: HYDROGEOLOGIC ATLAS OF MICHIGAN, WESTERN MICHIGAN UNIVERSITY, DEPARTMENT OF GEOLOGY

figure 3.3

REGIONAL BEDROCK GEOLOGY
FORMER PEREGRINE (US) INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA013 MAY 15/2000

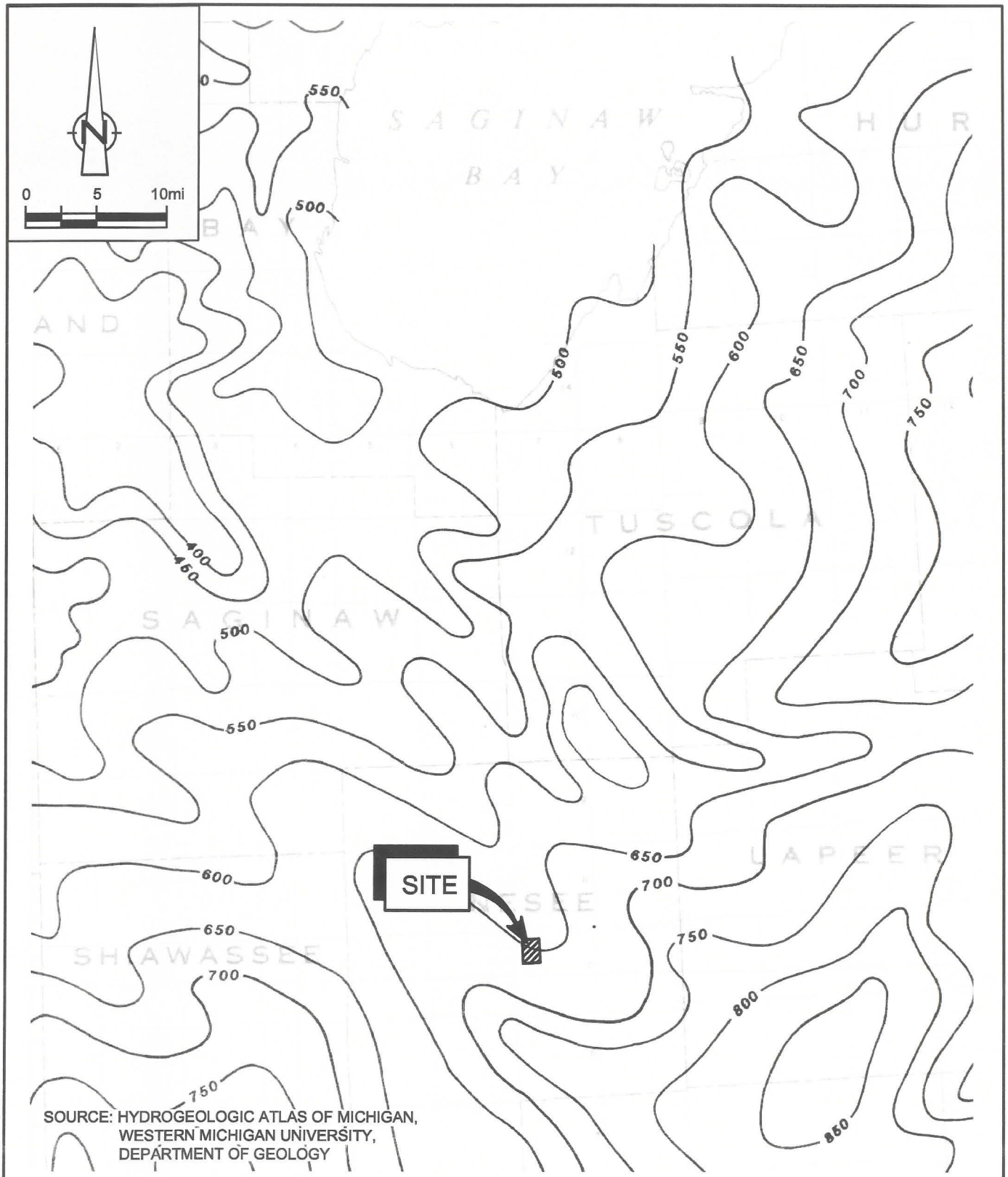
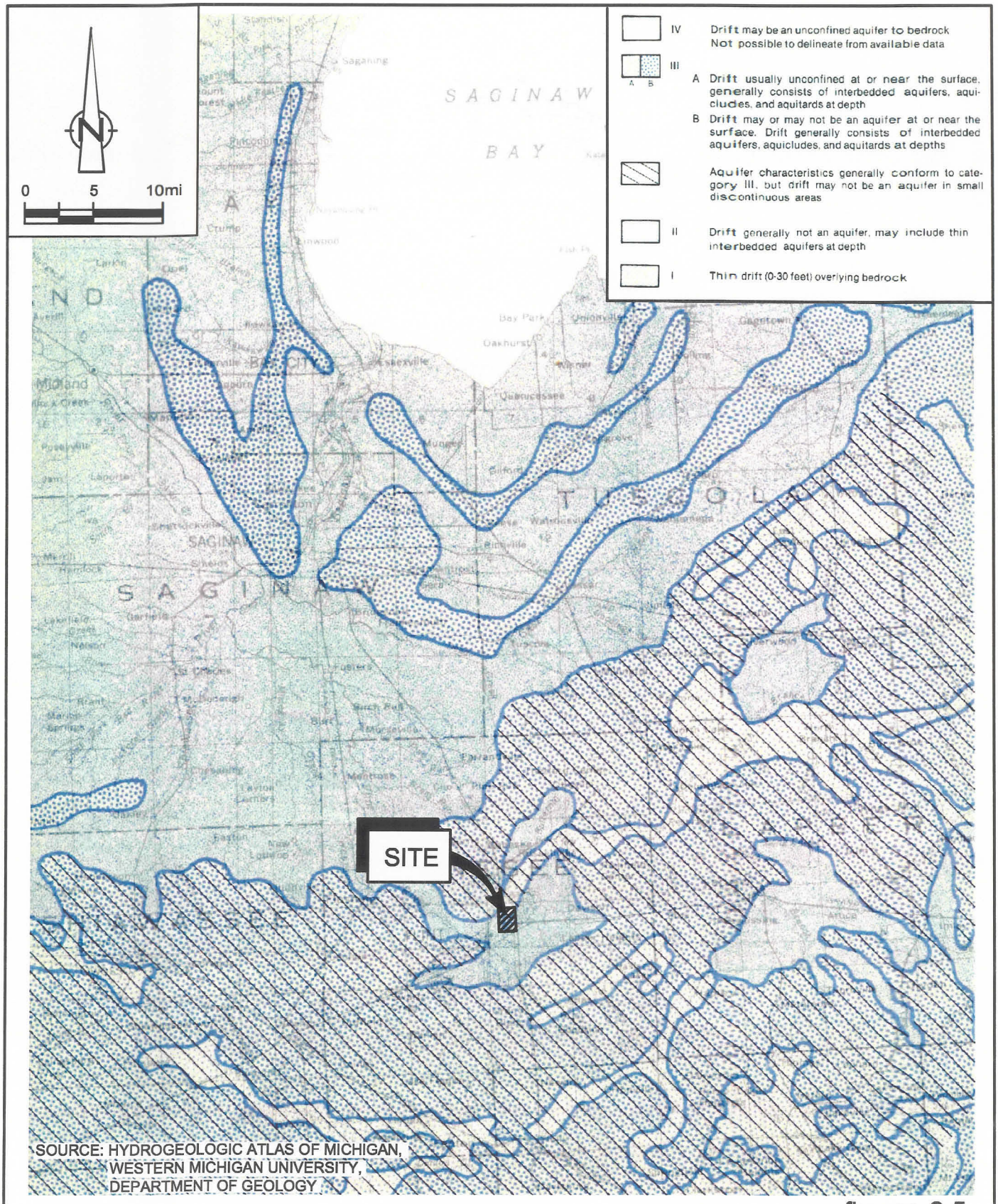


figure 3.4

REGIONAL BEDROCK TOPOGRAPHY
FORMER PEREGRINE (US) INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA014 MAY 15/2000



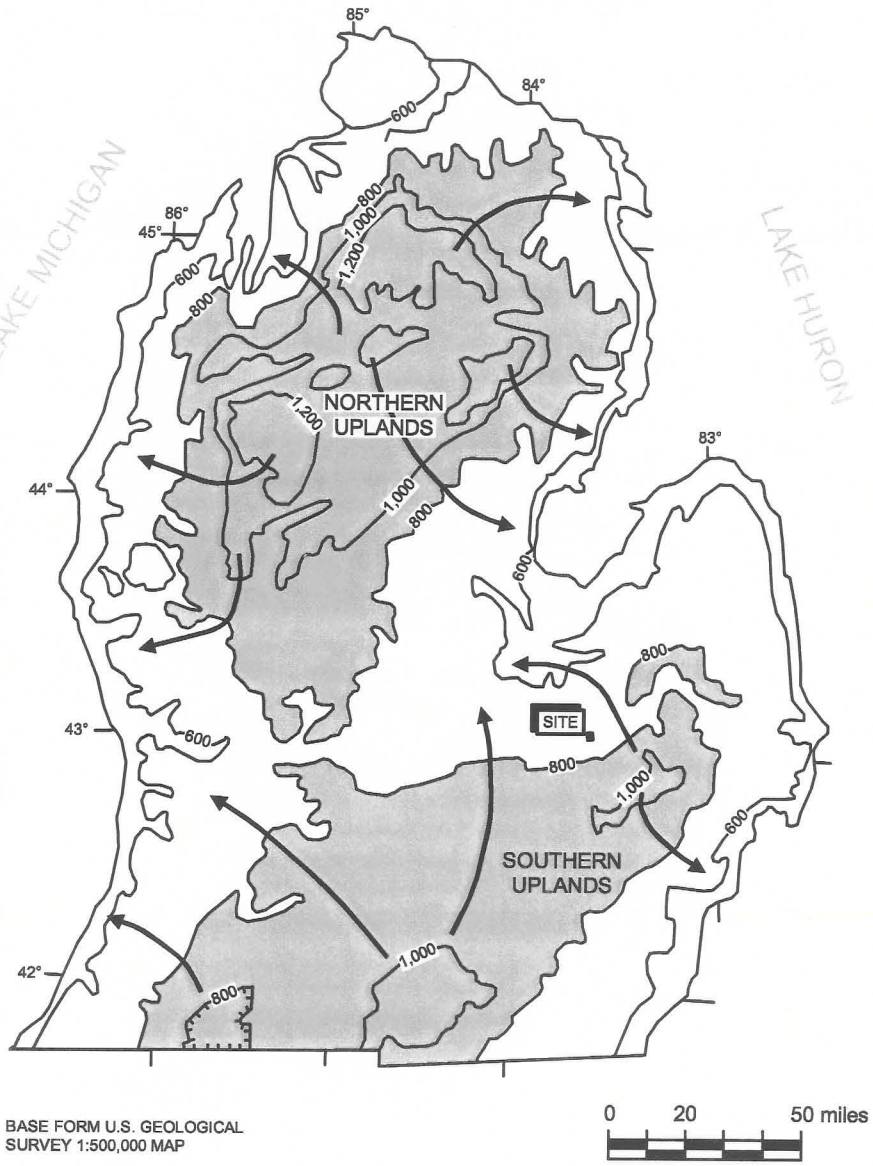
SOURCE: HYDROGEOLOGIC ATLAS OF MICHIGAN, WESTERN MICHIGAN UNIVERSITY, DEPARTMENT OF GEOLOGY

figure 3.5

REGIONAL GLACIAL DRIFT AQUIFER CHARACTERIZATION
FORMER PEREGRINE (US) INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA015 MAY 15/2000



LEGEND

AREA WERE WATER-TABLE ALTITUDE IS GREATER THAN 800 FEET ABOVE SEA LEVEL

800 — WATER-TABLE CONTOUR - SHOWS EQUAL ALTITUDE OF WATER TABLE. HACHURE INDICATES DEPRESSION. CONTOUR INTERVAL 200 FEET. DATUM IS SEA LEVEL.

GROUNDWATER FLOW - ARROW INDICATES DIRECTION OF FLOW

NOTE:

GENERAL WATER-TABLE CONFIGURATION FOR THE GLACIOFLUVIAL AQUIFER IN THE LOWER PENINSULA OF MICHIGAN. (MODIFIED FROM MANDLE AND WESTJOHN, 1989, FIG. 10.)

figure 3.6

REGIONAL GROUNDWATER FLOW
FORMER PERERGINE (US) INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA017 MAY 15/2000

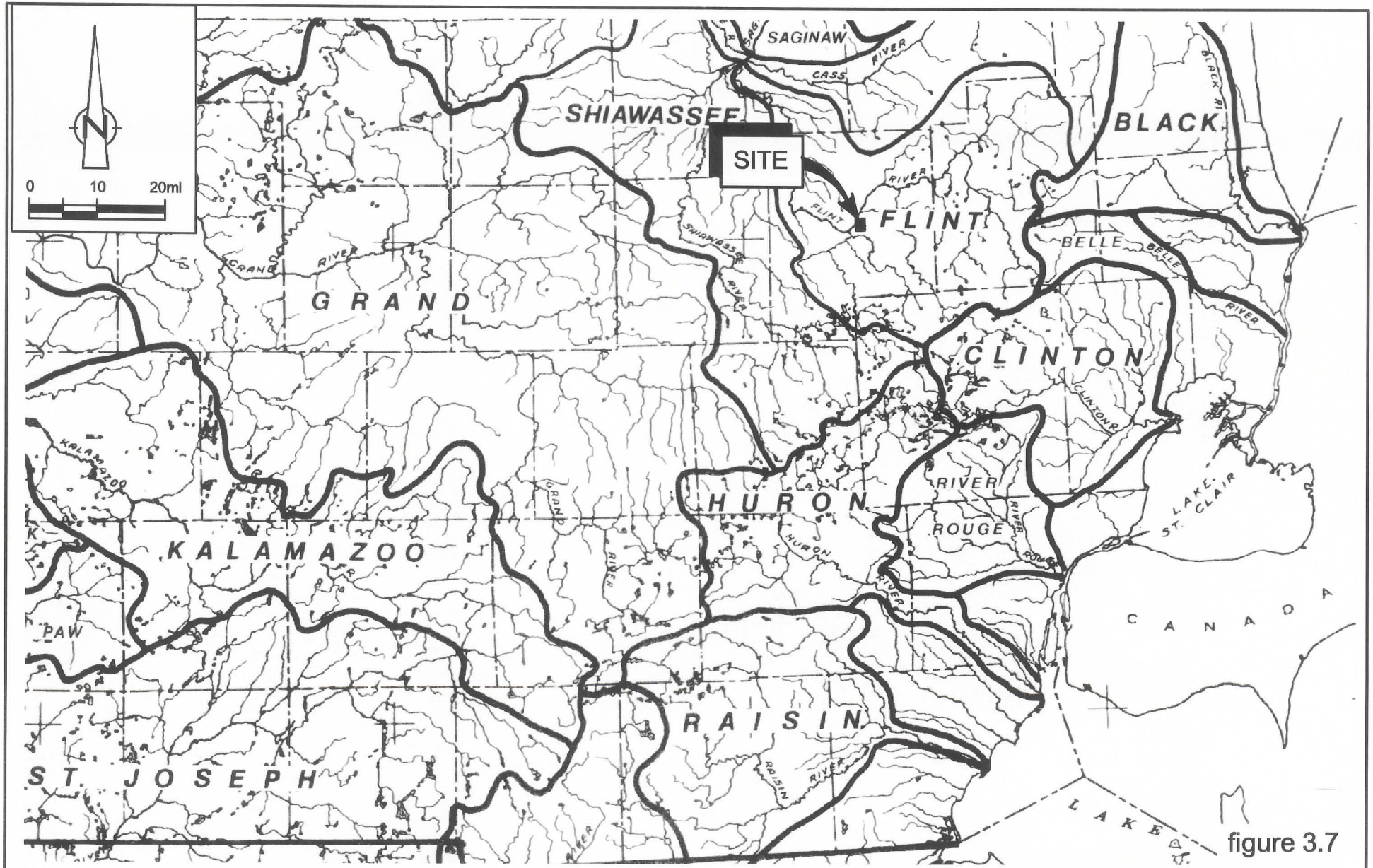


figure 3.7

SOURCE: HYDROGEOLOGIC ATLAS OF MICHIGAN,
WESTERN MICHIGAN UNIVERSITY,
DEPARTMENT OF GEOLOGY

REGIONAL RIVER DRAINAGE BASINS
FORMER PEREGRINE (US) INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA016 MAY 15/2000

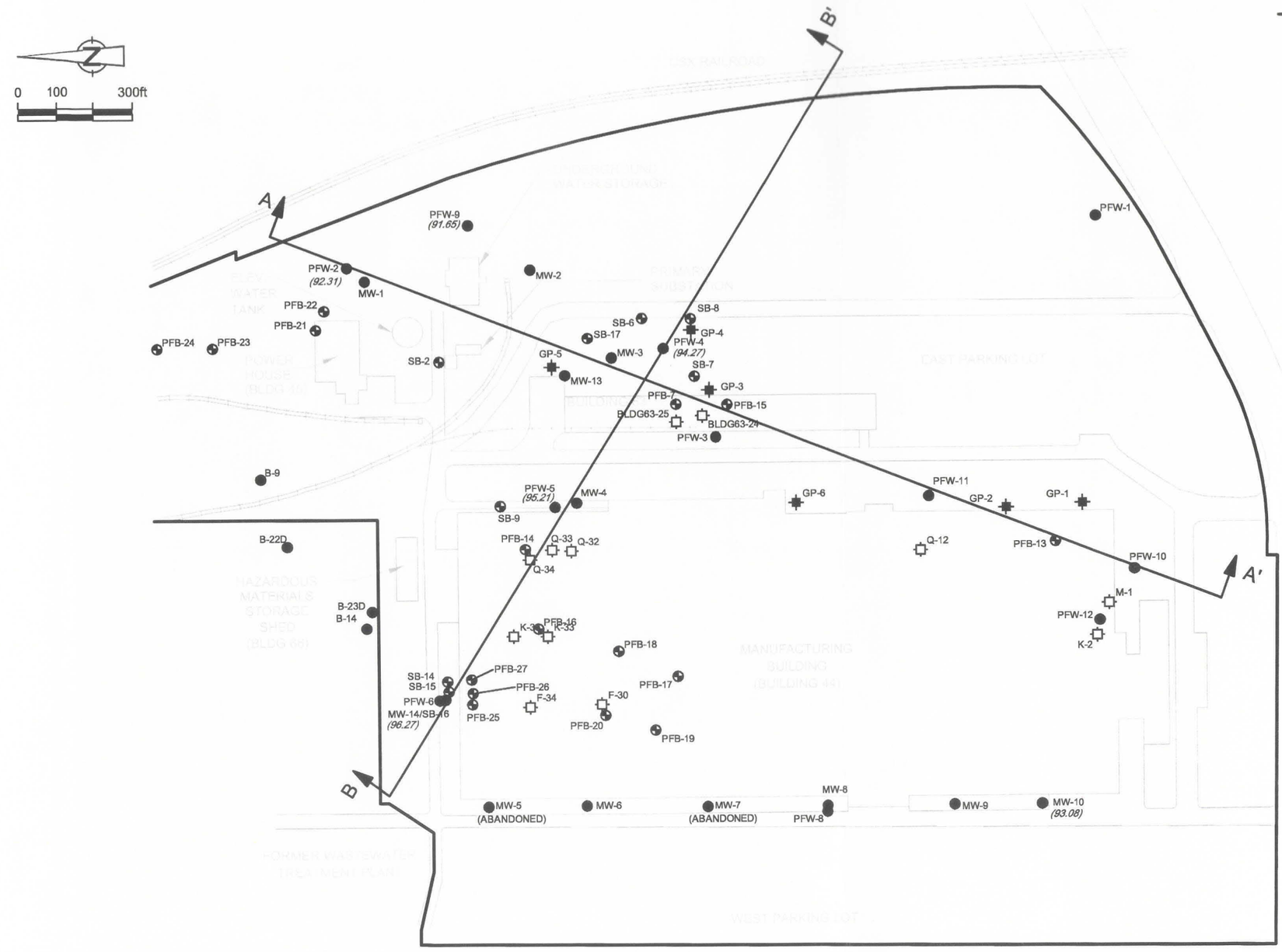
GEOLOGIC UNIT	HYDROGEOLOGIC UNIT	GROUNDWATER DESCRIPTION	THICKNESS (FEET)
SILTY CLAY/ SILTY SAND	PERCHED WATER TABLE ZONE	UNCONFINED PERCHED ZONE	10 - 15
SILT/CLAY	UPPER CONFINING UNIT	AQUITARD	30-50
SAND/ SILTY SAND	OVERBURDEN WATER BEARING ZONE	CONFINED	20-50
SILT/ CLAY TILL	LOWER CONFINING UNIT	AQUITARD	30-75
SANDSTONE/ LIMESTONE/ SHALE	BEDROCK WATER BEARING ZONE	CONFINED	100+

figure 4.1

TYPICAL GEOLOGIC CROSS-SECTION
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA018 MAY 15/2000



LEGEND

- FACILITY BOUNDARY
- MW-7 — STUDY 2 MONITORING WELL LOCATION AND NUMBER
- PFW-12 — STUDY 4 MONITORING WELL LOCATION AND NUMBER
- B-9 — GENERAL MOTORS RCRA UNIT CLOSURE MONITORING WELL LOCATION AND NUMBER
- PFB-17 — STUDY 4 SOIL BORING LOCATION AND NUMBER
- SB-9 — STUDY 2 SOIL BORING LOCATION AND NUMBER
- GP-1 — STUDY 1 GEOPROBE LOCATION AND NUMBER
- Q-34 — STUDY 2 GEOPROBE LOCATION AND NUMBER
- (93.08) — WATER LEVEL

NOTES

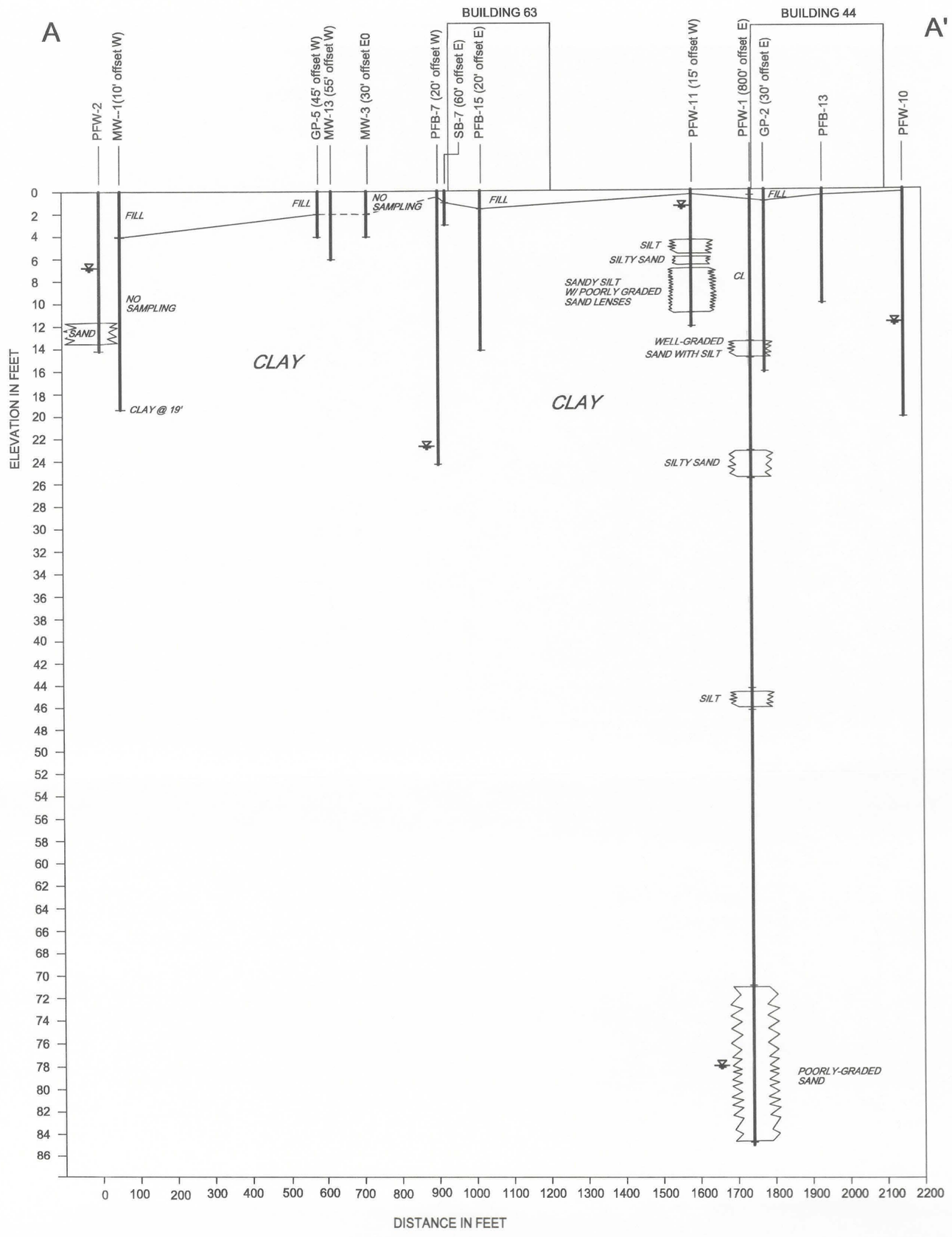
1. INFORMATION SHOWN ON THIS FIGURE TAKEN FROM STUDY 4 (JULY, 1997 REPORT)
2. THIS DRAWING IS FOR REFERENCE ONLY AND IS NEITHER COMPLETE NOR TO EXACTING SCALE.
3. INFERRED WATER TABLE SURFACE IS BASED ON DATA FROM SELECT EXTERIOR WELLS THAT ARE SCREENED ENTIRELY WITHIN THE UPPER 18 FEET OF THE PERCHED AQUIFER. LOCAL VARIATIONS MAY OCCUR IN AREAS NOT SUBJECT TO METEORIC RECHARGE.
4. ELEVATIONS ARE REFERENCED TO AN ON-SITE ARBITRARY DATUM OF 100.00 FEET.

figure 4.2

CROSS SECTION LOCATIONS
FORMER PEREGRINE (US) , INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA019 MAY 15/2000



NOTE

— WATER LEVELS MEASURED 3/31/97

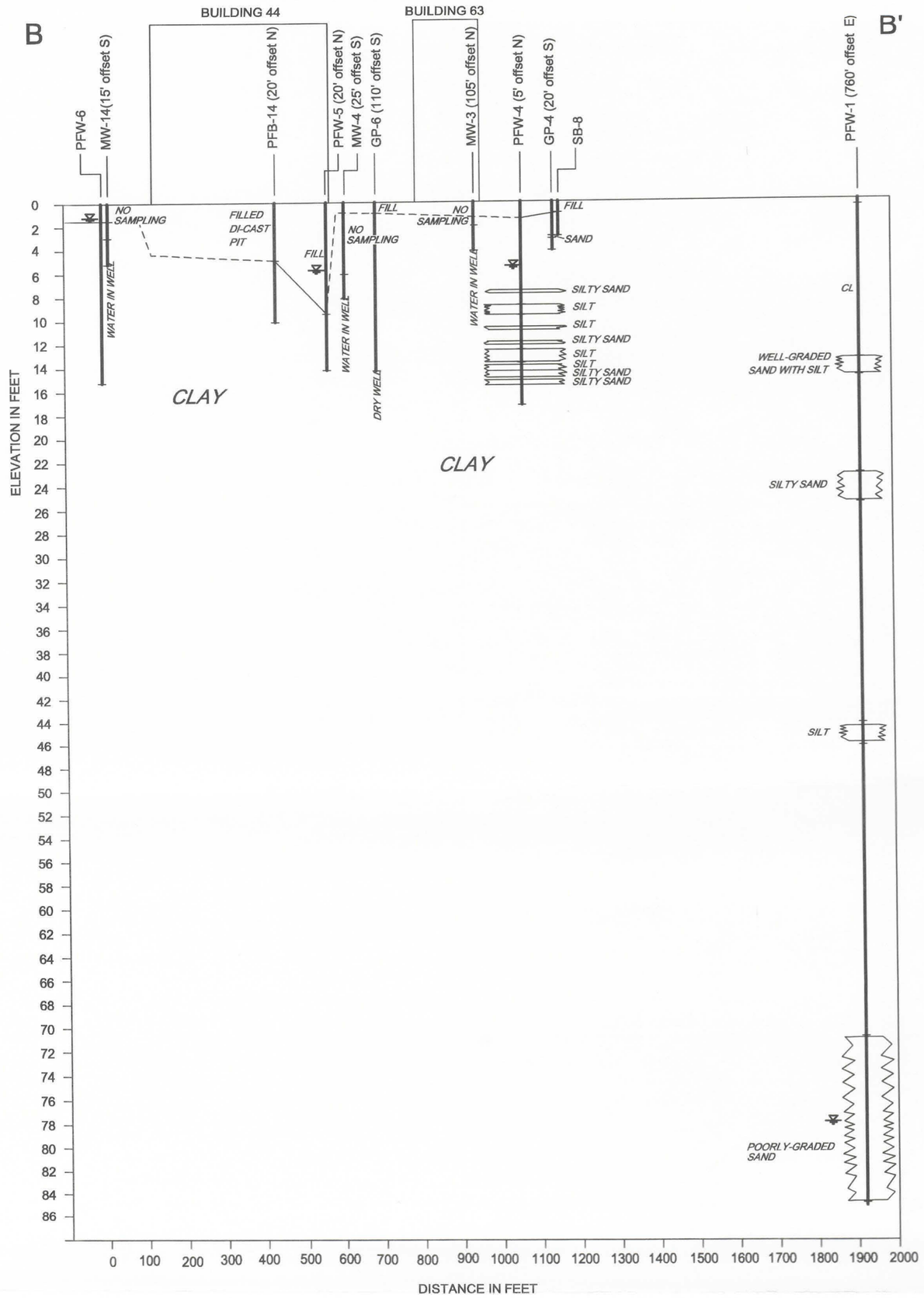
SCALES: HORIZ. 1"=300
VERT. 1"=10'

figure 4.3

GEOLOGIC CROSS-SECTION A-A'
FORMER PEREGRINE(US) INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA025 MAY 15/2000



NOTE

— WATER LEVELS MEASURED 3/31/97

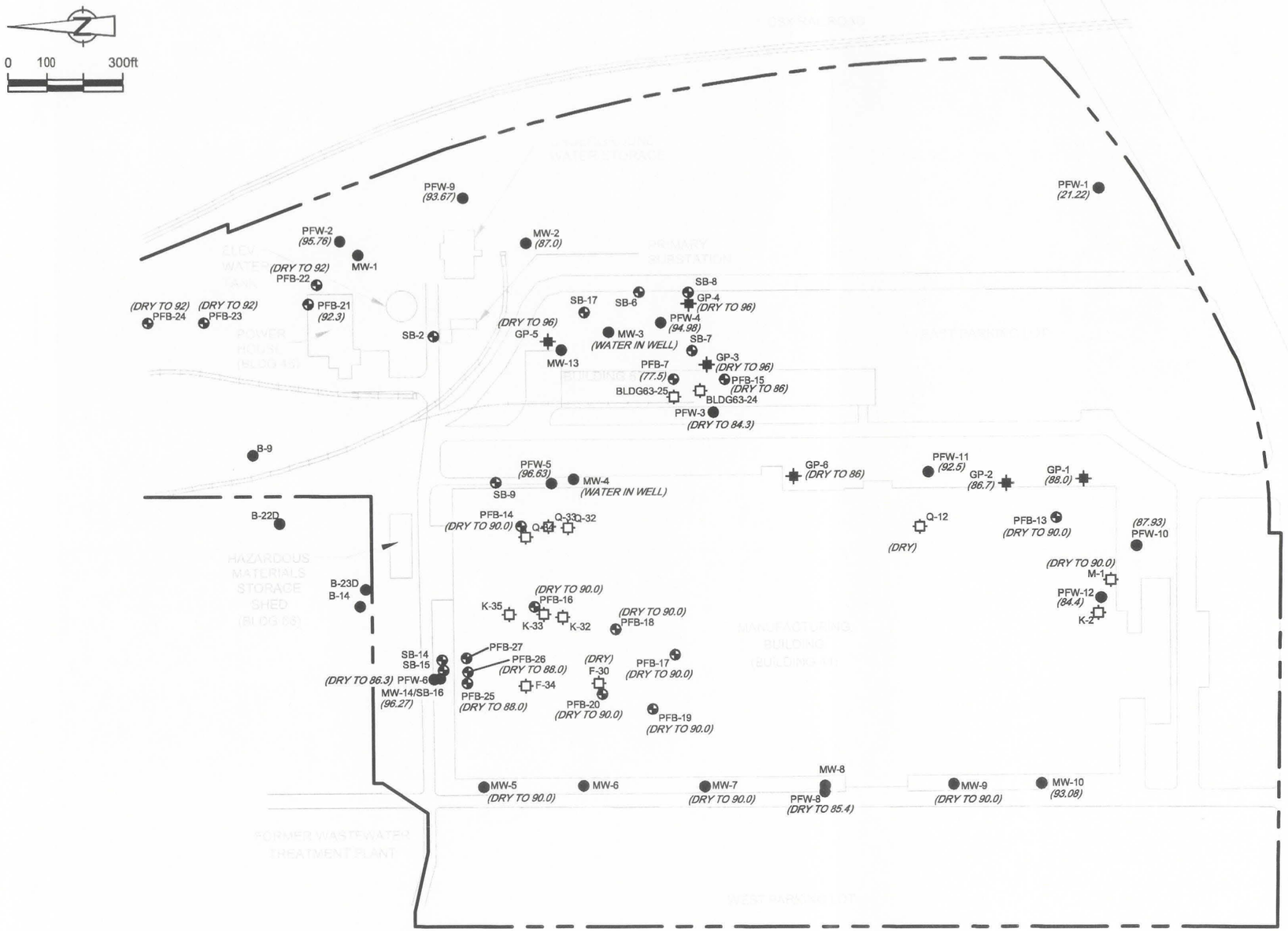
SCALES: HORIZ. 1"=300
VERT. 1"=10'

figure 4.4

GEOLOGIC CROSS-SECTION B-B'
FORMER PEREGRINE(US) INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA020 MAY 15/2000



LEGEND

- FACILITY BOUNDARY
- MW-7 — STUDY 2 MONITORING WELL LOCATION AND NUMBER
- PFW-12 — STUDY 4 MONITORING WELL LOCATION AND NUMBER
- B-9 — GENERAL MOTORS RCRA UNIT CLOSURE MONITORING WELL LOCATION AND NUMBER
- PFB-17 — STUDY 4 SOIL BORING LOCATION AND NUMBER
- SB-9 — STUDY 2 SOIL BORING LOCATION AND NUMBER
- GP-1 — STUDY 1 GEOPROBE LOCATION AND NUMBER
- Q-34 — STUDY 2 GEOPROBE LOCATION AND NUMBER
- (93.08) — WATER LEVEL

NOTES

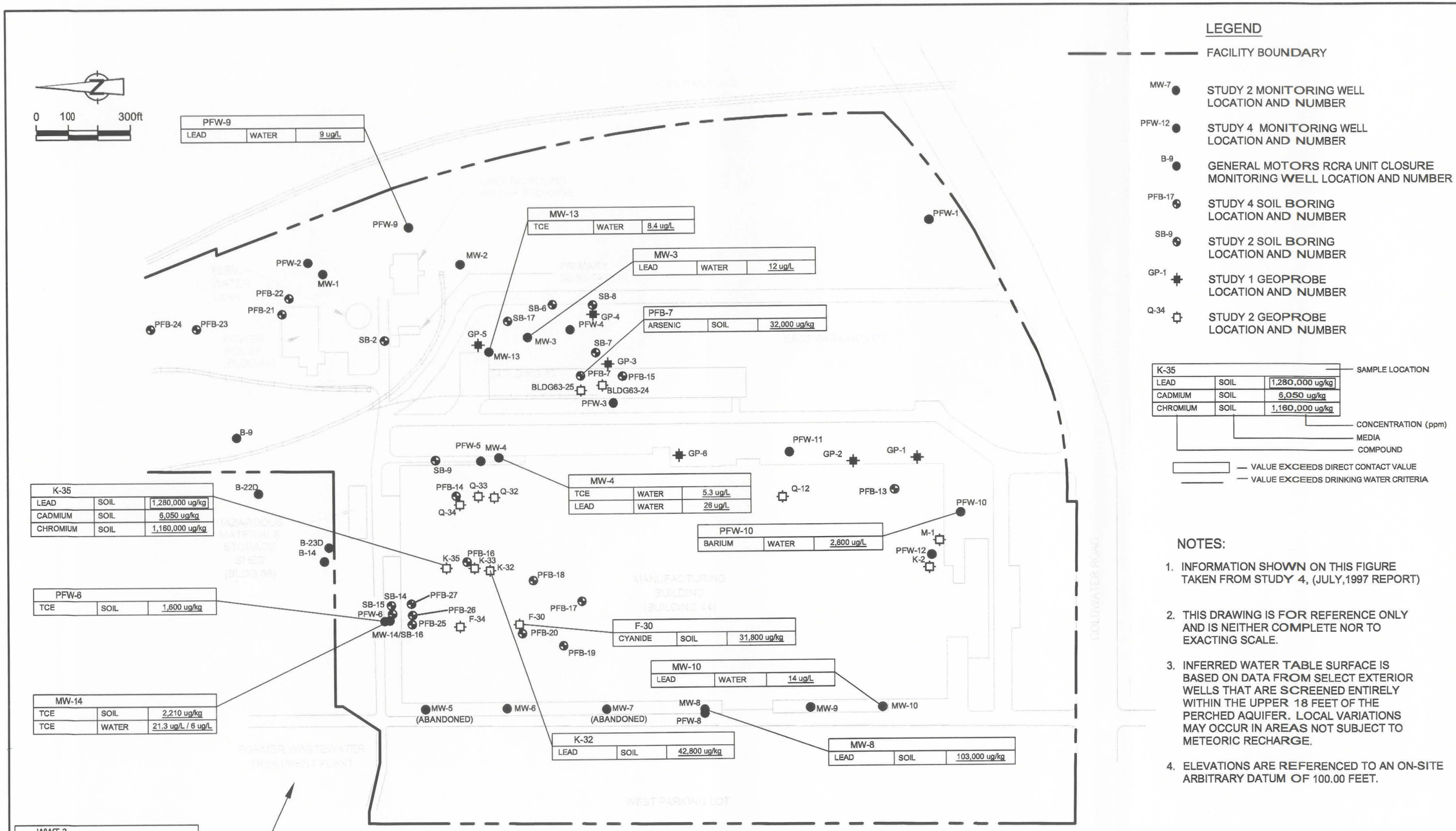
1. THIS DRAWING IS FOR REFERENCE ONLY AND IS NEITHER COMPLETE NOR TO EXACTING SCALE.
2. INFERRED WATER TABLE SURFACE IS BASED ON DATA FROM SELECT EXTERIOR WELLS THAT ARE SCREENED ENTIRELY WITHIN THE UPPER 18 FEET OF THE PERCHED AQUIFER. LOCAL VARIATIONS MAY OCCUR IN AREAS NOT SUBJECT TO METEORIC RECHARGE.
3. WATER ELEVATIONS ARE REFERENCED TO AN ON-SITE ARBITRARY DATUM OF 100.00 FEET FOR WELLS MONITORED BY STUDY 4 (JULY, 1997 REPORT)
   ALL OTHER WATER ELEVATIONS TAKEN FROM DRILLING/DEVELOPMENT INFORMATION WITH GROUND SURFACE ASSUMED AT 100.00 FEET.

figure 4.5

STATIC WATER ELEVATIONS
FORMER PEREGRINE (US) , INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA026 MAY 15/2000



LEGEND

- FACILITY BOUNDARY
- MW-7 — STUDY 2 MONITORING WELL LOCATION AND NUMBER
- PFW-12 — STUDY 4 MONITORING WELL LOCATION AND NUMBER
- B-9 — GENERAL MOTORS RCRA UNIT CLOSURE MONITORING WELL LOCATION AND NUMBER
- PFB-17 — STUDY 4 SOIL BORING LOCATION AND NUMBER
- SB-9 — STUDY 2 SOIL BORING LOCATION AND NUMBER
- GP-1 — STUDY 1 GEOPROBE LOCATION AND NUMBER
- Q-34 — STUDY 2 GEOPROBE LOCATION AND NUMBER

K-35			← SAMPLE LOCATION
LEAD	SOIL	1,280,000 ug/kg	← CONCENTRATION (ppm)
CADMIUM	SOIL	6,050 ug/kg	← MEDIA
CHROMIUM	SOIL	1,160,000 ug/kg	← COMPOUND

— VALUE EXCEEDS DIRECT CONTACT VALUE
— VALUE EXCEEDS DRINKING WATER CRITERIA

NOTES:

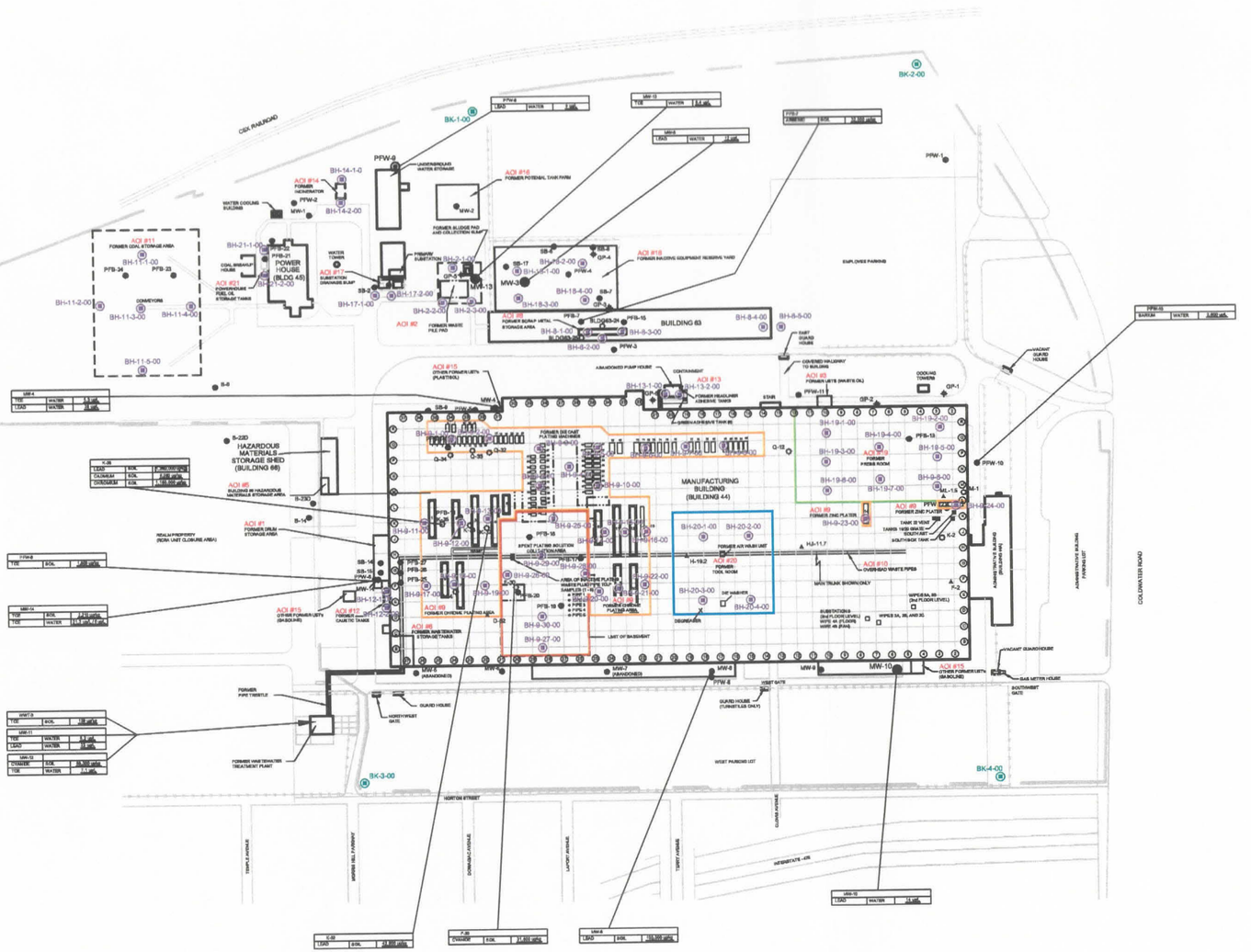
1. INFORMATION SHOWN ON THIS FIGURE TAKEN FROM STUDY 4, (JULY,1997 REPORT)
2. THIS DRAWING IS FOR REFERENCE ONLY AND IS NEITHER COMPLETE NOR TO EXACTING SCALE.
3. INFERRED WATER TABLE SURFACE IS BASED ON DATA FROM SELECT EXTERIOR WELLS THAT ARE SCREENED ENTIRELY WITHIN THE UPPER 18 FEET OF THE PERCHED AQUIFER. LOCAL VARIATIONS MAY OCCUR IN AREAS NOT SUBJECT TO METEORIC RECHARGE.
4. ELEVATIONS ARE REFERENCED TO AN ON-SITE ARBITRARY DATUM OF 100.00 FEET.

figure 5.1

EXCEEDANCES OF ACT 451, PART 201 GENERIC INDUSTRIAL STANDARDS
FORMER PEREGRINE (US) , INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA024 MAY 17/2000


CSX RAILROAD
BK-1-00
BK-2-00
PFW-9
BH-14-1-0
BH-14-2-00
MW-1
PFW-2
AOI #4
FORMER GENERATOR
AOI #16
FORMER POTENTIAL TANK FARM
MW-2
FORMER SLUDGE PAD AND COLLECTION SUMP
PFW-1
AOI #11
FORMER COAL STORAGE AREA
BH-11-1-00
PFB-34
PFB-33
BH-11-2-00
BH-11-3-00
BH-11-4-00
BH-11-5-00
COMMEYORS
AOI #21
POWERHOUSE FUEL OIL STORAGE TANKS
PFB-21
POWER HOUSE (BLDG 40)
BH-21-1-00
BH-21-2-00
BH-21-3-00
WATER TOWER
AOI #17
SUBSTATION DRAINAGE SUMP
BH-17-1-00
BH-17-2-00
SB-2
PRIMARY SUBSTATION
BH-2-1-00
BH-2-2-00
BH-2-3-00
GP-3
MW-13
AOI #2
FORMER WASTE PILE PAD
SB-17
BH-18-1-00
MW-3
SB-8
GP-4
PFW-4
BH-18-2-00
BH-18-4-00
SB-7
GP-5
BH-18-3-00
AOI #18
FORMER INACTIVE EQUIPMENT RESERVE YARD
AOI #8
FORMER SCRAP METAL STORAGE AREA
PFB-7
BLDG63-2F
BLDG63-1F
PFB-15
BUILDING 63
BH-8-1-00
BH-8-2-00
BH-8-3-00
BH-8-4-00
BH-8-5-00
PFW-3
EMPLOYEES PARKING
PFW-10
VACANT GUARD HOUSE
AOI #15
OTHER FORMER USTs (PLATING)
ABANDONED PUMP HOUSE
CONTAINMENT
AOI #13
FORMER HEADLINER ADHESIVE TANKS
BH-13-1-00
BH-13-2-00
COVERED WALKWAY TO BUILDING
AOI #5
FORMER USTS (WASTE OIL)
PFW-11
GP-2
GP-1
COOLING TOWERS
BH-19-1-00
BH-19-2-00
BH-19-3-00
BH-19-4-00
BH-19-5-00
BH-19-6-00
BH-19-7-00
BH-19-8-00
BH-9-5-00
PFB-18
AOI #9
FORMER PRESS ROOM
AOI #9
FORMER ZINC PLATER
PFW-12
TANKS 19/20 GRAVITY SOUTH PART
TANKS 19/20 SOUTH SIDE TANK
BH-6-24-00
K-2
HAZARDOUS MATERIALS STORAGE SHED (BUILDING 66)
S-22D
S-23D
S-23C
S-14
AOI #5
BUILDING 66 HAZARDOUS MATERIALS STORAGE AREA
AOI #1
FORMER DRUM STORAGE AREA
REALM PROPERTY (RCRA UNIT CLOSURE AREA)
MANUFACTURING BUILDING (BUILDING 44)
BH-9-1-00
BH-9-2-00
BH-9-3-00
BH-9-4-00
BH-9-5-00
BH-9-6-00
BH-9-7-00
BH-9-8-00
BH-9-9-00
BH-9-10-00
BH-9-11-00
BH-9-12-00
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BH-9-14-00
BH-9-15-00
BH-9-16-00
BH-9-17-00
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BH-9-23-00
BH-9-24-00
BH-9-25-00
BH-9-26-00
BH-9-27-00
BH-9-28-00
BH-9-29-00
BH-9-30-00
PFB-16
PFB-17
PFB-18
PFB-19
PFB-20
PFB-21
PFB-22
PFB-23
PFB-24
PFB-25
PFB-26
PFB-27
SB-15
SB-16
SB-20
SB-21
SB-22
SB-23
SB-24
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SB-26
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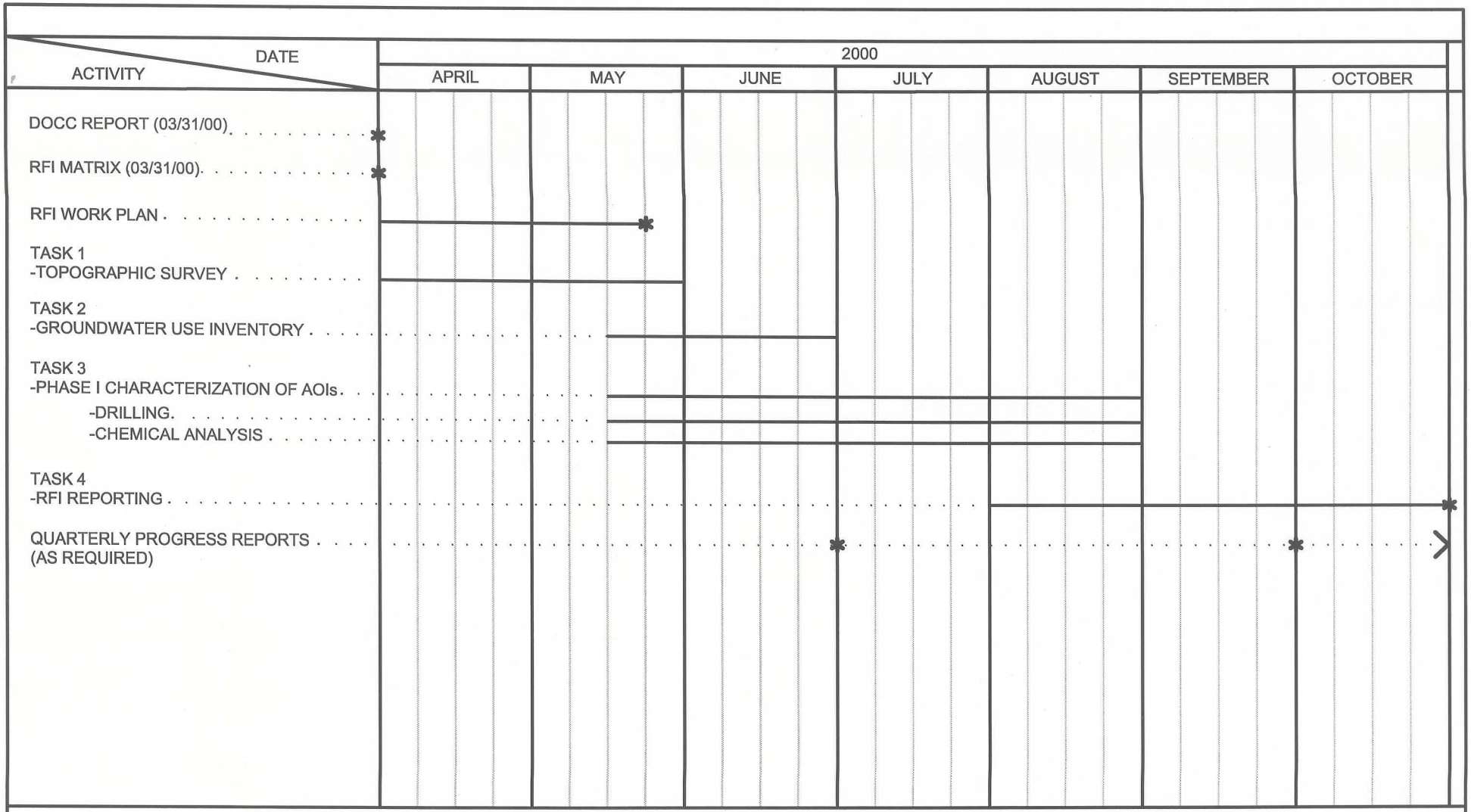
SB-58
SB-59



LEGEND

CONTINUOUS ACTIVITY

MILESTONE EVENT

NOTE

DEPENDENT UPON SITE ACCESS, WEATHER CONDITIONS, AND AGENCY APPROVAL REQUIREMENTS.

CRA

figure 8.1

RFI SCHEDULE
FORMER PEREGRINE (US) , INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

12636-40(011)GN-WA030 MAY 17/2000

TABLE 1.1

SUMMARY OF INVESTIGATION REQUIREMENTS
FORMER PEREGRINE (US), INC.
COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

AOI Number	*AOI Description*	*Investigation Requirements* [1]
AOI 1	Former Drum Storage Area	Ongoing RCRA Closure Sampling by others (outside of this Facility RFI)
AOI 2	Former Waste Pile Pad	Soil & surface soil sampling Re-sampling of existing well Installation and sampling of new monitoring wells if a significant aquifer is identified
AOI 3	Former USTs	No investigation required
AOI 4	Former Satellite Accumulation Areas	No investigation required
AOI 5	Former Hazardous Materials Storage Area	No investigation required
AOI 6	Former Wastewater Storage Tanks	No investigation required
AOI 7	Former Scrap Metal Management System	No investigation required
AOI 8	Former Scrap Metal Storage Area	Soil and surface soil sampling Installation and sampling of new monitoring wells if a significant aquifer is identified In addition, the investigation at AOI #8 will include 2 boreholes at the southern end of Building 63 (as requested by MDEQ-WMD)
AOI 9	Former Plating Areas	Soil and surface soil sampling Installation and sampling of new monitoring wells if a significant aquifer is identified
AOI 10	Overhead Waste Pipes	No investigation required

TABLE 1.1

SUMMARY OF INVESTIGATION REQUIREMENTS
FORMER PEREGRINE (US), INC.
COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

AOI Number	*AOI Description*	*Investigation Requirements* [1]
AOI 11	Former Coal Storage Area	Soil and surface soil sampling Installation and sampling of new monitoring wells if a significant aquifer is identified
AOI 12	Former Caustic Tanks	Soil and surface soil sampling Installation and sampling of new monitoring wells if a significant aquifer is identified
AOI 13	Former Headliner Adhesive Tanks	Soil and surface soil sampling Installation and sampling of new monitoring wells if a significant aquifer is identified
AOI 14	Former Incinerator	Soil and surface soil sampling Installation and sampling of new monitoring wells if a significant aquifer is identified
AOI 15	Other Former USTs	No investigation required
AOI 16	Former Potential Tank Farm	No investigation required
AOI 17	Substation Drainage Sump	Soil and surface soil sampling Installation and sampling of new monitoring wells if a significant aquifer is identified
AOI 18	Former IERY	Soil and surface soil sampling Resampling of existing well Installation and sampling of new monitoring wells if a significant aquifer is identified
AOI 19	Former Press Room	Soil and surface soil sampling Installation and sampling of new monitoring wells if a significant aquifer is identified

TABLE 1.1

SUMMARY OF INVESTIGATION REQUIREMENTS
FORMER PEREGRINE (US), INC.
COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

AOI Number	*AOI Description*	*Investigation Requirements* [1]
AOI 20	Former Tool Room	Soil and surface soil sampling Installation and sampling of new monitoring wells if a significant aquifer is identified
AOI 21	Powerhouse Fuel Oil Storage Tanks	Soil and surface soil sampling Installation and sampling of new monitoring wells if a significant aquifer is identified
Other	Non-specific	Resampling of existing wells

Note:

(1) Refer to Description of Current Conditions Report.

TABLE 5.1

PROPOSED RFI SUMMARY
FORMER PEREGRINE (US), INC.
COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

AOI Number	*AOI Description*	*Selection Rationale*	*Previous Investigation*	*# Boreholes or Wells*	*Total # of Samples*	*Exceedance of Standards*					*Proposed # of Boreholes/Samples (1)*	*Analytical Parameters Comments (2)(3)*
						Matrix	*Location*	*Parameter*	*Concentration*	*Standard*		
Bkgd	Background	Establish Background	No	0	0	--No sampling performed--					4/8	TAL Grain Size Hydraulic Conductivity (K)
AOI 1	Former Drum Storage Area	U.S. EPA SWMU 1	Yes	6 2	8 4	Soil Water	MW-14/PFB-6 MW-14	TCE TCE	2,210/1,600 µg/kg 21.3/6.0 µg/L	IDWP IDWC	None	Ongoing RCRA Closure Sampling (To Be Conducted by Others)
AOI 2	Former Waste Pile Pad	U.S. EPA SWMU 2	Yes	1 1	1 2	Soil Water	MW-13	TCE	No exceedances 8.4 µg/L	IDWC	3/6 Resample	VOC/SVOC/PCB/TAL VOC
AOI 3	Former USTs	U.S. EPA SWMU 3	Yes	1 1	1 1	Soil Water			No exceedances No exceedances		None	No sampling proposed (closed)
AOI 4	Former Satellite Accumulation Areas	U.S. EPA SWMU 4	No	0	0	- - No sampling performed - -					None	No sampling proposed U.S. EPA identified low potential for concern Addressed through facility - wide sampling
AOI 5	Former Hazardous Materials Storage Area	U.S. EPA SWMU 5	No	0	0	- - No sampling performed - -					None	No sampling proposed AOI 5 cleaned by Peregrine U.S. EPA identified low potential for concern
AOI 6	Former Wastewater Storage Tanks	U.S. EPA SWMU 6	No	0	0	- - No sampling performed - -					None	No sampling proposed Tanks removed by Peregrine U.S. EPA identified low potential for concern
AOI 7	Former Scrap Metal Management System	U.S. EPA SWMU 7	Yes	8 1	10 1	Soil Water	PFW-10	Barium	No exceedances 2,600 µg/kg	IDWC	None	No sampling proposed (see AOI #19)
AOI 8	Former Scrap Metal Storage Area	U.S. EPA SWMU 8	Yes	5 1	5 0	Soil Water	PFB-7	Arsenic	32,000 µg/kg Well was dry	IDWP	5/10 [4]	VOC/SVOC/PCB/TAL Includes 2 boreholes at the southern end of Building 63
AOI 9	Former Plating Areas	U.S. EPA SWMU 9	Yes	12 1	15 1	Soil Water	K-32 K-35 K-35 K-35 F-30	Lead Cadmium Chromium Lead Cyanide	42,800 µg/kg 6,050 µg/kg 1,160,000 µg/kg 1,280,000 µg/kg 31,800 µg/kg No exceedances	IDWP IDWP IDWP IDWP/IDC IDWP	30/60	VOC/TAL Modified Appendix IX (2 samples)

TABLE 5.1

PROPOSED RFI SUMMARY
FORMER PEREGRINE (US), INC.
COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

AOI Number	AOI Description	Selection Rationale	Previous Investigation	# Boreholes or Wells	Total # of Samples	Exceedance of Standards					Proposed # of Boreholes/Samples (1)	Analytical Parameters Comments (2)(3)
						Matrix	Location	Parameter	Concentration	Standard		
AOI 10	Overhead Waste Pipes	U.S. EPA SWMU 10	Yes	--	5	Waste-TCLP			No exceedances		None	No sampling proposed
AOI 11	Former Coal Storage Area	U.S.EPA AOC	Yes	2 0	2 0	Soil Water			No exceedances No samples collected		5/10 [5]	SVOC/PCB/TAL
AOI 12	Former Caustic Tanks	U.S.EPA AOC	No (moderate potential)	0	0	-- No sampling performed --					2/4	pH/VOC/TAL
AOI 13	Former Headliner Adhesive Tanks	REALM AOI	Yes	1 0 --	2 0 1	Soil Water Waste-TCLP	 AST No. 6 AST No. 6	 Benzene Flammability	No exceedances No samples collected 79000 µg/L <78 F	 Haz Waste Disposal	2/4 None	VOC
AOI 14	Former Incinerator	REALM AOI	Yes	2 2	2 3	Soil Water			No exceedances No exceedances		2/4	SVOC/PCB/TAL
AOI 15	Other Former USTs	REALM AOI	Yes	4	5	Soil			No exceedances		None	No sampling proposed (closed)
AOI 16	Former Potential Tank Farm	REALM AOI	Yes	2	2	Soil			No exceedances		None	No sampling proposed No storage tanks ever installed
AOI 17	Substation Drainage Sump	REALM AOI	Yes	1 0	2 0	Soil Water			No exceedances No samples collected		2/4 [6]	SVOC/PCB
AOI 18	Former IERY	REALM AOI	Yes	8 2	10 4	Soil Water	 MW-3	 Lead	No exceedances 12 µg/L	 IDWC	4/8 [7] Resample	VOC/SVOC/PCB/TAL Lead only
AOI 19	Former Press Room	REALM AOI	Yes	2 0	2 0	Soil Water			No exceedances No samples collected		8/16	VOC/SVOC/PCB/TAL Modified Appendix IX (1 sample)
AOI 20	Former Tool Room	REALM AOI	Yes	0 0 --	0 0 1	Soil Water Waste-TCLP	 Air Wash Unit Air Wash Unit	 Chromium Lead	No samples collected No samples collected 8800 µg/L 6100 µg/L	 Haz Waste Disposal	4/8	VOC/SVOC/PCB/TAL Modified Appendix IX (1 sample)

TABLE 5.1

PROPOSED RFI SUMMARY
FORMER PEREGRINE (US), INC.
COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

AOI Number	*AOI Description*	*Selection Rationale*	*Previous Investigation*	*# Boreholes or Wells*	*Total # of Samples*	*Exceedance of Standards*					*Proposed # of Boreholes/Samples (1)*	*Analytical Parameters Comments (2)(3)*
						Matrix	*Location*	*Parameter*	*Concentration*	*Standard*		
AOI 21	Powerhouse Fuel Oil Storage Tanks	REALM AOI	Yes	1 1	1 1	Soil Water			No exceedances No exceedances		2/4	VOC/SVOC/TAL
Other	Non-specific	Previous Sampling	Yes	3	4	Water Water Water	MW-4 MW-4 MW-10	TCE Lead Lead	5.3 µg/L 26 µg/L 14 µg/L	IDWC IDWC IDWC	Resample Resample Resample	VOCs Lead only Lead only
				2	3	Water	PFW-9	Lead	9.0 µg/L	IDWC	Resample	Lead only
		TOTAL # BOREHOLES & SAMPLES (performed during previous investigations)		**73**	**99**						**TOTAL # BOREHOLES/SAMPLES** (to be performed during proposed RFI)	**73/146**

Notes:

(1) It is expected that a maximum of two soil samples will be collected from each borehole. The first sample will be collected at the 0 to 2 foot bgs horizon. The second sample will be collected at the interval exhibiting the greatest degree of staining or positive response on the PID. If no staining or PID response is encountered, the lower soil sample may not be collected. All boreholes will be installed to a depth of approximately 5 feet into the native clay till horizon. All boreholes will be sampled continuously to define stratigraphy and the potential presence of a continuous perched water table zone.

(2) pH - pH
VOC - Target Compound List Volatile Organic Compounds
SVOC - Target Compound List Semi-Volatile Organic Compounds
PCB - Target Compound List Polychlorinated Byphenols
TAL - Target Analyte List Inorganics (include both total and hexavalent chromium)

(3) A perched water table zone exists in isolated locations of the near surface at the Facility. Perched water table monitoring wells will be installed during a second phase of sampling if a significant groundwater aquifer is identified at each of the AOIs. A significant groundwater aquifer may be present if groundwater is identified to be continuous over the Facility and the formation is capable of producing a significant yield (eg. 5 feet of porous media, 2 feet saturated, continuous horizontally). The exact location and number of monitoring wells will be determined after the boreholes have been completed.

(4) The boreholes will be completed either through or immediately adjacent to the former scrap metal collection bins.

(5) The boreholes will be completed after the residual coal has been removed.

(6) The boreholes will be completed either through or immediately adjacent to the substation drainage sump.

(7) Borehole locations will be biased to low areas with broken/cracked concrete or asphalt.

APPENDIX A

HISTORICAL SOIL BORING AND MONITORING WELL LOGS

APPENDIX A

LIST OF BOREHOLE AND MONITORING WELL LOGS
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Boring/ Well ID	*Date Completed*
Study 1 (08/96)	
GP-1	08/30/96
GP-2	08/30/96
GP-3	08/30/96
GP-4	08/30/96
GP-5	08/30/96
GP-6	08/30/96
GP-7	08/30/96
Study 4 (03,04/97)	
PFW-1	03/13/97
PFW-2	03/14/97
PFW-3	03/17/97
PFW-4	03/17/97
PFB-5	03/18/97
PFW-5	03/18/97
PFW-6	03/18/97
PFB-7	03/18/97
PFW-8	03/19/97
PFW-9	03/19/97
PFW-10	03/20/97
PFW-11	03/20/97
PFW-12	03/21/97
PFB-13	03/24/97
PFB-14	03/24/97
PFB-15	03/24/97
PFB-16	03/25/97
PFB-17	03/26/97
PFB-18	03/26/97
PFB-19	03/26/97
PFB-20	03/26/97
PFB-21	04/24/97
PFB-22	04/24/97
PFB-23	04/24/97
PFB-24	04/24/97
PFB-25	04/24/97
PFB-26	04/25/97
PFB-27	04/25/97

ENVIRONMENTAL AUDIT REPORT: PRIVILEGED DOCUMENT

General Motors Corporation
Phase II Environmental Site Assessment

September 1996

SOIL BOREHOLE LOG

E NAME & LOCATION:
GM Delphi
Coldwater Road Plant
Press Room (outside)
South End GP-1

DRILLING METHOD: Geoprobe
SAMPLING METHOD: 4' Continuous sampler

BORING NO. GP-1
SHEET 1 OF 1

	DRILLING START	DRILLING FINISH
WATER LEVEL		
TIME	Time 1125	Time 1200
DATE	Date 8/30/96 →	Date
CASING DEPTH		

TUM ELEVATION
ILL RIG
GLE BEARING
MPLE HAMMER TORQUE Ft.-Lbs.

SURFACE CONDITIONS: Grassy area near (2") water main for sprinkling system adjacent to east side of Press Room

Drilling Contractor Stearns Drilling

EPTH FEET vation)	BLOWS / 6 in. ON SAMPLER (Recovery)	RECOVERY	SAMPLE NUMBER AND DESCRIPTION OF MATERIAL	GRAPHIC SYMBOL	DEPTH IN FEET From	DEPTH IN FEET To	DESCRIPTION OF OPERATION AND REMARKS
1							
2	NO SAMPLE						
3							
4			Brown St.ff Clay w/ some sand and a few pebbles slight sheen on inside of plastic sample tube @ 7'. Collected composite soil sample for DRO TPH and MI 10 Metals.				
5							Sample GP-1 4'-8' Composite
6		100%					
7							
8							
9			Brown / Gray Stiff Clay				
10							
11		100%					
12			Saturated @ ~ 11.5' Brown fine wet sand.		12		
13					13		Temporary well Screened from 12-14' did not produce enough water to

Logged By Angela Cook
Date 8/30/96
Chk'd By

~~ENVIRONMENTAL AUDIT REPORT: PRIVILEGED DOCUMENT~~

~~General Motors Corporation~~
~~Phase II Environmental Site Assessment~~

September 1996

SOIL BOREHOLE LOG

TE NAME & LOCATION:
GM Delphi
Coldwater Road Plant
Press Room (outside)
GP-2

DRILLING METHOD: Geoprobe
SAMPLING METHOD: 4' Continuous

BORING NO. GP-2
SHEET 1 OF 1

	DRILLING START	DRILLING FINISH
WATER LEVEL		
TIME	Time 1230	Time 1310
DATE	Date 8/30/96	Date 8/30/96
CASING DEPTH		

DATUM ELEVATION
DRILL RIG
ANGLE BEARING
SAMPLE HAMMER TORQUE Ft-Lbs.

SURFACE CONDITIONS: Gravel area adjacent to the east side of the press room next to piping outfall w/ concrete catchment pad.

Drilling Contractor Sterns Drilling

Logged By Angela Cook

DEPTH IN FEET (Elevation)	BLOWS / 6 in. ON SAMPLER (Recovery)	RECOVERY	SAMPLE NUMBER AND DESCRIPTION OF MATERIAL	GRAPHIC SYMBOL	DEPTH IN FEET From	DEPTH IN FEET To	DESCRIPTION OF OPERATION AND REMARKS
		50%	Gravelly slightly wet, sheen inside of sample tube from 0-1'				
2		100%	Brown stiff clay with some pebbles. Does not appear to be impact past 1' by visual inspection. No odor				Sample taken 0-2' composite for DRO TPH and MI 10 Metals
3							
4			From 4'→ until hit water table				
			Tried to collect a water sample from 11-13' did not produce. 12-14' saturated and very silty, did not produce. 14-16' drove back into clay and could not collect a water				

SOIL BOREHOLE LOG

TE NAME & LOCATION: GM Delphi Coldwater Road Plant Bone Yard GP-3

DRILLING METHOD: Geoprobe

SAMPLING METHOD: 4' Continuous Sampler

BORING NO. GP-3

SHEET 1 OF 1

	DRILLING START	DRILLING FINISH
WATER LEVEL		
TIME	Time 1320	Time 1445
DATE	Date 8/30/96	Date 8/30/96
CASING DEPTH		

ATUM ELEVATION

RILL RIG

NGLE BEARING

AMPLE HAMMER TORQUE Ft.-Lbs.

SURFACE CONDITIONS: 10" of concrete. Visable hydraulic oil ponded on surface

Drilling Contractor Stone

DEPTH IN FEET (Elevation)	BLOWS / 6 in. ON SAMPLER (Recovery)	RECOVERY	SAMPLE NUMBER AND DESCRIPTION OF MATERIAL	GRAPHIC SYMBOL	DEPTH IN FEET From	DEPTH IN FEET To	DESCRIPTION OF OPERATION AND REMARKS
-1			10" of concrete w/ rebar				Sampled Composite 1-4' • Metals • DRO TPH • VOC's
-2			Lt. Brown fill sand. No odor				
-3		80%	↓				
-4							

Logged By Douglas Castle

ENVIRONMENTAL AUDIT REPORT PRIVILEGED DOCUMENT

Geraghty & Miller, Inc.
Environmental Services

SOIL BOREHOLE LOG

TE NAME & LOCATION: GM Delphi Coldwater Road Plant Boneyard GP-4

DRILLING METHOD: Geoprobe
SAMPLING METHOD: 4" Macro Core Sampler
BORING NO. GP-4
SHEET 1 OF 1

	DRILLING START	DRILLING FINISH
WATER LEVEL		
TIME	Time 1500	Time 1556
DATE	Date 8/30/96	Date 8/30/96
CASING DEPTH		

ATUM ELEVATION
RILL RIG
NGLE BEARING
AMPLE HAMMER TORQUE Ft.-Lbs.

SURFACE CONDITIONS: Concrete pad, stored press equipment leaking hydraulic oil

DEPTH IN FEET (Elevation)	BLOWS / 6 in. ON SAMPLER	RECOVERY (Inches)	SAMPLE NUMBER AND DESCRIPTION OF MATERIAL	GRAPHIC SYMBOL	DEPTH IN FEET From	DEPTH IN FEET To	DESCRIPTION OF OPERATION AND REMARKS
-1			2" Concrete on surface				0-4' Composite taken for · VOCs · DRO TPH · Metals
-2			Stiff brown clay				
-3		90%	Sandy lense at 3' Dark brown moist sand.				
-4			Stiff clay from 3.5-4'				

Drilling Contractor: Geoprobe

ENVIRONMENTAL AUDIT REPORT: PRIVILEGED DOCUMENT

General Motors Corporation
Phase II Environmental Site Assessment

SOIL BOREHOLE LOG

TE NAME & LOCATION: GM Delphi Coldwater Road Plant Boneyard GP-5

DRILLING METHOD: Geoprobe
SAMPLING METHOD: 4' continuous Sleeve
BORING NO. GP-5
SHEET 1 OF 1

					DRILLING START	DRILLING FINISH
WATER LEVEL						
TIME					Time 1520	Time 1530
DATE					Date 8/30/00	Date 8/30/00
CASING DEPTH						

ATUM ELEVATION
RILL RIG
NGLE BEARING
AMPLE HAMMER TORQUE Ft.-Lbs.

SURFACE CONDITIONS: 1" Dirt over 2" concrete. Visually stained soil scrap area

Drilling Contractor Stergis

DEPTH IN FEET (Elevation)	BLOWS / 6 In. ON SAMPLER (Recovery)	RECOVERY	SAMPLE NUMBER AND DESCRIPTION OF MATERIAL	GRAPHIC SYMBOL	DEPTH IN FEET From	DEPTH IN FEET To	DESCRIPTION OF OPERATION AND REMARKS
0			1" Black Dirt				
			2" concrete ~~on surface~~ (FC) over remaining subsurface.				0-4' Composite taken for • VOCs • DRO TPH • MT 10 Metals
2			Stiff Brown to Dark Grey Clay 2-4'				
3		90%					
4							

Logged By Angela Cook
Chk'd By

SOIL BOREHOLE LOG

SITE NAME & LOCATION: GM Delphi Goodwater Plant Abandoned Chemical Storage Tower Goodwater, AL

DRILLING METHOD: Geoprobe
SAMPLING METHOD: Direct Push
BORING NO.: GP-6
SHEET: 1 OF 1
DRILLING: START 1130 FINISH 1230
WATER LEVEL: TIME 1400 DATE 4/30/96
CASING DEPTH: 14'
DATUM: ELEVATION
DRILL RIG:
ANGLE: BEARING:
SAMPLE HAMMER TORQUE: Ft-Lbs.
SURFACE CONDITIONS: Vegetated Lot Grass & Chemical Storage Towers

DEPTH IN FEET (Elevation)	BLOWS / 6 In. ON SAMPLER (Recovery)	RECOVERY	SAMPLE NUMBER AND DESCRIPTION OF MATERIAL	GRAPHIC SYMBOL	DEPTH IN FEET From	DEPTH IN FEET To	DESCRIPTION OF OPERATION AND REMARKS
1			0-4" Organic Soils				Sampled 0-4' Composite for VOCs SVOCs
2		90%	4"-12" Sandy Brown Soil w/ pebbles/rubble				
3			1'-4' Brown stiff clay No Odor				
4							
11			4-14' Very Stiff Material (clay)				
12							
13			14' No Water				
14							

Drilling Contractor: [illegible]

SOIL BOREHOLE LOG

SITE NAME & LOCATION: GM Delphi Coldwater Road Plant West Side of WWTP GP-7

DRILLING METHOD: Geoprobe — BORING NO. GP-7

SAMPLING METHOD: Macro Core — SHEET 1 of 1

DRILLING — START — FINISH

WATER LEVEL					Time	Time
TIME	1730				1730	1730
DATE	8/30/96				Date	Date
CASING DEPTH	NA				8/30/96	8/30/96

DATUM — ELEVATION — DRILL RIG — SURFACE CONDITIONS: Vegetated lawn SW of WWTP

ANGLE — BEARING — SAMPLE HAMMER TORQUE — Ft.-Lbs.

DEPTH IN FEET (Elevation)	BLOWS / 6 In. ON SAMPLER	RECOVERY	SAMPLE NUMBER AND DESCRIPTION OF MATERIAL	GRAPHIC SYMBOL	DEPTH IN FEET From	DEPTH IN FEET To	DESCRIPTION OF OPERATION AND REMARKS
1							
2			Measured Depth to Bottom of WWTP basin ~ 10' BGS water level 11' BGS bottom of basin				
3							
4							
5							
6							
7							
8			Decided to take sample @ 10-12' interval				
9							
10		12"	Saturated Brown Sand. Lost drive pt on top of 2" casing				Sampled 10-12' Composite for DRO-TPH and Metals
11		100%					
12		12"	Dk Gray Stiff Clay Approx 5'-6' Standing water in the hole				

Logged By: [illegible]

ENVIRONMENTAL AUDIT REPORT
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: STEARNS DRILLING CO
DRILLING METHOD: 4.25" HSA

BORING NO.: PFW-1
SHEET NO.: 1 OF 7
PROJECT NO.: 4036.05
INSTALLATION: 3-13-97
SURFACE ELEV.: 99.6
BOREHOLE DIA.: 8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
1	SS	17	16	0.6		Asphalt, broken. Fill: well-graded sand with gravel, little silt, little clay, brown, wet.	
2	SS	15	18	0		SANDY LEAN CLAY (CL), little fine to coarse sand, few fine to coarse gravel, slightly plastic, brown 10YR 4/3 with some mottling to grayish brown and yellowish brown, moist, (hard Pp > 4.5) (Glacial Till).	
3	SS	16	24	0		As above (CL), very stiff from 2 to 2.5 ft.	
4	SS	15	24	0	5	As above (CL), fractured, very stiff (Pp=3.4) below 5.7 feet.	
5	SS	22	24	0		As above (CL), becoming mottled brown, dark grayish brown and dark gray 10YR 4/1 - 4/2.	
6	SS	16	24	0	10	As above (CL), wet at sand partings @ 8.5' and 9.1', brown, trace fractures.	

GENERAL NOTES

DATE STARTED: 12 MAR 97
DATE COMPLETED: 13 MAR 97
RIG: CME 750 ATV
CREW CHIEF: R. BENNETT
LOGGED: DPR CHECKED: LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽: 78.0 ft. bgl
AT COMPLETION ▼:
AFTER DRILLING
CAVE-IN: DATE/TIME _____ DEPTH _____
WATER: DATE/TIME _____ DEPTH _____

ENVIRONMENTAL AUDIT REPORT
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFW-1
LOCATION	FLINT, MICHIGAN	SHEET NO.	2 OF 7
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-13-97
		SURFACE ELEV.	99.6
		BOREHOLE DIA.	8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	INTERVAL TYPE	RECOVERY N	RECOVERY IN	PID	DEPTH		
7	SS	15	24			As above (CL), stiff to very stiff, (Pp= 1.6 to 2.7).	
						As above (CL).	
						WELL-GRADED SAND WITH SILT (SW), fine to medium, trace gravel, few clay, brown 10YR 4/3, moist, pieces of clay till.	
					15	SANDY LEAN CLAY (CL), some fine to coarse sand, few fine to coarse gravel, slightly plastic, brown 10YR 4/3, moist, stiff to very stiff (Glacial Till).	
8	SS	15	24	0		As above (CL), hard (Pp > 4).	
					20		
						As above (CL).	
9	SS	19	24	0			
						LEAN CLAY (CL), gradational areas of	

ENVIRONMENTAL AUDIT REPORT:
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LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFW-1
LOCATION	FLINT, MICHIGAN	SHEET NO.	3 OF 7
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-13-97
		SURFACE ELEV.	99.6
		BOREHOLE DIA.	8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
						clayey silt and sandy silt, slightly plastic, mottled brown, dark yellowish brown, and brownish gray, hard, fractured, friable, (Glacial Till).	
					25	SILTY SAND (SM), fine, brown, wet.	
10	SS	40	24	0			
						LEAN CLAY (CL), slightly plastic, brown with dark yellowish brown and black precipitate along fractures, mostly dark grayish brown below 29.6', moist to wet along silt partings, very hard, faint lamination (Glaciolacustrine).	
					30		
					35		

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFW-1
LOCATION	FLINT, MICHIGAN	SHEET NO.	4 OF 7
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-13-97
		SURFACE ELEV.	99.6
		BOREHOLE DIA.	8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
11	SS	20	0			As above (CL), gray 10YR 5/1 (based on cuttings).	
					40		
12	SS	17	24	0.6		As above (CL), wet along silt partings, very stiff (Pp = 3.2 to 3.7).	
					45	SILT (ML), grading from above clay, nonplastic, gray 10YR 5/1, moist, very stiff.	

ENVIRONMENTAL AUDIT REPORT
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFW-1
LOCATION	FLINT, MICHIGAN	SHEET NO.	5 OF 7
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-13-97
		SURFACE ELEV.	99.6
		BOREHOLE DIA.	8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
13	SS	14	24	0.3	50	LEAN CLAY (CL), trace fine gravel, trace fine to coarse sand, medium plastic, gray 10YR 5/1, moist, very stiff (Pp = 2.3 to 2.7), faint lamination (Glaciolacustrine).	
					55		
					60		

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFW-1
LOCATION	FLINT, MICHIGAN	SHEET NO.	6 OF 7
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-13-97
		SURFACE ELEV.	99.6
		BOREHOLE DIA.	8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
14	SS	16	24	0.2		As above (CL), abundant silt partings.	
					65		
					70	<— Drillers note change in resistance @ 71 feet.	
15	SS	88	18			POORLY-GRADED SAND (SP), fine, trace silt, light gray 10YR 7/1, moist to dry, faint stratification.	
					75		

ENVIRONMENTAL AUDIT REPOF
PRIVILEGED DOCUMENT

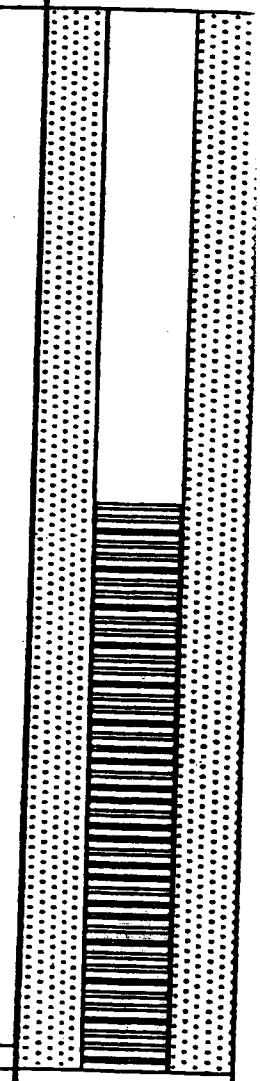
LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFW-1
LOCATION	FLINT, MICHIGAN	SHEET NO.	7 OF 7
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-13-97
		SURFACE ELEV.	99.6
		BOREHOLE DIA.	8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
16	SS	23	18		80	As above (SP), some medium sand, gray 10YR 6/1, wet.	
17	SS	40	24		85	As above (SP), mostly medium grained.	
						LEAN CLAY (CL).	
						End of boring at 85 feet.	

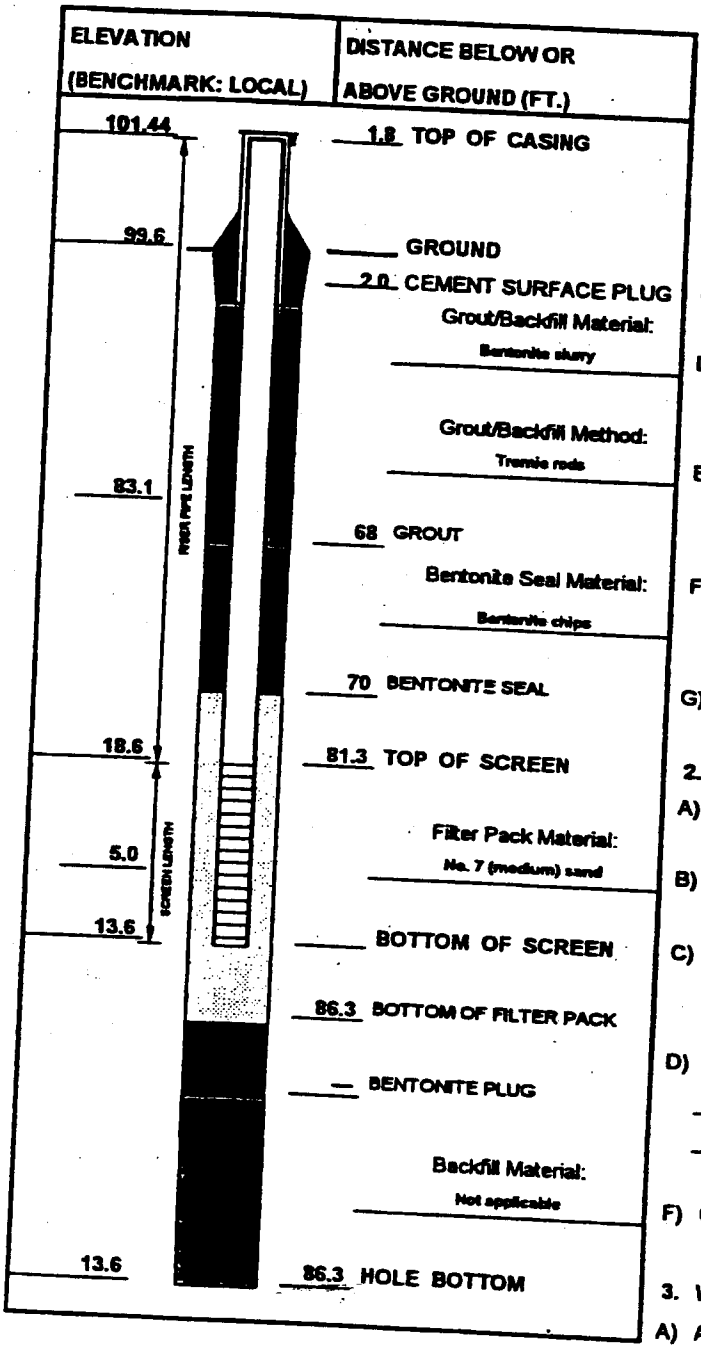


ENVIRONMENTAL AUDIT REPORT
PRIVILEGED DOCUMENT

RMT INC.

WELL CONSTRUCTION DIAGRAM

PROJECT:	Peregrine - Flint					WELL NO.:	PFW-1
PROJ. NO:	4036.05	DATE INSTALLED:	3-13-97	OBSV. BY:	DPR	CHECKED BY:	DPR



1. CASING AND SCREEN DETAILS:

A) Type Of Pipe: 2" PVC — Pipe Schedule: 40

B) Pipe Joints: Flush with O-ring

C) Solvent Used? No

D) Screen Type: 2" with machined slots, flush joint — Screen Slot Size: 0.01"

E) Borehole Diameter: 8 In. From 0 To 86.3 Ft.
___ In. From ___ To ___ Ft.

F) Surf. Casing Diameter: ___ In. From ___ To ___ Ft.
2nd Surface Casing: ___ In. From ___ To ___ Ft.

G) Installed Protective Cover W/ Lock? Yes

2. WELL DEVELOPMENT:

A) Method: Surge/pump with Bremer check valve

B) Time Spent Developing: 2.4 Hours

C) Water Removed: 300 Gallons
Added: 10 Gallons

D) Water Clarity Before/After Development:
Before: Opaque, gray
After: Slightly turbid (approximately 50 NTU)

F) Odor (Descr. if present) None

3. WATER LEVEL SUMMARY:

A) After Developing: ___ Ft. Below Top Of Casing

B) Other Date/Time: 3-31-97/1358 80.52 Ft.
Other Date/Time: ___ Ft.

Notes: Approximately 10 gallons of clean water were added to eliminate a temporary bridge during filter packing.

ENVIRONMENTAL AUDIT REPORT
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

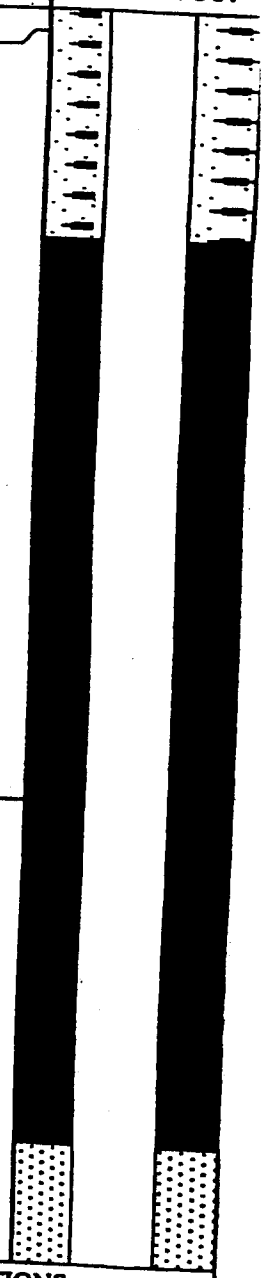
RMT

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFW-2
LOCATION	FLINT, MICHIGAN	SHEET NO.	1 OF 2
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-14-97
		SURFACE ELEV.	98.5
		BOREHOLE DIA.	8 IN.

SAMPLING NOTES

INTERVAL NO.	INTERVAL TYPE	RECOVERY N	RECOVERY IN	PID	DEPTH	VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
1	SS	26	18	0		Topsoil, wet.
						SANDY LEAN CLAY WITH GRAVEL (CL), some fine to coarse sand, little fine to coarse gravel, slightly plastic, brown 10YR 4/3 with some dark yellowish brown mottling, moist, hard (Pp > 4.5).
2	SS	15	16	0		
						As above (CL), increasing moisture below 3 feet, stiff (Pp = 1.2 to 1.5).
3	SS	12	16	0		
					5	As above (CL), some brownish gray mottling, some very stiff areas (Pp = 2.5).
4	SS	5	18	0		
						As above (CL), with gray areas.
					▽	WELL-GRADED SAND WITH CLAY (SW-SC), fine to medium sand, little coarse sand, few clay, trace gravel, brown 10YR 4/3, wet.
5	SS	1	18			
						As above (SW-SC), 2" to 4" zone stiff sandy clay.
6	SS	2	6		10	

GENERAL WELL CONSTRUCT.



GENERAL NOTES

DATE STARTED	14 MAR 97
DATE COMPLETED	14 MAR 97
RIG	CME 750 ATV
CREW CHIEF	R. BENNETT
LOGGED	DPR CHECKED DL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽	7.3 ft. bgl
AT COMPLETION ▽	
AFTER DRILLING	
CAVE-IN: DATE/TIME	DEPTH
WATER: DATE/TIME	DEPTH

ENVIRONMENTAL AUDIT REPORT
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFW-2
LOCATION	FLINT, MICHIGAN	SHEET NO.	2 OF 2
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-14-97
		SURFACE ELEV.	98.5
		BOREHOLE DIA.	8 IN.

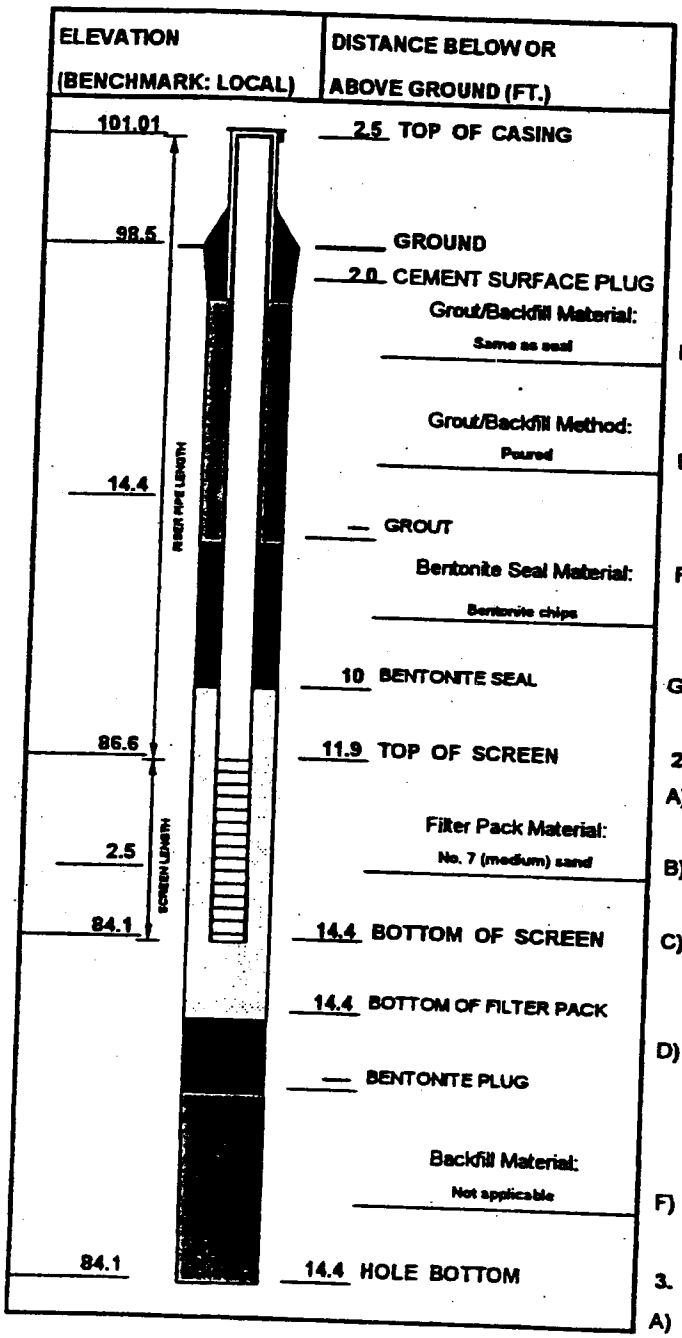
SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	INTERVAL TYPE	RECOVERY N	RECOVERY IN	PID	DEPTH		
7	SS	4	20			As above (SP-SC), trace clay, dark yellowish brown 10YR 4/4.	
						WELL-GRADED SAND (SW), fine to coarse, trace silt, trace clay, dark yellowish brown becoming dark grayish brown @ 13.4' with black steaks, diesel hydrocarbon odor.	
						SANDY LEAN CLAY (CL), some sand, trace fine to coarse gravel, slightly plastic, dark grayish brown, moist, hard (Pp > 4).	
					15	End of boring at 14 feet.	
					20		

ENVIRONMENTAL AUDIT REPORT
PRIVILEGED DOCUMENT

RMT INC.

WELL CONSTRUCTION DIAGRAM

PROJECT:	Peregrine - Flint					WELL NO.:	PFW-2
PROJ. NO:	4036.05	DATE INSTALLED:	3-14-97	OBSV. BY:	DPR	CHECKED BY:	DPR



1. CASING AND SCREEN DETAILS:

A) Type Of Pipe: 2" PVC — Pipe Schedule: 40

B) Pipe Joints: Flush with O-ring

C) Solvent Used? No

D) Screen Type: 2" with machined slots, flush joint — Screen Slot Size: 0.01"

E) Borehole Diameter: 8 In. From 0 To 14 Ft.
____ In. From ____ To ____ Ft.

F) Surf. Casing Diameter: ____ In. From ____ To ____ Ft.
2nd Surface Casing: ____ In. From ____ To ____ Ft.

G) Installed Protective Cover W/ Lock? Yes

2. WELL DEVELOPMENT:

A) Method: Surge/pump with Bremer check valve

B) Time Spent Developing: 1 Hours

C) Water Removed: 27 Gallons
Added: 0 Gallons

D) Water Clarity Before/After Development:
Before: Opaque, brown
After: Slightly turbid (approximately 100 NTU)

F) Odor (Descr. if present) diesel range hydrocarbons

3. WATER LEVEL SUMMARY:

A) After Developing: ____ Ft. Below Top Of Casing

B) Other Date/Time: 3-31-97/0946 6.74 Ft.

Other Date/Time: ____ Ft.

Notes:

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: STEARNS DRILLING CO
DRILLING METHOD: 4.25" HSA

BORING NO.: PFW-3
SHEET NO.: 1 OF 2
PROJECT NO.: 4036.05
INSTALLATION: 3-17-97
SURFACE ELEV.: 100.3
BOREHOLE DIA.: 8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
1	SS	49	18	0.6		Fill: sand, gravel crushed limestone, upper few inches frozen.	
						SANDY LEAN CLAY (CL), some fine to coarse sand, trace fine gravel, slightly plastic, brown 10YR 4/3 with some gray and dark yellowish brown mottling, moist, hard.	
2	SS	17	16	0.2		WELL-GRADED SAND WITH CLAY (SW-SC), fine to coarse, dark brown 10YR 3/3, moist.	
						SANDY LEAN CLAY WITH GRAVEL (CL), little coarse gravel, dark grayish brown 10YR 4/2, moist, hard.	
3	SS	6	18	0.2		SILTY SAND (SM), fine, brown 10YR 4/3, moist.	
					5	SANDY LEAN CLAY WITH GRAVEL (CL), some fine to coarse sand, little fine to coarse gravel, moderately plastic, dark gray 10YR 4/1, moist, very stiff (Pp = 2.6).	
4	SS	12	20			As above (CL).	
5	SS	14	24			As above (CL), stiff (Pp = 1.3 to 2.0).	
6	SS	12	24		10	As above (CL).	

GENERAL NOTES

DATE STARTED: 17 MAR 97
DATE COMPLETED: 17 MAR 97
RIG: CME LC 60
CREW CHIEF: M. HEFFERAN
LOGGED: DPR CHECKED: LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽: none observed
AT COMPLETION ▼:
AFTER DRILLING
CAVE-IN: DATE/TIME _____ DEPTH _____
WATER: DATE/TIME _____ DEPTH _____

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: STEARNS DRILLING CO
DRILLING METHOD: 4.25" HSA

BORING NO.: PFW-3
SHEET NO.: 2 OF 2
PROJECT NO.: 4036.05
INSTALLATION: 3-17-97
SURFACE ELEV.: 100.3
BOREHOLE DIA.: 8 IN.

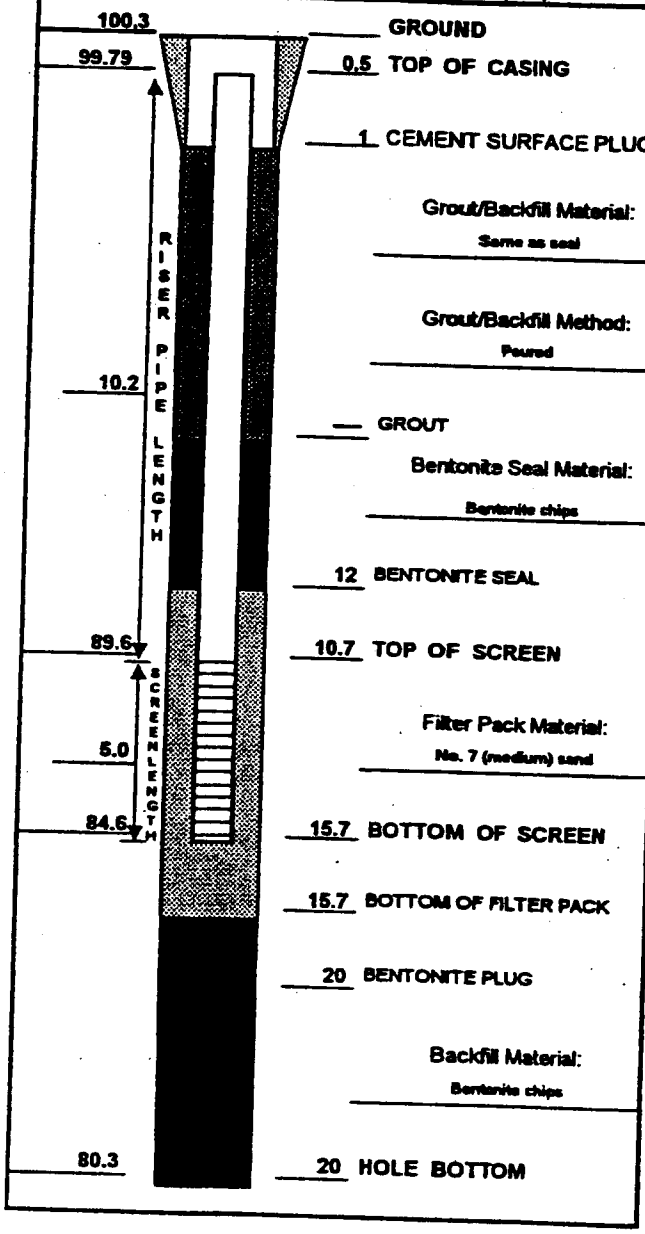
SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
7	SS	14	24				
						POORLY-GRADED SAND WITH SILT (SP-SM), fine, gray, moist.	
8	SS	15	24			SANDY LEAN CLAY (CL), some sand, little fine to coarse gravel, slightly plastic, dark gray 10YR 4/1, moist, stiff.	
					15		
						As above (CL), medium stiff (Pp = 1.0).	
9	SS	11	24				
						As above (CL), (Pp = 0.5).	
10	SS	10	24				
						As above (CL), (Pp = 1.0).	
					20		
						End of boring at 20 feet.	

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

RMT INC.

WELL CONSTRUCTION DIAGRAM

PROJECT:	Peregrine - Flint					WELL NO.:	PFW-3
PROJ. NO:	4036.05	DATE INSTALLED:	3-17-97	OBSV. BY:	DPR	CHECKED BY:	DPR



1. CASING AND SCREEN DETAILS:

A) Type Of Pipe: 2" PVC — Pipe Schedule: 40

B) Pipe Joints: Flush with O-ring

C) Solvent Used? No

D) Screen Type: 2" with machined slots, flush joint — Screen Slot Size: 0.01"

E) Borehole Diameter: 8 In. From 0 To 18 Ft.
3 In. From ____ To 20 Ft.

F) Surf. Casing Diameter: ____ In. From ____ To ____ Ft.
2nd Surface Casing: ____ In. From ____ To ____ Ft.

G) Installed Protective Cover W/ Lock? Yes

2. WELL DEVELOPMENT:

A) Method: None - dry well

B) Time Spent Developing: ____ Hours

C) Water Removed: ____ Gallons
Added: ____ Gallons

D) Water Clarity Before/After Development:
Before:
After:

F) Odor (Descr. if present) None

3. WATER LEVEL SUMMARY:

A) After Developing: ____ Ft. Below Top Of Casing

B) Other Date/Time: ____ Ft.
Other Date/Time: ____ Ft.

Notes:

ENVIRONMENTAL AUDIT REPORT
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: STEARNS DRILLING CO
DRILLING METHOD: 4.25" HSA

BORING NO.: PFW-4
SHEET NO.: 1 OF 3
PROJECT NO.: 4036.05
INSTALLATION: 3-17-97
SURFACE ELEV.: 99.8
BOREHOLE DIA.: 8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
						Concrete.	
1	SS	23	18	0.2		Fill: sand and gravel, few fines, pale brown, moist.	
						SANDY LEAN CLAY WITH GRAVEL (CL), some fine to coarse sand, little fine gravel, slightly plastic, brown 10YR 4/3 with pale brown along occasional silt partings and fractures, some dark yellowish brown mottling, no odor, moist, hard (Pp > 4).	
2	SS	31	24	0			
						Silt and silty sand stringers at 4.4 feet.	
3	SS	29	24		5	SANDY LEAN CLAY (CL) as above to 4.4 feet, wet sand parting at 4.8 feet.	
						As above (CL), some dark yellowish brown along fractures, occasional wet	

GENERAL NOTES

DATE STARTED: 17 MAR 97
DATE COMPLETED: 17 MAR 97
RIG: CME LC 60
CREW CHIEF: M. HEFFERAN
LOGGED: DPR CHECKED: LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽: none observed
AT COMPLETION ▼:
AFTER DRILLING
CAVE-IN: DATE/TIME _____ DEPTH _____
WATER: DATE/TIME _____ DEPTH _____

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

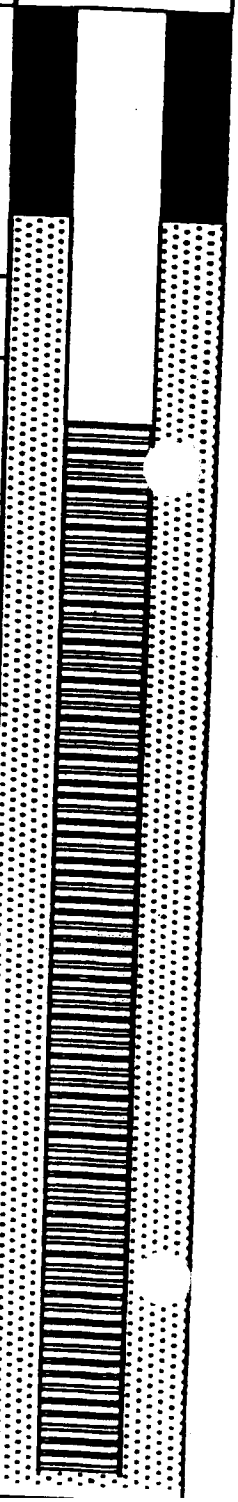
LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFW-4
LOCATION	FLINT, MICHIGAN	SHEET NO.	2 OF 3
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-17-97
		SURFACE ELEV.	99.8
		BOREHOLE DIA.	8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
						sand partings, very stiff.	
4	SS	21	24			As above (CL).	
						SILTY SAND (SM), brown, wet, laminated.	
						SANDY LEAN CLAY as above to 7.3 feet, very stiff to hard.	
5	SS	30	24			SILT (ML), little clay, few fine sand, brown with dark yellowish brown mottling, wet, nonlaminated.	
					10	SANDY LEAN CLAY WITH GRAVEL (CL), slightly plastic, dark grayish brown 10YR 4/2 with dark yellowish brown along fractures, moist, very stiff, sand partings.	
						SILT (ML), some clay, few sand, mottled dark grayish brown and dark yellowish brown, moist.	
6	SS	28	24			SANDY LEAN CLAY WITH GRAVEL (CL), slightly plastic, dark grayish brown 10YR 4/2 with dark yellowish brown along fractures, moist, very stiff, sand partings.	
						SILTY SAND (SM), fine to medium, trace clay, brown, wet.	
						SANDY LEAN CLAY WITH GRAVEL (CL), dark grayish brown, slightly plastic, 10YR 4/2, moist, very stiff.	
						SILT (ML), mottled grays and dark yellowish brown, moist to wet, nonlaminated, fractured.	



ENVIRONMENTAL AUDIT REPORT
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: STEARNS DRILLING CO
DRILLING METHOD: 4.25" HSA

BORING NO.: PFW-4
SHEET NO.: 3 OF 3
PROJECT NO.: 4036.05
INSTALLATION: 3-17-97
SURFACE ELEV.: 99.8
BOREHOLE DIA.: 8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	INTERVAL TYPE	RECOVERY N	RECOVERY IN	PID	DEPTH		
7	SS	44	24			SANDY LEAN CLAY (CL), brown to dark yellowish brown, hard.	
						SILT (ML), little clay, few fine sand, brown, nonlaminated, moist to wet.	
						SILTY SAND (SM), trace clay, brown to dark yellowish brown to dark grayish brown, stratified.	
8	SS	16	24		15	SANDY LEAN CLAY (CL), dark grayish brown, moist.	
						SILTY SAND (SM), trace clay, dark grayish brown, wet, stratified.	
						SANDY LEAN CLAY (CL), some sand, few fine gravel, medium plastic, dark gray 10YR 4/1, moist, very stiff (Pp = 3.0).	
						End of boring at 17 feet.	
					20		

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

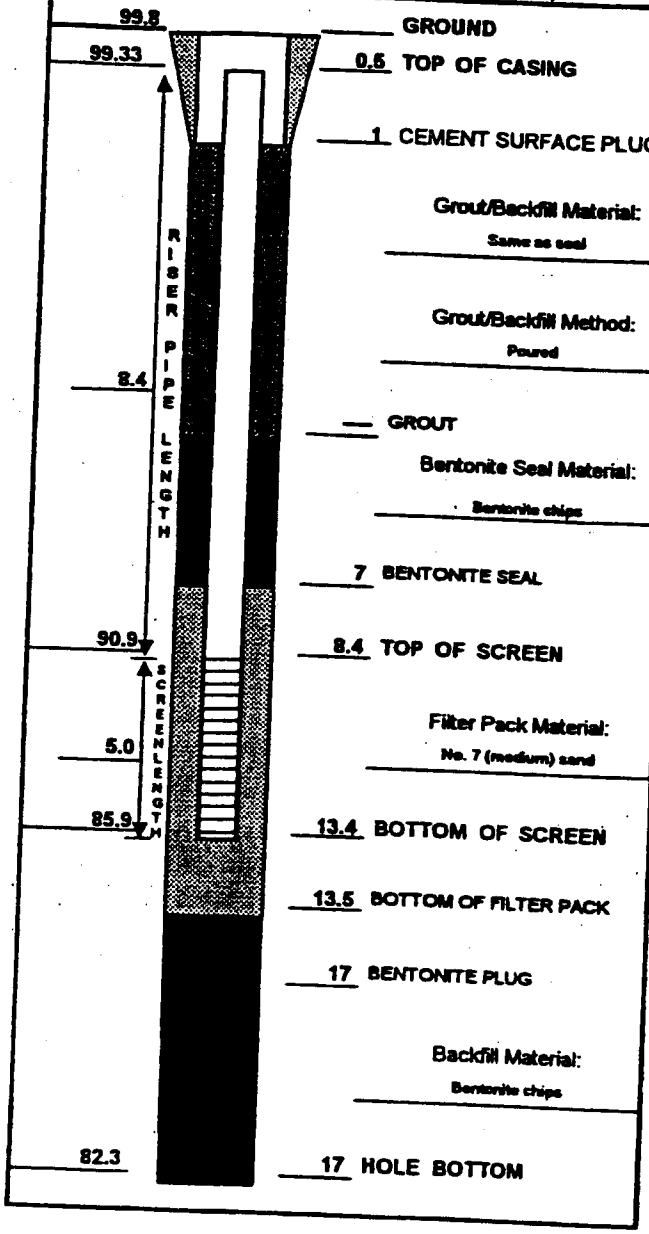
RMT INC.

WELL CONSTRUCTION DIAGRAM

PROJECT:	Peregrine - Flint					WELL NO.:	PFW-4
PROJ. NO:	4036.05	DATE INSTALLED:	3-17-97	OBSV. BY:	DPR	CHECKED BY:	DPR

ELEVATION: (BENCHMARK: LOCAL)

DISTANCE BELOW OR ABOVE GROUND (FT.):



- ___ GROUND
- 0.5 TOP OF CASING
- 1 CEMENT SURFACE PLUG
- Grout/Backfill Material: Same as seal
- Grout/Backfill Method: Poured
- ___ GROUT
- Bentonite Seal Material: Bentonite chips
- 7 BENTONITE SEAL
- 8.4 TOP OF SCREEN
- Filter Pack Material: No. 7 (medium) sand
- 13.4 BOTTOM OF SCREEN
- 13.5 BOTTOM OF FILTER PACK
- 17 BENTONITE PLUG
- Backfill Material: Bentonite chips
- 17 HOLE BOTTOM

1. CASING AND SCREEN DETAILS:

A) Type Of Pipe: 2" PVC — Pipe Schedule: 40

B) Pipe Joints: Flush with O-ring

C) Solvent Used? No

D) Screen Type: 2" with machined slots, flush joint — Screen Slot Size: 0.01"

E) Borehole Diameter: 8 In. From 0 To 15 Ft.
3 In. From 15 To 17 Ft.

F) Surf. Casing Diameter: ___ In. From ___ To ___ Ft.
2nd Surface Casing: ___ In. From ___ To ___ Ft.

G) Installed Protective Cover W/ Lock? Yes

2. WELL DEVELOPMENT:

A) Method: Gently bail

B) Time Spent Developing: 1 Hours

C) Water Removed: 7.1 Gallons
Added: 0 Gallons

D) Water Clarity Before/After Development:
Before: Clear
After: Slightly turbid (approximately 50 NTU)

F) Odor (Descr. if present) None

3. WATER LEVEL SUMMARY:

A) After Developing: ___ Ft. Below Top Of Casing

B) Other Date/Time: 3-31-97/1240 5.52 Ft.
Other Date/Time: ___ Ft.

Notes: ___

ENVIRONMENTAL AUDIT REPORT
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: STEARNS DRILLING CO
DRILLING METHOD: 4.25" HSA

BORING NO.: PFB-5
SHEET NO.: 1 OF 1
PROJECT NO.: 4036.05
INSTALLATION: 3-18-97
SURFACE ELEV.: 98.9
BOREHOLE DIA.: 8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
INTERVAL NO.	INTERVAL TYPE	RECOVERY N	RECOVERY IN	PID	DEPTH	
1	SS	12	12			Asphalt and concrete.
						Fill: poorly graded sand, medium, some coarse gravel.
						Obstruction @ 3 feet; move and redrill PFW-5.
						End of boring at 3 feet. Boring backfilled with cuttings.
					5	
					10	

GENERAL NOTES

DATE STARTED: 18 MAR 97
DATE COMPLETED: 18 MAR 97
RIG: CME LC 60
CREW CHIEF: M. HEFFERAN
LOGGED: DPR CHECKED: LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽: none observed
AT COMPLETION ▼:
AFTER DRILLING
CAVE-IN: DATE/TIME: DEPTH:
WATER: DATE/TIME: DEPTH:

ENVIRONMENTAL AUDIT REPOR
PRIVILEGED DOCUME

LOG OF TEST BORING

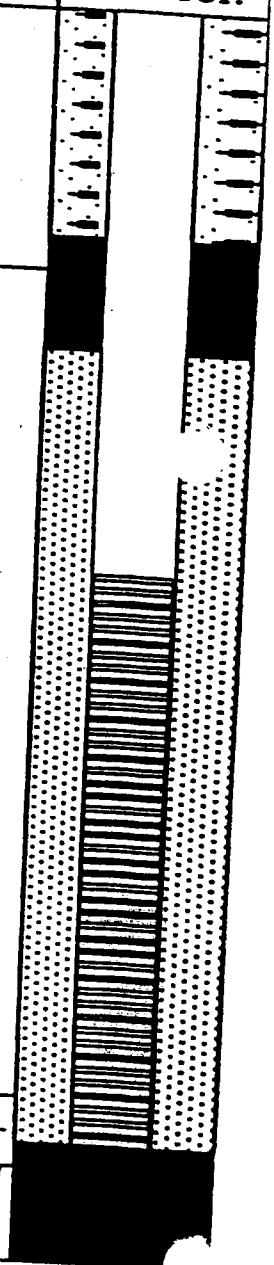
F-203 (R 01-87)

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFW-5
LOCATION	FLINT, MICHIGAN	SHEET NO.	1 OF 2
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-18-97
		SURFACE ELEV.	98.9
		BOREHOLE DIA.	8 IN.

SAMPLING NOTES

INTERVAL NO.	INTERVAL TYPE	RECOVERY N	RECOVERY IN	PID	DEPTH	VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
1	SS	8	20	0.1		Fill: mostly medium sand, little fine to coarse rounded gravel, brown to 15 inches, then dark yellowish brown, no odor, moist.
2	SS	9	22	0.1		As above.
3	SS	2	12	0		Fill: fine to medium sand, dark yellowish brown, becoming brown and wet @ 3.7 ft.
					5	
4	SS	1	0			As above, dark grayish brown.
5	SS	2	20			As above (based on blow counts and contents of 8 to 10 ft. sample).
6	SS	10	16		10	As above.
						SANDY LEAN CLAY (CL), medium plastic, gray.
						SILT (ML), dark gray, laminated with clayey silt and sandy silt, wet.

GENERAL WELL CONSTRUCT.



GENERAL NOTES

DATE STARTED	18 MAR 97
DATE COMPLETED	18 MAR 97
RIG	CME LC 60
CREW CHIEF	M. HEFFERAN
LOGGED	DPR CHECKED LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽	3.7 ft. bgl
AT COMPLETION ▼	
AFTER DRILLING	
CAVE-IN: DATE/TIME	DEPTH
WATER: DATE/TIME	DEPTH

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFW-5
LOCATION	FLINT, MICHIGAN	SHEET NO.	2 OF 2
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-18-97
		SURFACE ELEV.	98.9
		BOREHOLE DIA.	8 IN.

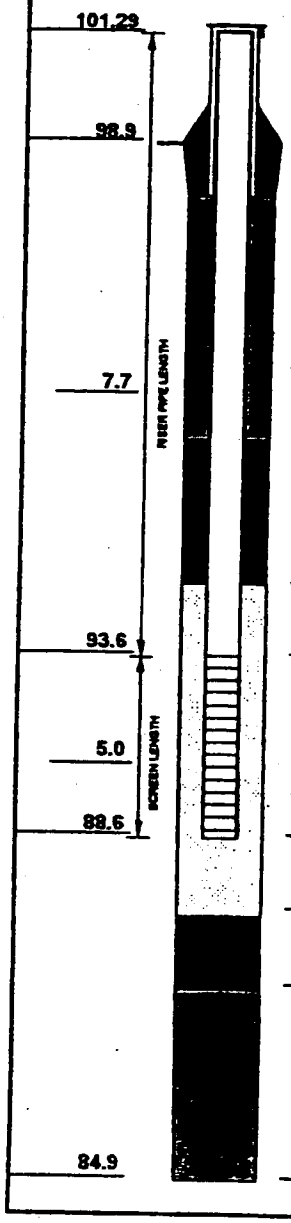
SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
7	SS	10	18			SANDY LEAN CLAY (CL), some fine to coarse sand, few fine gravel, slightly plastic, dark gray 10YR 4/1, moist, very stiff (Pp = 3.0 to 3.7).	
						As above (CL).	
						End of boring at 14 feet.	
					15		
					20		

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

RMT INC.

WELL CONSTRUCTION DIAGRAM

PROJECT:	Peregrine - Flint					WELL NO.:	PFW-5
PROJ. NO:	4036.05	DATE INSTALLED:	3-18-97	OBSV. BY:	DPR	CHECKED BY:	DPR



1. CASING AND SCREEN DETAILS:

A) Type Of Pipe: 2" PVC — Pipe Schedule: 40

B) Pipe Joints: Flush with O-ring

C) Solvent Used? No

D) Screen Type: 2" with machined slots, flush joint — Screen Slot Size: 0.01"

E) Borehole Diameter: 8 In. From 0 To 10 Ft.
3 In. From 10 To 14 Ft.

F) Surf. Casing Diameter: ____ In. From ____ To ____ Ft.
2nd Surface Casing: ____ In. From ____ To ____ Ft.

G) Installed Protective Cover W/ Lock? Yes

2. WELL DEVELOPMENT:

A) Method: Gently bail

B) Time Spent Developing: 1.5 Hours

C) Water Removed: 25 Gallons
Added: 0 Gallons

D) Water Clarity Before/After Development:
Before: Opaque brown
After: Slightly turbid (approximately 100 NTU)

F) Odor (Descr. if present) None

3. WATER LEVEL SUMMARY:

A) After Developing: ____ Ft. Below Top Of Casing

B) Other Date/Time: 3-31-97/1305 5.77 Ft.

Other Date/Time: ____ Ft.

Notes: ____

ENVIRONMENTAL AUDIT REPOR
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: STEARNS DRILLING CO
DRILLING METHOD: 4.25" HSA

BORING NO.: PFW-6
SHEET NO.: 1 OF 2
PROJECT NO.: 4036.05
INSTALLATION: 3-18-97
SURFACE ELEV.: 98.6
BOREHOLE DIA.: 8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
						Asphalt.	
						Concrete.	
1	SS	16	18	9.4		Fill: medium to coarse sand with coarse rounded gravel, few silt, few clay, dark grayish brown, wet.	
						SANDY LEAN CLAY (CL), some fine to coarse sand, few fine to coarse gravel, slightly plastic, brown 10YR 4/3 with some gray mottling, no odor, moist, hard (Pp > 4.0), fractured.	
2	SS	25	20	10.2		As above (CL).	
3	SS	22	24		5	As above (CL).	
4	SS	21	24			As above (CL), dark gray 10YR 4/1 below 7.3 feet, very stiff (Pp = 2.7 to 3.5).	
5	SS	23	6			As above (CL).	
					10		

GENERAL NOTES

DATE STARTED: 18 MAR 97
DATE COMPLETED: 18 MAR 97
RIG: CME LC 60
CREW CHIEF: M. HEFFERAN
LOGGED: DPR CHECKED: LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING: none observed
AT COMPLETION:
AFTER DRILLING
CAVE-IN: DATE/TIME ____ DEPTH ____
WATER: DATE/TIME ____ DEPTH ____

ENVIRONMENTAL AUDIT REPORT
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: STEARNS DRILLING CO
DRILLING METHOD: 4.25" HSA

BORING NO.: PFW-6
SHEET NO.: 2 OF 2
PROJECT NO.: 4036.05
INSTALLATION: 3-18-97
SURFACE ELEV.: 98.6
BOREHOLE DIA.: 8 IN.

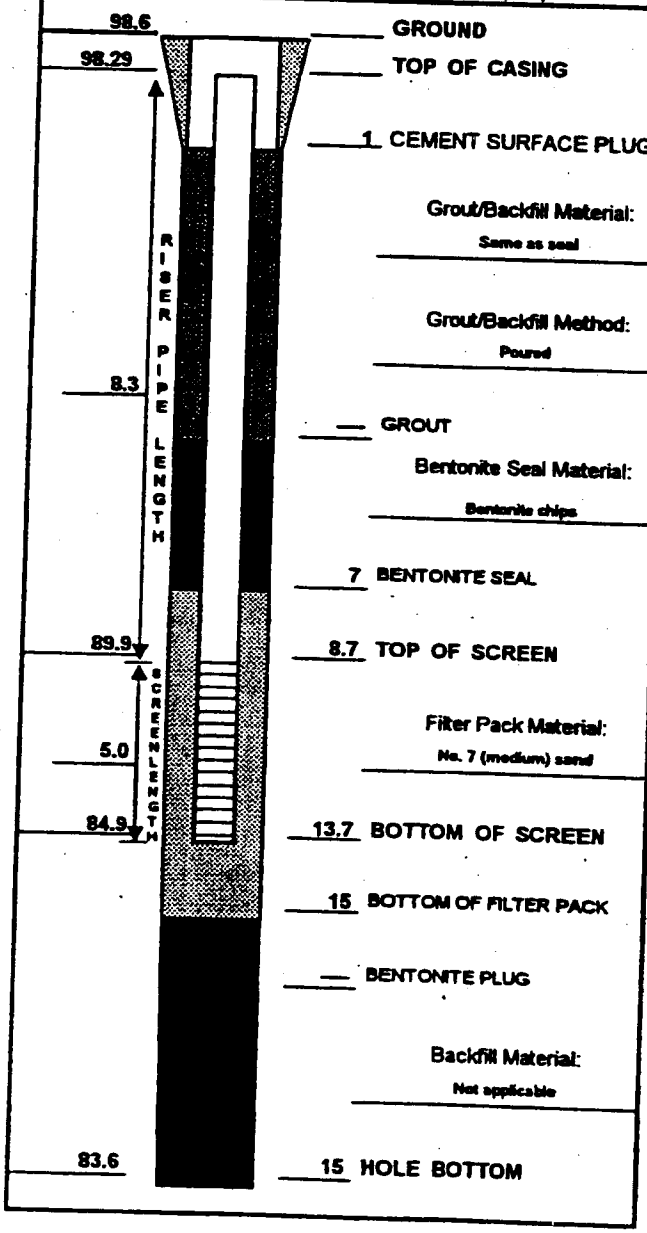
SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	INTERVAL TYPE	RECOVERY N	RECOVERY IN	PID	DEPTH		
6	SS	16	24			As above (CL), stiff (Pp = 1.4 to 1.7).	
7	SS	17	24			As above (CL), medium stiff (Pp = 1.0).	
					15	End of boring at 15 feet.	
					20		

ENVIRONMENTAL AUDIT REPOR
PRIVILEGED DOCUMENT

RMT INC.

WELL CONSTRUCTION DIAGRAM

PROJECT:	Peregrine - Flint					WELL NO.:	PFW-6
PROJ. NO:	4036.05	DATE INSTALLED:	3-18-97	OBSV. BY:	DPR	CHECKED BY:	DPR



1. CASING AND SCREEN DETAILS:

A) Type Of Pipe: 2" PVC — Pipe Schedule: 40

B) Pipe Joints: Flush with O-ring

C) Solvent Used? No

D) Screen Type: 2" with machined slots, flush joint — Screen Slot Size: 0.01"

E) Borehole Diameter: 8 In. From 0 To 13 Ft.
3 In. From 13 To 15 Ft.

F) Surf. Casing Diameter: ____ In. From ____ To ____ Ft.
2nd Surface Casing: ____ In. From ____ To ____ Ft.

G) Installed Protective Cover W/ Lock? Yes

2. WELL DEVELOPMENT:

A) Method: Gently bail

B) Time Spent Developing: 0.2 Hours

C) Water Removed: 3.7 Gallons
Added: 0 Gallons

D) Water Clarity Before/After Development:
Before: Slightly turbid, light brown
After: Slightly turbid, light gray

F) Odor (Descr. if present) None

3. WATER LEVEL SUMMARY:

A) After Developing: (dry) Ft. Below Top Of Casing

B) Other Date/Time: 3-18-97/1500 dry Ft.
Other Date/Time: 3-31-97/1320 1.05 Ft.

Notes:

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: STEARNS DRILLING CO
DRILLING METHOD: 4.25" HSA

BORING NO.: PFB-7
SHEET NO.: 1 OF 2
PROJECT NO.: 4036.05
INSTALLATION: 3-18-97
SURFACE ELEV.:
BOREHOLE DIA.: 8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH	
						Concrete.
1	SS	24	16	0		Fill: fine to medium sand, dark yellowish brown, no odor, moist.
2	SS	29	20	0		SANDY LEAN CLAY (CL), some fine to coarse sand, few fine to coarse gravel, slightly plastic, dark brown to brown, dry to moist, hard (Pp > 4.5), fractured, some areas of fill sand as above.
3	SS	29	24	0	5	SANDY LEAN CLAY (CL), some fine to coarse sand, few fine to coarse gravel, slightly plastic, brown with some gray and dark yellowish brown mottling, no odor, dry to moist, hard (Pp > 4.5), fractured.
4	SS	20	24	0		As above (CL), dark gray below 7 feet, dark yellowish brown along sporatic fractures, very stiff (Pp = 2.5 to 3.5).
5	SS	18	18	2.2		As above (CL), dark yellowish brown to dark brown along fracture from 9 to 9.5 feet, hard.
6	SS	14	24	0	10	As above (CL), dark gray with occasional dark yellow brown zones, stiff (Pp = 1.5).

GENERAL NOTES

DATE STARTED: 18 MAR 97
DATE COMPLETED: 18 MAR 97
RIG: CME LC 60
CREW CHIEF: M. HEFFERAN
LOGGED: DPR CHECKED: LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽: 22.5 ft. bgl
AT COMPLETION ▼:
AFTER DRILLING
CAVE-IN: DATE/TIME _____ DEPTH _____
WATER: DATE/TIME _____ DEPTH _____

ENVIRONMENTAL AUDIT REPO
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFB-7
LOCATION	FLINT, MICHIGAN	SHEET NO.	2 OF 2
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-18-97
		SURFACE ELEV.	
		BOREHOLE DIA.	8 IN.

SAMPLING NOTES						
INTERVAL NO.	INTERVAL TYPE	RECOVERY N	RECOVERY IN	PID	DEPTH	VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
7	SS	14	24	0		As above (CL).
8	SS	16	24	0	15	As above (CL), all dark gray below 14.3 feet, stiff to very stiff (Pp = 1.5 to 2.2).
9	SS	14	24	0		As above (CL), stiff (Pp = 1.5 to 1.7).
10	SS	16	24	0		As above (CL).
11	SS	13	24	0	20	As above (CL).
12	SS	15	24	0		As above (CL), 1/4-inch wet fine to coarse sand stringer @ 22.5'.
						LEAN CLAY (CL), moderately plastic, pale red 10YR 6/2, moist.
						SANDY LEAN CLAY (CL), some fine to coarse sand, few fine to coarse gravel, slightly plastic, dark gray, moist, stiff.
						End of boring at 24 feet.
					25	

ENVIRONMENTAL AUDIT REPC
PRIVILEGED DOCUMENT

LOG OF TEST BORING

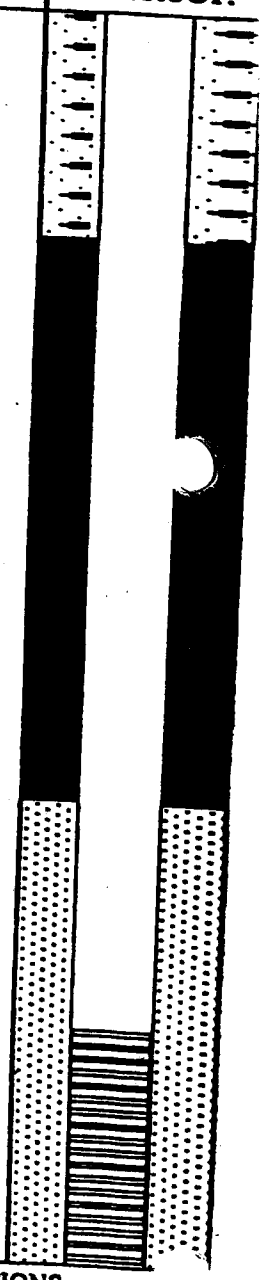
F-203 (R 01-87)

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFW-8
LOCATION	FLINT, MICHIGAN	SHEET NO.	1 OF 2
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-19-97
		SURFACE ELEV.	101.1
		BOREHOLE DIA.	8 IN.

SAMPLING NOTES

INTERVAL NO.	INTERVAL TYPE	RECOVERY N	RECOVERY IN	PID	DEPTH	VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
1	SS	18	24	0		SANDY LEAN CLAY (CL), some sand, few fine to coarse gravel, slightly plastic, dark grayish brown 10YR 4/2 with some dark yellowish brown and dark gray mottling, black precipitate along fractures, moist, hard (Pp > 4.5) (Glacial Till).
2	SS	19	18	0		As above (CL), dark gray below 2.5 feet, (Pp = 4.2 to 4.5).
3	SS	17	18	0	5	As above (CL).
4	SS	18	21	0.4		As above (CL), very stiff (Pp = 3.2 to 3.5).
5	SS	17	24	0		As above (CL), stiff (Pp = 1.4 to 1.7).
6	SS	21	24	0	10	As above (CL).

GENERAL WELL CONSTRUCT.



GENERAL NOTES

DATE STARTED	19 MAR 97
DATE COMPLETED	19 MAR 97
RIG	CME LC 60
CREW CHIEF	M. HEFFERAN
LOGGED	DPR CHECKED LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽	none observed
AT COMPLETION ▼	
AFTER DRILLING	
CAVE-IN: DATE/TIME	DEPTH
WATER: DATE/TIME	DEPTH

ENVIRONMENTAL AUDIT REPOR
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFW-8
LOCATION	FLINT, MICHIGAN	SHEET NO.	2 OF 2
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-19-97
		SURFACE ELEV.	101.1
		BOREHOLE DIA.	8 IN.

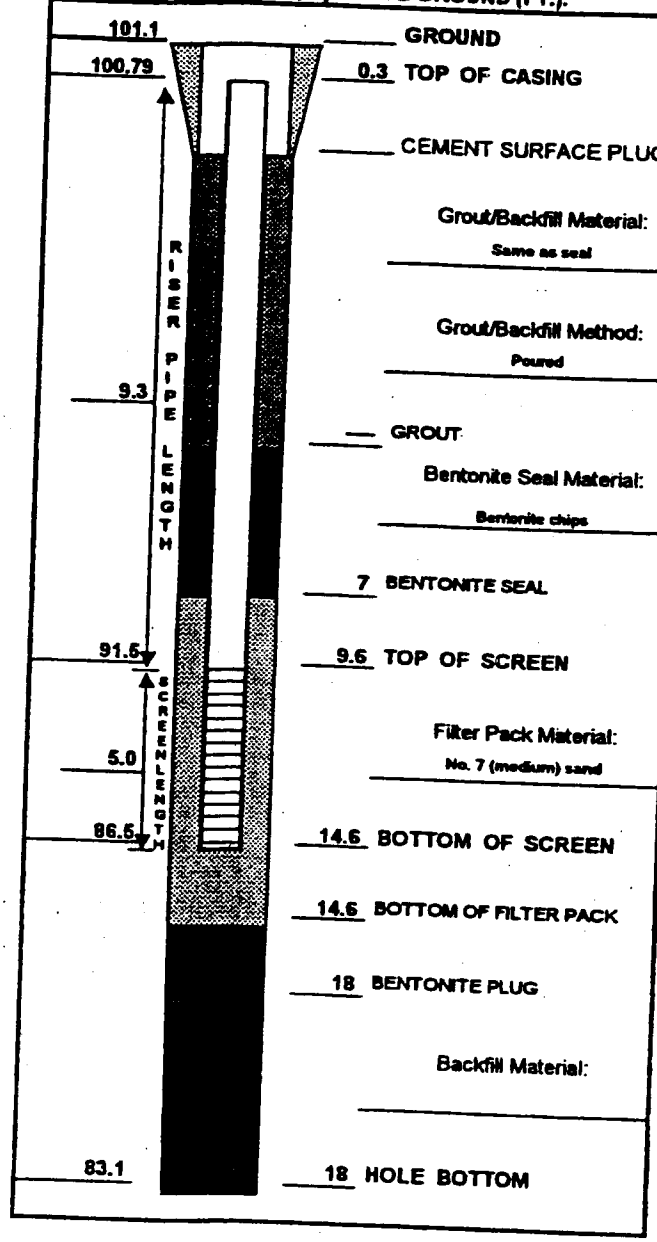
SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
7	SS	14	24	0		As above (CL).	
8	SS	15	24	0		As above (CL), medium stiff (Pp = 0.5 to 1.0).	
					15		
9	SS	12	24	0		As above (CL).	
						End of boring at 18 feet.	
					20		

ENVIRONMENTAL AUDIT REP(
PRIVILEGED DOCUMENT

RMT INC.

WELL CONSTRUCTION DIAGRAM

PROJECT:	Peregrine - Flint					WELL NO.:	PFW-8
PROJ. NO:	4036.05	DATE INSTALLED:		OBSV. BY:	DPR	CHECKED BY:	DPR



1. CASING AND SCREEN DETAILS:

A) Type Of Pipe: 2" PVC — Pipe Schedule: 40

B) Pipe Joints: Flush with O-ring

C) Solvent Used? No

D) Screen Type: 2" with machined slots, flush joint — Screen Slot Size: 0.01"

E) Borehole Diameter: 8 In. From 0 To 16 Ft.
3 In. From 16 To 18 Ft.

F) Surf. Casing Diameter: ____ In. From ____ To ____ Ft.
2nd Surface Casing: ____ In. From ____ To ____ Ft.

G) Installed Protective Cover W/ Lock? Yes

2. WELL DEVELOPMENT:

A) Method: None - dry well

B) Time Spent Developing: ____ Hours

C) Water Removed: ____ Gallons
Added: ____ Gallons

D) Water Clarity Before/After Development:
Before:
After:

F) Odor (Descr. if present) None

3. WATER LEVEL SUMMARY:

A) After Developing: ____ Ft. Below Top Of Casing

B) Other Date/Time: ____ Ft.
Other Date/Time: ____ Ft.

Notes:

ENVIRONMENTAL AUDIT REPO
PRIVILEGED DOCUMENT

LOG OF TEST BORING

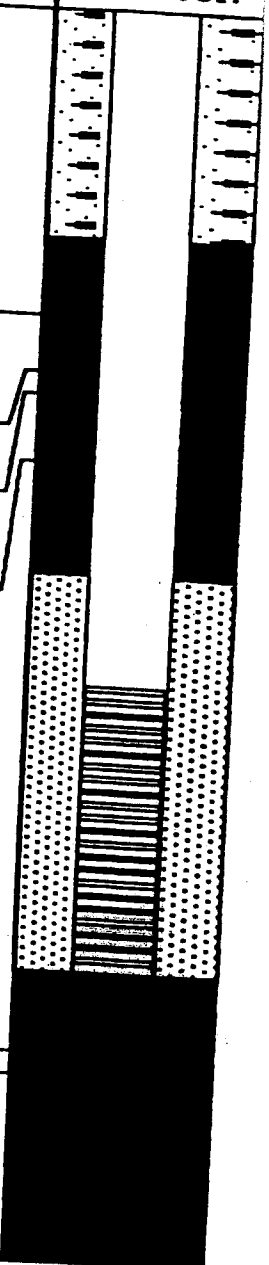
F-203 (R 01-87)

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFW-9
LOCATION	FLINT, MICHIGAN	SHEET NO.	1 OF 2
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-19-97
		SURFACE ELEV.	98.8
		BOREHOLE DIA.	8 IN.

SAMPLING NOTES

INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH	VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
1	SS	7	20	0		SANDY LEAN CLAY (CL), some fine to coarse sand, few fine to coarse gravel, slightly plastic, mottled brown, dark yellowish brown, and gray, moist, very stiff to hard, fractured (Glacial Till).
2	SS	13	24	0		As above (CL).
						WELL-GRADED SAND (SW), fine to coarse, trace silt, trace clay, dark yellowish brown 10YR 3/6, moist.
3	SS	6	20	0		SILT (ML), dark yellowish brown, moist, laminated.
						WELL-GRADED SAND (SW), fine to coarse, trace silt, trace clay, dark yellowish brown 10Y R3/6, moist.
					5 ▽	
4	SS	5	20	0		SANDY LEAN CLAY (CL), few fine to coarse gravel, slightly plastic, brown, moist, stiff (Pp = 1.5), abundant moist to wet sand partings below 5 feet (Glacial Till).
5	SS	8	19	0		As above (CL), very stiff (Pp = 2.2).
						PEAT, black humic material, some plant fragments, no odor.
6	SS	16	24	0	10	SANDY LEAN CLAY (CL), brown 10YR 4/3 with yellowish brown and gray mottling and fractures, moist, hard.

GENERAL WELL CONSTRUCT.



GENERAL NOTES

DATE STARTED	19 MAR 97
DATE COMPLETED	19 MAR 97
RIG	CME LC 60
CREW CHIEF	M. HEFFERAN
LOGGED	DPR CHECKED LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽	5.0 ft. bgl
AT COMPLETION ▼	
AFTER DRILLING	
CAVE-IN: DATE/TIME	DEPTH
WATER: DATE/TIME	DEPTH

ENVIRONMENTAL AUDIT REPORT
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFW-9
LOCATION	FLINT, MICHIGAN	SHEET NO.	2 OF 2
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-19-97
		SURFACE ELEV.	98.8
		BOREHOLE DIA.	8 IN.

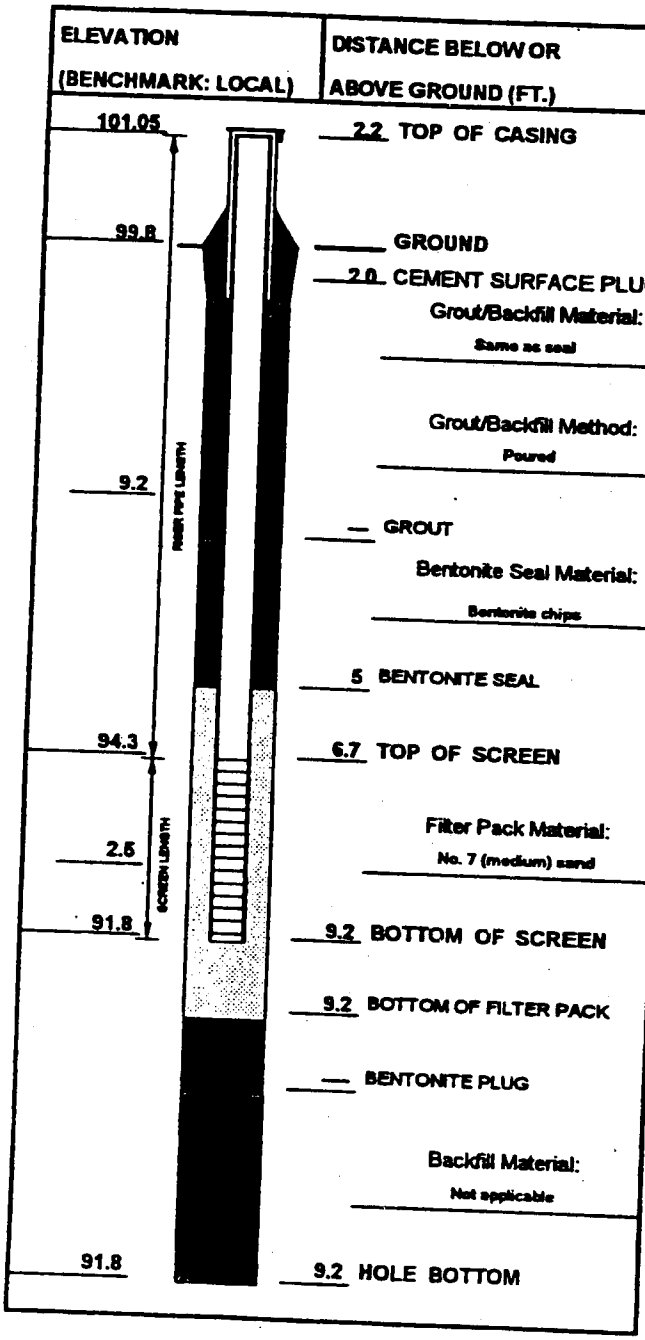
SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
7	SS	17	24	0		As above (CL), very stiff (Pp = 3.5 to 4.0), olive brown, continued mottling.	
						As above (CL).	
8	SS	37	24	0			
					15	As above (CL), hard (Pp > 4.5), mostly brown 10YR 4/3, gray along fractures.	
9	SS	39	24	0			
						As above (CL), fewer fractures.	
10	SS	46	24	0			
						As above (CL).	
					20	End of boring at 20 feet. Original boring backfilled with bentonite slurry. Moved 5 feet northwest to install well.	

ENVIRONMENTAL AUDIT REPOR
PRIVILEGED DOCUMENT

RMT INC.

WELL CONSTRUCTION DIAGRAM

PROJECT:	Peregrine - Flint					WELL NO.:	PFW-9
PROJ. NO:	4036.05	DATE INSTALLED:	3-19-97	OBSV. BY:	DPR	CHECKED BY:	DPR



1. CASING AND SCREEN DETAILS:

A) Type Of Pipe: 2" PVC — Pipe Schedule: 40

B) Pipe Joints: Flush with O-ring

C) Solvent Used? No

D) Screen Type: 2" with machined slots, flush joint — Screen Slot Size: 0.01"

E) Borehole Diameter: 8 In. From 0 To 8.5 Ft.
____ In. From ____ To ____ Ft.

F) Surf. Casing Diameter: ____ In. From ____ To ____ Ft.
2nd Surface Casing: ____ In. From ____ To ____ Ft.

G) Installed Protective Cover W/ Lock? Yes

2. WELL DEVELOPMENT:

A) Method: Gently bail

B) Time Spent Developing: 0.2 Hours

C) Water Removed: 1.5 Gallons
Added: 0 Gallons

D) Water Clarity Before/After Development:
Before: Clear
After: Slightly turbid, light brown

F) Odor (Descr. if present) None

3. WATER LEVEL SUMMARY:

A) After Developing: ____ Ft. Below Top Of Casing

B) Other Date/Time: 3-21-97/1515 dry Ft.
Other Date/Time: 3-31-97/0938 8.53 Ft.

Notes:

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: STEARNS DRILLING CO
DRILLING METHOD: 4.25" HSA

BORING NO.: PFW-10
SHEET NO.: 1 OF 2
PROJECT NO.: 4036.05
INSTALLATION: 3-20-97
SURFACE ELEV.: 100.5
BOREHOLE DIA.: 8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
1	SS	11	15	0.1		Asphalt. Fill: fine to medium sand, little coarse gravel, dark yellowish brown, moist to wet.	
2	SS	12	20	0.2		SANDY LEAN CLAY (CL), some fine to coarse sand, few fine to coarse gravel, slightly plastic, brown 10YR 4/3 with some gray mottling along fractures, moist, hard (Pp > 4.5) (Glacial Till).	
3	SS	18	24	0		As above (CL), very dark gray 10YR 3/1 to olive brown 2.5Y 3/6 to grayish brown 10YR 4/2, stiff to very stiff (Pp = 1.5 to 2.5).	
					5	As above (CL), brown 10YR 4/3, hard (Pp = 4.4) below 5 feet.	
4	SS	29	24	0		As above (CL), less gray mottling below 7 feet.	
5	SS	29	24	0		As above (CL), (Pp = 4.0 to 4.5), fracture from 9.2 to 9.8 ft.	
6	SS	24	24	0	10	As above (CL), fracture 11.5 to 11.8'.	

GENERAL NOTES

DATE STARTED: 20 MAR 97
DATE COMPLETED: 20 MAR 97
RIG: CME LC 60
CREW CHIEF: M. HEFFERAN
LOGGED: DPR CHECKED: LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽: 13.8 ft. bgl
AT COMPLETION ▼:
AFTER DRILLING
CAVE-IN: DATE/TIME _____ DEPTH _____
WATER: DATE/TIME _____ DEPTH _____

ENVIRONMENTAL AUDIT REP(
PRIVILEGED DOCUMENT

LOG OF TEST BORING

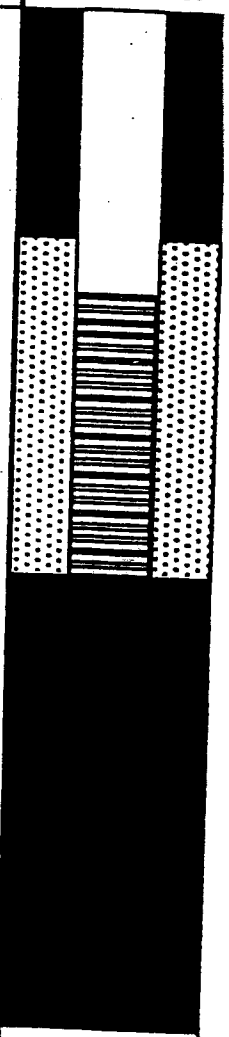
F-203 (R 01-87)

RMT

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: STEARNS DRILLING CO
DRILLING METHOD: 4.25" HSA

BORING NO.: PFW-10
SHEET NO.: 2 OF 2
PROJECT NO.: 4036.05
INSTALLATION: 3-20-97
SURFACE ELEV.: 100.5
BOREHOLE DIA.: 8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
7	SS	24	22	0		As above (CL), areas of dark grayish brown below 12.2 ft, very stiff (Pp = 2.5 to 3.5).	
8	SS	19	24	0		<— Wet sand parting at 13.8 feet.	
					15	As above (CL), dark gray 10YR 4/1, stiff (Pp = 1.7).	
9	SS	14	24	0		As above (CL).	
10	SS	13	24	0		As above (CL).	
					20	End of boring at 20 feet.	

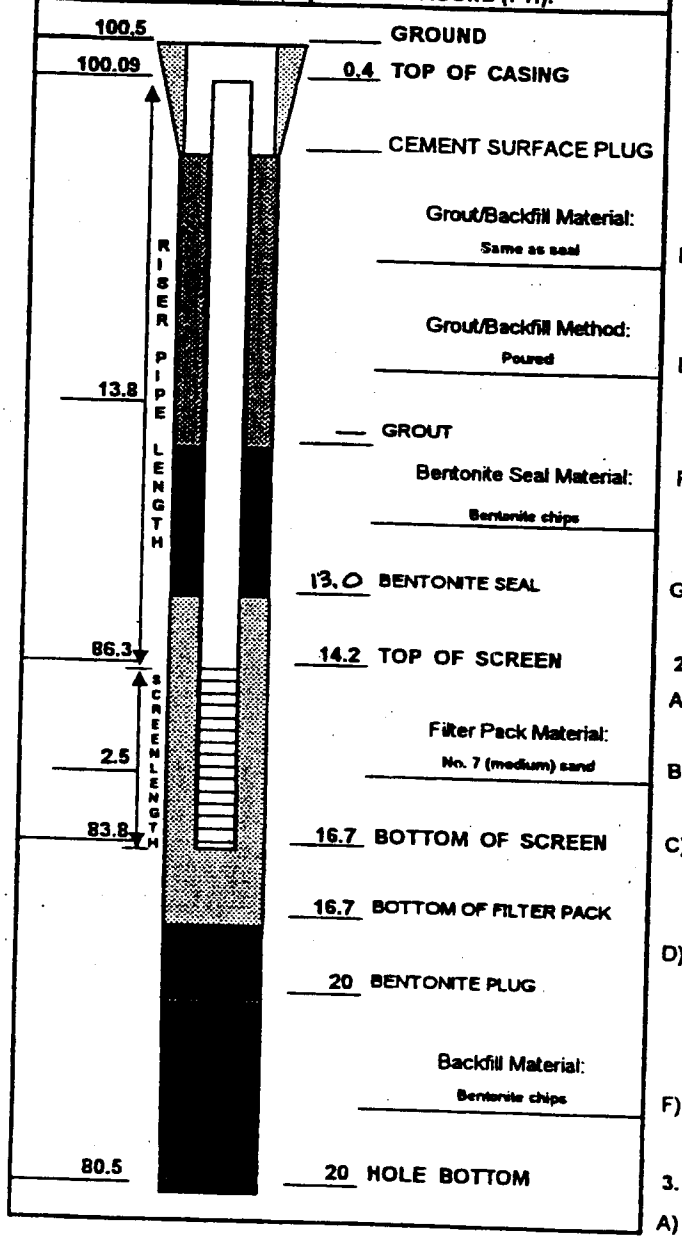


ENVIRONMENTAL AUDIT REPORT
PRIVILEGED DOCUMENT

RMT INC.

WELL CONSTRUCTION DIAGRAM

PROJECT:	Peregrine - Flint					WELL NO.:	PFW-10
PROJ. NO:	4036.05	DATE INSTALLED:	3-20-97	OBSV. BY:	DPR	CHECKED BY:	



1. CASING AND SCREEN DETAILS:

A) Type Of Pipe: 2" PVC — Pipe Schedule: 40

B) Pipe Joints: Flush with O-ring

C) Solvent Used? No

D) Screen Type: 2" with machined slots, flush joint — Screen Slot Size: 0.01"

E) Borehole Diameter: 8 In. From 0 To 18 Ft.
3 In. From 18 To 20 Ft.

F) Surf. Casing Diameter: ____ In. From ____ To ____ Ft.
2nd Surface Casing: ____ In. From ____ To ____ Ft.

G) Installed Protective Cover W/ Lock? Yes

2. WELL DEVELOPMENT:

A) Method: Gently bail

B) Time Spent Developing: 0.2 Hours

C) Water Removed: 1.5 Gallons
Added: 0 Gallons

D) Water Clarity Before/After Development:
Before: Slightly turbid, light brown
After: Clear

F) Odor (Descr. if present) None

3. WATER LEVEL SUMMARY:

A) After Developing: _____ Ft. Below Top Of Casing

B) Other Date/Time: 3-20-97/1145 dry Ft.
Other Date/Time: 3-31-97/1410 11.67 Ft.

Notes:

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: STEARNS DRILLING CO
DRILLING METHOD: 4.25" HSA

BORING NO. PFW-11
SHEET NO. 1 OF 2
PROJECT NO. 4036.05
INSTALLATION 3-20-97
SURFACE ELEV. 101.3
BOREHOLE DIA. 8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
1	SS	19	18	0		Asphalt and concrete.	
						Fill: medium sand, dark yellowish brown, moist.	
						SANDY LEAN CLAY (CL), some fine to coarse sand, few fine to coarse gravel, slightly plastic, brown 10YR 4/3 with some yellowish brown mottling and gray along fractures, moist, hard (Pp > 4.5) (Glacial Till).	
2	SS	23	24	0			
3	SS	24	24	0		As above (CL).	
					5	SILT (ML), dark yellowish brown, moist, stratified.	
						LEAN CLAY (CL), dark grayish brown, hard, moist, laminated.	
						SILT (ML), brown, moist, laminated.	

GENERAL NOTES

DATE STARTED 20 MAR 97
DATE COMPLETED 20 MAR 97
RIG CME LC 60
CREW CHIEF M. HEFFERAN
LOGGED DPR CHECKED LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽ 6.9 ft. bgl
AT COMPLETION ▼
AFTER DRILLING
CAVE-IN: DATE/TIME ____ DEPTH ____
WATER: DATE/TIME ____ DEPTH ____

ENVIRONMENTAL AUDIT REPORT
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFW-11
LOCATION	FLINT, MICHIGAN	SHEET NO.	2 OF 2
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-20-97
		SURFACE ELEV.	101.3
		BOREHOLE DIA.	8 IN.

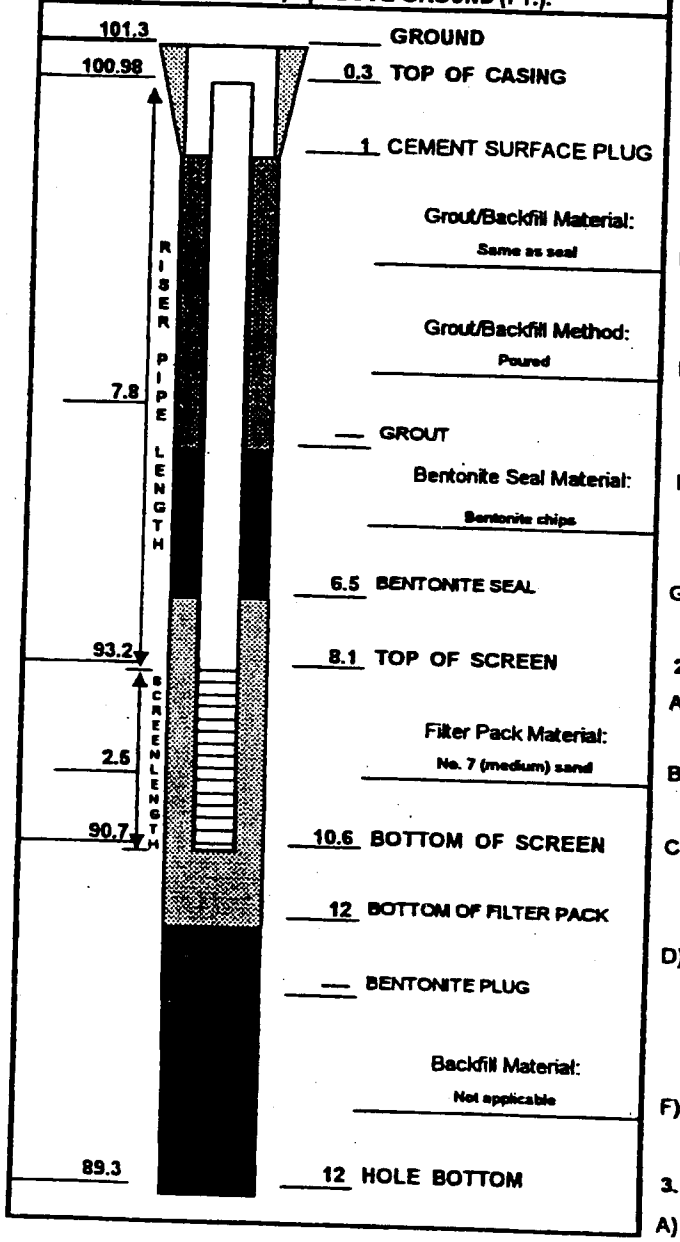
SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	INTERVAL TYPE	RECOVERY N	RECOVERY IN	PID	DEPTH		
4	SS	19	20	0		SILTY SAND (SM), fine, very pale brown 10YR 7/3, moist.	
						SANDY LEAN CLAY (CL), gray 10YR 5/1, moist, very stiff.	
						SANDY SILT (ML), little fine sand, gray 10YR 5/1, moist to wet, stratified and laminated.	
5	SS	21	24			As above (ML).	
						POORLY-GRADED SAND (SP), wet.	
						SANDY SILT (ML), wet.	
						POORLY-GRADED SAND (SP), wet.	
						SILT (ML), wet.	
6	SS	14	20		10	SANDY SILT (ML), wet.	
						POORLY-GRADED SAND (SP), wet.	
						SANDY SILT (ML), wet.	
						SANDY LEAN CLAY (CL), some fine to coarse sand, few fine to coarse gravel, slightly plastic, dark gray 10YR 4/1, moist, very stiff (Pp = 3.3).	
						End of boring at 12 feet.	

ENVIRONMENTAL AUDIT REPC
PRIVILEGED DOCUMENT

RMT INC.

WELL CONSTRUCTION DIAGRAM

PROJECT:	Peregrine - Flint					WELL NO.:	PFW-11
PROJ. NO:	4036.05	DATE INSTALLED:	3-20-97	OBSV. BY:	DPR	CHECKED BY:	DPR



1. CASING AND SCREEN DETAILS:

A) Type Of Pipe: 2" PVC — Pipe Schedule: 40

B) Pipe Joints: Flush with O-ring

C) Solvent Used? No

D) Screen Type: 2" with machined slots, flush joint — Screen Slot Size: 0.01"

E) Borehole Diameter: 8 In. From 0 To 11 Ft.
3 In. From 11 To 12 Ft.

F) Surf. Casing Diameter: ____ In. From ____ To ____ Ft.
2nd Surface Casing: ____ In. From ____ To ____ Ft.

G) Installed Protective Cover W/ Lock? Yes

2. WELL DEVELOPMENT:

A) Method: Gently bail

B) Time Spent Developing: 0.3 Hours

C) Water Removed: 1.6 Gallons
Added: 0 Gallons

D) Water Clarity Before/After Development:
Before: Slightly turbid, light brown
After: Moderately turbid, light brown

F) Odor (Descr. if present) None

3. WATER LEVEL SUMMARY:

A) After Developing: (dry) Ft. Below Top Of Casing

B) Other Date/Time: 3-20-97/1400 7.2 Ft.
Other Date/Time: 3-31-97/1406 1.57 Ft.

Notes:

ENVIRONMENTAL AUDIT REPOR
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFW-12
LOCATION	FLINT, MICHIGAN	SHEET NO.	1 OF 2
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-21-97
		SURFACE ELEV.	101.7
		BOREHOLE DIA.	8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
						Concrete.	
1	SS	21	15	12		Fill: wood blocks.	
						SANDY LEAN CLAY (CL), some fine to coarse sand, few fine to coarse gravel, slightly plastic, dark grayish brown to brown 10YR 4/3 with gray mottling, moist, very stiff to hard, fractures, strong hydrocarbon odor.	
2	SS	26	24	8.1			
						As above (CL), hard, less hydrocarbon odor.	
3	SS	28	24	23.3			
					5		
						As above (CL), heavy fracturing with dark red precipitate along with gray fractures.	
4	SS	23	24	28.3			
						As above (CL).	
5	SS	23	24	5.6			
						As above (CL), fewer fractures, dark grayish brown.	
6	SS	20	24	0	10		

GENERAL NOTES

DATE STARTED	21 MAR 97
DATE COMPLETED	21 MAR 97
RIG	CME LC 60
CREW CHIEF	M. HEFFERAN
LOGGED	DPR CHECKED LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽	17.8 ft. bgl
AT COMPLETION ▼	
AFTER DRILLING	
CAVE-IN: DATE/TIME	DEPTH
WATER: DATE/TIME	DEPTH

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: STEARNS DRILLING CO
DRILLING METHOD: 4.25" HSA

BORING NO.: PFW-12
SHEET NO.: 2 OF 2
PROJECT NO.: 4036.05
INSTALLATION: 3-21-97
SURFACE ELEV.: 101.7
BOREHOLE DIA.: 8 IN.

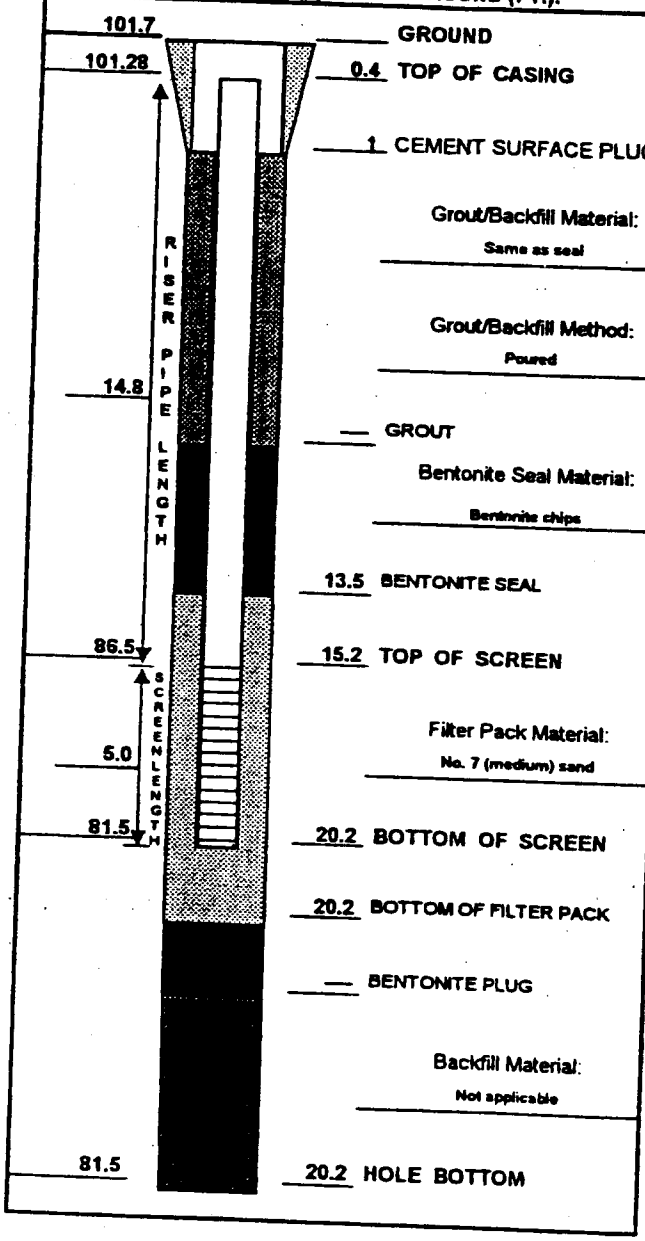
SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS	GENERAL WELL CONSTRUCT.
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH		
7	SS	20	24	6		As above (CL), no odor.	
						As above (CL).	
8	SS	24	24	3.1		WELL-GRADED SAND (SW), fine to coarse, trace silt, trace clay, yellow to brown to dark gray and black, hydrocarbon odor, moist.	
					15	SANDY LEAN CLAY (CL), some fine to coarse sand, few fine to coarse gravel, slightly plastic, dark grayish brown, moist, stiff (Pp = 1.5 to 2.0), some sand partings.	
9	SS	16	24	0			
						SANDY SILT (ML), dark grayish brown, moist.	
10	SS	8	20		▽	SILTY SAND (SM), dark grayish brown, wet, some laminated with sandy silt and clayey silt.	
					20	SANDY LEAN CLAY (CL), slightly plastic, dark gray 10YR 4/1, medium stiff (Pp = 0.8), moist.	
						End of boring at 20 feet.	

RMT INC.

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

WELL CONSTRUCTION DIAGRAM

PROJECT:	Peregrine - Flint					WELL NO.:	PFW-12
PROJ. NO:	4036.05	DATE INSTALLED:	3-21-97	OBSV. BY:	DPR	CHECKED BY:	DPR



1. CASING AND SCREEN DETAILS:

A) Type Of Pipe: 2" PVC — Pipe Schedule: 40

B) Pipe Joints: Flush with O-ring

C) Solvent Used? No

D) Screen Type: 2" with machined slots, flush joint — Screen Slot Size: 0.01"

E) Borehole Diameter: 8 In. From 0 To 19.5 Ft.
3 In. From 19.5 To 20 Ft.

F) Surf. Casing Diameter: ____ In. From ____ To ____ Ft.
2nd Surface Casing: ____ In. From ____ To ____ Ft.

G) Installed Protective Cover W/ Lock? Yes

2. WELL DEVELOPMENT:

A) Method: Gently bail

B) Time Spent Developing: 1 Hours

C) Water Removed: 5.9 Gallons
Added: 0 Gallons

D) Water Clarity Before/After Development:
Before: Opaque dark grayish brown
After: Slightly turbid (approximately 100 NTU)

F) Odor (Descr. if present) None

3. WATER LEVEL SUMMARY:

A) After Developing: ____ Ft. Below Top Of Casing

B) Other Date/Time: 3-24-97/1040 15.19 Ft.
Other Date/Time: 3-31-97/1418 15.21 Ft.

Notes:

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFB-13
LOCATION	FLINT, MICHIGAN	SHEET NO.	1 OF 1
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-24-97
		SURFACE ELEV.	101.7
		BOREHOLE DIA.	8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH	
						Concrete.
1	SS	12	13	0.8		Fill: sand and coarse gravel, brown, no odor, dry to moist.
2	SS	15	19	0		SANDY LEAN CLAY (CL), some fine to coarse sand, few gravel, slightly plastic, grayish brown 10YR 5/2 to dark grayish brown 10YR 5/3 with gray along fractures, dry to moist, hard (Pp > 4.5) (Glacial Till).
3	SS	13	18	0		As above (CL), no odor, moist, very stiff (Pp = 3.6), fracture at 3.3'.
					5	
4	SS	17	20	0		As above (CL), few fractures.
5	SS	28	24	0		As above (CL), little gravel, mottled dark grayish brown, dark gray, and dark yellowish brown, very stiff to hard (Pp = 3.3 to 4.5).
						As above (CL), brown 10YR 4/3, dark gray along occasional fractures.
					10	End of boring at 10 feet. Boring backfilled with bentonite chips.

GENERAL NOTES

DATE STARTED 24 MAR 97
DATE COMPLETED 24 MAR 97
RIG CME LC 60
CREW CHIEF M. HEFFERAN
LOGGED DPR CHECKED LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽ none observed
AT COMPLETION ▼
AFTER DRILLING
CAVE-IN: DATE/TIME _____ DEPTH _____
WATER: DATE/TIME _____ DEPTH _____

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: STEARNS DRILLING CO
DRILLING METHOD: 4.25" HSA

BORING NO.: PFB-14
SHEET NO.: 1 OF 1
PROJECT NO.: 4036.05
INSTALLATION: 3-25-97
SURFACE ELEV.: 101.7
BOREHOLE DIA.: 8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
INTERVAL NO.	INTERVAL TYPE	RECOVERY N	RECOVERY IN	PID	DEPTH	
						Concrete.
1	SS	NA	12			Fill: medium sand, dark yellowish brown, no odor, moist (backfill from die cast area pit).
2	SS	NA	16			
						Split spoon refusal at 4.2 feet.
						Concrete.
3	SS	12	12	29.2	5	Fill: medium to coarse sand, dark gray, moist, strong hydrocarbon odor.
4	SS	10	22	23.2		SANDY LEAN CLAY (CL), some sand, few gravel, slightly plastic, dark gray 10YR 4/1, strong hydrocarbon odor, moist, stiff (Pp = 1.6) (Glacial Till).
						As above (CL), less odor.
5	SS	12	24	11.2		As above (CL), medium stiff (Pp = 0.8), 1/8" sand parting @ 8.3 feet.
					10	End of boring at 10 feet. Boring backfilled with bentonite chips.

GENERAL NOTES

DATE STARTED: 24 MAR 97
DATE COMPLETED: 25 MAR 97
RIG: CME LC 60
CREW CHIEF: M. HEFFERAN
LOGGED: DPR CHECKED: LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽: none observed
AT COMPLETION ▼:
AFTER DRILLING
CAVE-IN: DATE/TIME: DEPTH:
WATER: DATE/TIME: DEPTH:

ENVIRONMENTAL AUDIT REPORT
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LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: STEARNS DRILLING CO
DRILLING METHOD: 4.25" HSA

BORING NO. PFB-15
SHEET NO. 1 OF 2
PROJECT NO. 4036.05
INSTALLATION 3-24-97
SURFACE ELEV.
BOREHOLE DIA. 8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH	
						Concrete.
1	SS	15	17	0		Fill: medium sand, dark yellowish brown, no odor, moist.
2	SS	35	15	0		SANDY LEAN CLAY (CL), some sand, few gravel, slightly plastic, brown 10YR 4/3, no odor, moist, hard (Pp > 4.5), occasional fracture.
3	SS	19	20	0		As above (CL), dark grayish brown 10YR 4/2 below 3.1 ft.
					5	As above (CL), hard to stiff, no fracturing.
4	SS	18	22	0		
						As above (CL).
5	SS	17	24	0		
						As above (CL).
6	SS	18	24	0	10	

GENERAL NOTES

DATE STARTED 24 MAR 97
DATE COMPLETED 24 MAR 97
RIG CME LC 60
CREW CHIEF M. HEFFERAN
LOGGED DPR CHECKED LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽ none observed
AT COMPLETION ▼
AFTER DRILLING
CAVE-IN: DATE/TIME DEPTH
WATER: DATE/TIME DEPTH

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFB-15
LOCATION	FLINT, MICHIGAN	SHEET NO.	2 OF 2
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-24-97
		SURFACE ELEV.	
		BOREHOLE DIA.	8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
INTERVAL NO.	INTERVAL TYPE	RECOVERY N	RECOVERY IN	PID	DEPTH	
7	SS	17	24	0		As above (CL), 1/4" sand parting @ 11.2 ft, no odor.
						As above (CL).
						End of boring at 14 feet. Boring backfilled with bentonite chips.
					15	
					20	

ENVIRONMENTAL AUDIT REPOR
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFB-16
LOCATION	FLINT, MICHIGAN	SHEET NO.	1 OF 1
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-25-97
		SURFACE ELEV.	101.7
		BOREHOLE DIA.	8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH	
						Concrete.
1	SS	6	12			Fill: fine to medium sand, dark yellowish brown, moist, no odor.
2	SS	5	6			Concrete.
3	SS	15	22	32.2		SANDY LEAN CLAY (CL), some sand, few gravel, slightly plastic, dark gray 10YR 4/1, no odor, moist, very stiff (Pp = 3.3), occasional fracture.
4	SS	15	24	4.4	5	As above (CL), 1/8" silty sand parting @ 5.4 ft, no fractures.
5	SS	16	24	5.6		As above (CL), (Pp = 2.2 to 2.5).
6	SS	6	12	22.2		As above (CL).
					10	End of boring at 10 feet. Boring backfilled with bentonite chips.

GENERAL NOTES

DATE STARTED 25 MAR 97
DATE COMPLETED 25 MAR 97
RIG CME LC 60
CREW CHIEF M. HEFFERAN
LOGGED DPR CHECKED LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽ none observed
AT COMPLETION ▼
AFTER DRILLING
CAVE-IN: DATE/TIME _____ DEPTH _____
WATER: DATE/TIME _____ DEPTH _____

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: STEARNS DRILLING CO
DRILLING METHOD: 4.25" HSA

BORING NO. PFB-17
SHEET NO. 1 OF 1
PROJECT NO. 4036.05
INSTALLATION 3-26-97
SURFACE ELEV.
BOREHOLE DIA. 8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH	
						Concrete.
1	SS	10	15	0		Fill: fine sand, brown 10YR 4/3, pieces of dark gray sandy lean clay, no odor, dry to moist.
2	SS	4	22	0		As above.
3	SS	10	24	0		As above, dark yellowish brown.
					5	
4	SS	26	20	0		Fill: silty fine sand, small pieces of dark gray sandy lean clay, brown to grayish brown, no odor, dry to moist.
						SANDY SILT (ML), light brownish gray 10YR 6/2, dry to moist, laminated and stratified.
5	SS	14	20	0		LEAN CLAY (CL), medium plastic, dark gray, moist, stiff (Pp = 1.4), increasingly laminated.
					10	SANDY LEAN CLAY (CL), some sand, few gravel, slightly plastic, dark gray 10YR 4/1, moist, stiff (Glacial Till).
						End of boring at 10 feet. Boring backfilled with bentonite chips.

GENERAL NOTES

DATE STARTED 26 MAR 97
DATE COMPLETED 26 MAR 97
RIG CME LC 60
CREW CHIEF M. HEFFERAN
LOGGED DPR CHECKED LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽ none observed
AT COMPLETION ▼
AFTER DRILLING
CAVE-IN: DATE/TIME ____ DEPTH ____
WATER: DATE/TIME ____ DEPTH ____

ENVIRONMENTAL AUDIT REPORT
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: STEARNS DRILLING CO
DRILLING METHOD: 4.25" HSA

BORING NO.: PFB-18
SHEET NO.: 1 OF 1
PROJECT NO.: 4036.05
INSTALLATION: 3-26-97
SURFACE ELEV.:
BOREHOLE DIA.: 8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH	
						Concrete.
1	SS	15	16	0.6		Fill: fine sand with silt, dark yellowish brown, dry to moist.
2	SS	19	24	0		SANDY LEAN CLAY (CL), some sand, few gravel, slightly plastic, dark gray 10YR 4/1, no odor, dry to moist, hard, occasional gray silt parting (Glacial Till).
3	SS	12	24	0		As above (CL), moist, very stiff (Pp = 3.3 to 3.8).
					5	As above (CL).
4	SS	13	18	0		As above (CL), (Pp = 2.7 to 3.3).
						SANDY SILT (ML), dark grayish brown, moist.
5	SS	15	24	0		SANDY LEAN CLAY (CL), some sand, few gravel, slightly plastic, dark gray 10YR 4/1, no odor, moist, stiff (Pp = 1.7), no silt partings.
					10	End of boring at 10 feet. Boring backfilled with bentonite chips.

GENERAL NOTES

DATE STARTED: 26 MAR 97
DATE COMPLETED: 26 MAR 97
RIG: CME LC 60
CREW CHIEF: M. HEFFERAN
LOGGED: DPR CHECKED: LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽: none observed
AT COMPLETION ▼:
AFTER DRILLING
CAVE-IN: DATE/TIME _____ DEPTH _____
WATER: DATE/TIME _____ DEPTH _____

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: STEARNS DRILLING CO
DRILLING METHOD: 4.25" HSA

BORING NO.: PFB-19
SHEET NO.: 1 OF 1
PROJECT NO.: 4036.05
INSTALLATION: 3-26-97
SURFACE ELEV.:
BOREHOLE DIA.: 8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH	
						Concrete.
1	SS	12	16	0		Fill: silty fine sand, dark yellowish brown, dry to moist.
2	SS	12	24	0		SANDY LEAN CLAY (CL), some sand, few gravel, slightly plastic, dark gray 10YR 4/1, no odor, dry to moist, hard (Pp > 4.5) (Glacial Till).
3	SS	14	20	0		As above (CL), dark yellowish brown along fractures, very stiff (Pp = 2.7 to 3.3).
					5	As above (CL), no fractures.
4	SS	12	24	0		As above (CL), (Pp = 2.1).
5	SS	12	24	0		<— 1/8" moist gray silty sand parting @ 8.3 ft.
						As above (CL), but gray to dark gray along occasional fractures, stiff (Pp = 1.0).
					10	End of boring at 10 feet. Boring backfilled with bentonite chips.

GENERAL NOTES

DATE STARTED: 26 MAR 97
DATE COMPLETED: 26 MAR 97
RIG: CME LC 60
CREW CHIEF: M. HEFFERAN
LOGGED: DPR CHECKED: LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽: none observed
AT COMPLETION ▼:
AFTER DRILLING
CAVE-IN: DATE/TIME: DEPTH:
WATER: DATE/TIME: DEPTH:

ENVIRONMENTAL AUDIT REPORT
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

KMT

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFB-20
LOCATION	FLINT, MICHIGAN	SHEET NO.	1 OF 1
CONTRACTOR	STEARNS DRILLING CO	PROJECT NO.	4036.05
DRILLING METHOD	4.25" HSA	INSTALLATION	3-26-97
		SURFACE ELEV.	
		BOREHOLE DIA.	8 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
INTERVAL NO.	TYPE	RECOVERY N	IN	PID	DEPTH	
						Concrete.
1	SS	17	20	0		Fill: silty fine sand, dark yellowish brown, no odor, moist.
						SANDY LEAN CLAY (CL), some sand, few gravel, slightly plastic, dark gray 10YR 4/1, dry to moist, hard (Pp > 4.5), occasional fractures (Glacial Till).
2	SS	11	22	0		SANDY SILT (ML), pale brown, moist.
						SANDY LEAN CLAY (CL), some sand, few gravel, slightly plastic, dark gray 10YR 4/1, dry to moist, very stiff (Pp = 3.3 to 3.8), occasional fractures.
3	SS	8	24	0	5	SANDY SILT (ML), dark yellowish brown, moist.
						SANDY LEAN CLAY (CL), some sand, few gravel, slightly plastic, dark gray 10YR 4/1, dry to moist, stiff (Pp = 1.4 to 1.8), gray along occasional fractures.
4	SS	NA	24	0		As above (CL), no fractures.
5	SS	NA	12	0		As above (CL).
					10	End of boring at 10 feet. Boring backfilled with bentonite chips.

GENERAL NOTES

DATE STARTED	26 MAR 97
DATE COMPLETED	26 MAR 97
RIG	CME LC 60
CREW CHIEF	M. HEFFERAN
LOGGED	DPR CHECKED LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽	none observed
AT COMPLETION ▼	
AFTER DRILLING	
CAVE-IN: DATE/TIME	DEPTH
WATER: DATE/TIME	DEPTH

ENVIRONMENTAL AUDIT REPORT
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

RMT

PROJECT NAME	PEREGRINE FLINT	BORING NO.	PFB-21
LOCATION	FLINT, MICHIGAN	SHEET NO.	1 OF 1
CONTRACTOR	FIBERTEC	PROJECT NO.	4036.05
DRILLING METHOD	DIRECT PUSH	INSTALLATION	4-24-97
		SURFACE ELEV.	
		BOREHOLE DIA.	2 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
INTERVAL NO.	TYPE	RECOVERY N	IN	MOISTURE	DEPTH	
1	GP		27	M		FILL: Mostly fine to medium sand, little fine gravel, black, moist, oily residue and heavy hydrocarbon odor, PID = 5.1.
						FILL: poorly-graded sand, fine, yellowish brown, moist, slight odor above 1.5 feet.
2	GP		34	M/W		As above.
					5	FILL: mostly poorly-graded fine sand, little lean clay, mostly brown to dark brown, slight hydrocarbon odor, moist, PID = 1.0 .
						FILL: poorly-graded sand, fine, yellowish brown with occasional black streaks, no odor, wet @ 6.6 ft.
3	GP		18	W		As above.
						Obstruction @ 9.5 ft.
					10	End of boring at 9.5 feet. Collected groundwater sample from 1" PVC temporary well screened from 4.5 to 9.5 ft.

GENERAL NOTES

DATE STARTED	24 APR 97
DATE COMPLETED	24 APR 97
RIG	GEOPROBE
CREW CHIEF	K. BELL
LOGGED	DPR CHECKED LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING	6.6 ft. bgl
AT COMPLETION	7.7 ft. bgl
AFTER DRILLING	
CAVE-IN: DATE/TIME	DEPTH
WATER: DATE/TIME	DEPTH

ENVIRONMENTAL AUDIT REPC
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: FIBERTEC
DRILLING METHOD: DIRECT PUSH

BORING NO.: PFB-22
SHEET NO.: 1 OF 1
PROJECT NO.: 4036.05
INSTALLATION: 4-24-97
SURFACE ELEV.:
BOREHOLE DIA.: 2 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
INTERVAL NO.	INTERVAL TYPE	RECOVERY N	RECOVERY IN	MOISTURE	DEPTH	
1	GP		34	M		Topsoil.
						FILL: mostly lean clay with gravel, some areas of yellowish brown sand, brown, dark grayish brown, no odor, moist, stiff (reworked glacial till).
						FILL: poorly graded sand, fine, yellow brown, no odor, moist.
2	GP		28	M		
					5	FILL: poorly-graded sand, fine, few medium to coarse sand, trace fine gravel, trace silt and clay, yellow brown, no odor, moist.
						End of boring at 8 feet.
					10	

GENERAL NOTES

DATE STARTED: 24 APR 97
DATE COMPLETED: 24 APR 97
RIG: GEOPROBE
CREW CHIEF: K. BELL
LOGGED: DPR CHECKED: TPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING: ▽ none observed
AT COMPLETION: ▼
AFTER DRILLING
CAVE-IN: DATE/TIME _____ DEPTH _____
WATER: DATE/TIME _____ DEPTH _____

ENVIRONMENTAL AUDIT REPO
PRIVILEGED DOCU

LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: FIBERTEC
DRILLING METHOD: DIRECT PUSH

BORING NO.: PFB-23
SHEET NO.: 1 OF 1
PROJECT NO.: 4036.05
INSTALLATION: 4-24-97
SURFACE ELEV.:
BOREHOLE DIA.: 2 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
INTERVAL NO.	TYPE	RECOVERY N	IN	MOISTURE	DEPTH	
1	GP		48	M		Coal.
						SANDY LEAN CLAY (CL), some fine to coarse sand, few percent fine to coarse subangular gravel, occasional silt and sand parting, mottled mostly brown, dark yellowish brown, with gray along abundant fractures, no odor, moist, hard, PID = 0.2.
2	GP		48	M		
					5	As above (CL), mostly dark olive brown 2.5Y 4/2 below 5.8 ft, increasing moisture, very stiff, PID =0.1 .
						As above (CL), PID = 0.1.
						End of boring at 8 feet.
					10	

GENERAL NOTES

DATE STARTED: 24 APR 97
DATE COMPLETED: 24 APR 97
RIG: GEOPROBE
CREW CHIEF: K. BELL
LOGGED: DPR CHECKED: LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽: none observed
AT COMPLETION ▼:
AFTER DRILLING
CAVE-IN: DATE/TIME _____ DEPTH _____
WATER: DATE/TIME _____ DEPTH _____

ENVIRONMENTAL AUDIT REPO
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: FIBERTEC
DRILLING METHOD: DIRECT PUSH

BORING NO.: PFB-24
SHEET NO.: 1 OF 1
PROJECT NO.: 4036.05
INSTALLATION: 4-24-97
SURFACE ELEV.:
BOREHOLE DIA.: 2 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
INTERVAL NO.	INTERVAL TYPE	RECOVERY N	RECOVERY IN	MOISTURE	DEPTH	
1	GP		48	M		Coal.
						SANDY LEAN CLAY (CL), some fine to coarse sand, few percent fine to coarse subangular gravel, occasional silt and sand parting, mottled mostly brown, dark yellowish brown, with gray along abundant fractures, no odor, moist, hard, PID = 0.1.
2	GP		48	M		
					5	As above (CL), increasing moisture, very stiff, PID = 0.1.
						As above (CL), PID = 0.1.
						End of boring at 8 feet.
					10	

GENERAL NOTES

DATE STARTED: 24 APR 97
DATE COMPLETED: 24 APR 97
RIG: GEOPROBE
CREW CHIEF: K. BELL
LOGGED: DPR CHECKED: LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING: none observed
AT COMPLETION:
AFTER DRILLING
CAVE-IN: DATE/TIME: DEPTH:
WATER: DATE/TIME: DEPTH:

ENVIRONMENTAL AUDIT REPORT:
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: FIBERTEC
DRILLING METHOD: DIRECT PUSH

BORING NO.: PFB-25
SHEET NO.: 1 OF 1
PROJECT NO.: 4036.05
INSTALLATION: 4-24-97
SURFACE ELEV.:
BOREHOLE DIA.: 2 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
INTERVAL NO.	INTERVAL TYPE	RECOVERY N	RECOVERY IN	MOISTURE	DEPTH	
1	GP		32	M		Concrete.
						FILL: poorly-graded sand, fine, yellow brown, no odor, moist, PID = 0.1.
2	GP		34	M	5	SANDY LEAN CLAY (CL), some fine to coarse sand, few percent fine gravel, slightly plastic, mottled mostly brown, dark yellowish brown, with gray along abundant fractures, no odor, moist, hard (Glacial Till), PID = 0.2.
						As above (CL), PID = 0.2.
3	GP		48	M		As above (CL), reddish black precipitate along fractures, mostly gray below 9.5 feet, no odor, very stiff, PID = 0.2.
					10	
						As above (CL), yellowish brown in fewer fractures, PID = 0.2.
						End of boring at 12 feet.

GENERAL NOTES

DATE STARTED: 24 APR 97
DATE COMPLETED: 24 APR 97
RIG: GEOPROBE
CREW CHIEF: K. BELL
LOGGED: DPR CHECKED: LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽: none observed
AT COMPLETION ▼:
AFTER DRILLING
CAVE-IN: DATE/TIME: DEPTH:
WATER: DATE/TIME: DEPTH:

ENVIRONMENTAL AUDIT REPOR
PRIVILEGED DOCUMENT

LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: FIBERTEC
DRILLING METHOD: DIRECT PUSH

BORING NO. PFB-26
SHEET NO. 1 OF 1
PROJECT NO. 4036.05
INSTALLATION 4-25-97
SURFACE ELEV.
BOREHOLE DIA. 2 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
INTERVAL NO.	TYPE	RECOVERY N	IN	MOISTURE	DEPTH	
1	GP		30	M		Concrete.
						FILL: poorly-graded sand, fine, yellow brown, no odor, moist.
2	GP		32	M	5	SANDY LEAN CLAY (CL), some fine to coarse sand, few percent fine gravel, slightly plastic, mottled mostly brown, dark yellowish brown, with gray along abundant fractures, no odor, moist, hard, PID = 0 (Glacial Till).
3	GP		48	M		As above (CL), dark precipitate along fractures.
						<-- Abrupt color change at 9.2 feet, PID = 0.
					10	As above (CL), mostly gray, yellowish brown along occasional fractures, PID = 0.
						End of boring at 12 feet.

GENERAL NOTES

DATE STARTED 25 APR 97
DATE COMPLETED 25 APR 97
RIG GEOPROBE
CREW CHIEF K. BELL
LOGGED DPR CHECKED LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING ▽ none observed
AT COMPLETION ▼
AFTER DRILLING
CAVE-IN: DATE/TIME DEPTH
WATER: DATE/TIME DEPTH

LOG OF TEST BORING

F-203 (R 01-87)

PROJECT NAME: PEREGRINE FLINT
LOCATION: FLINT, MICHIGAN
CONTRACTOR: FIBERTEC
DRILLING METHOD: DIRECT PUSH

BORING NO.: PFB-27
SHEET NO.: 1 OF 1
PROJECT NO.: 4036.05
INSTALLATION: 4-25-97
SURFACE ELEV.:
BOREHOLE DIA.: 2 IN.

SAMPLING NOTES						VISUAL CLASSIFICATION AND GENERAL OBSERVATIONS
INTERVAL NO.	INTERVAL TYPE	RECOVERY N	RECOVERY IN	MOISTURE	DEPTH	
1	GP		28	M		Concrete.
						FILL: poorly-graded sand, fine, yellow brown, no odor, moist.
2	GP		10	M		As above.
					5	End of boring at 5 feet. Encountered obstruction. Moved 3 feet to the N/NE; hit (same?) obstruction at approximately 5 feet. Did not attempt to redrill within area of pillars G-H/37-38.
					10	

GENERAL NOTES

DATE STARTED: 25 APR 97
DATE COMPLETED: 25 APR 97
RIG: GEOPROBE
CREW CHIEF: K. BELL
LOGGED: DPR CHECKED: LPL

WATER LEVEL OBSERVATIONS

WHILE DRILLING: none observed
AT COMPLETION:
AFTER DRILLING
CAVE-IN: DATE/TIME _____ DEPTH _____
WATER: DATE/TIME _____ DEPTH _____

APPENDIX B

PROJECT MANAGEMENT PLAN

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LIST OF ATTACHMENTS

ATTACHMENT B-1 CURRICULA VITAE

1.0 INTRODUCTION

This Project Management Plan (PMP) is submitted as an appendix to and forms part of the RCRA Facility Investigation (RFI) Work Plan prepared by Remediation and Liability Management Company Inc. (REALM) for the Former Peregrine (US), Inc. Coldwater Road Facility (Facility).

The PMP presents a discussion of the technical approach, schedules, and personnel for performing the RFI.

2.0 TECHNICAL APPROACH

The overall approach that will be used for the RFI will be to focus on the data that is necessary to complete the RFI Report and to allow for the evaluation of corrective measures (if required). As discussed in the Data Management Plan (DMP), Appendix D, Mr. Glenn Turchan will be responsible for all aspects of data management. The goal of completing the RFI Report will be achieved by implementing the approved RFI Work Plan.

3.0 SCHEDULES

3.1 SCHEDULES

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.

4.0 PROJECT MANAGEMENT ORGANIZATION

Figure B.4.1 presents REALM's overall project management organization for the RFI.

The Project Manager for MDEQ is:

Mr. Suresh Thomas
Michigan Department of Environmental Quality
Waste Management Division (MDEQ-WMD)
P.O. Box 30241
John C. Hannah Building
Lansing, Michigan 48909-7741
Phone: (517) 373-2487

The Project Manager for REALM is:

Ms. Cheryl Hiatt
General Motors Corporation
Worldwide Facilities Group
Environmental & Regulatory Support
Remediation Team
485 West Milwaukee Avenue
Mail Code: 482-310-004
Detroit, Michigan 48202-3220
Phone: (313) 556-0821
Fax: (313) 556-0803

All communications between MDEQ-WMD and REALM, and all documents, plans, reports, approvals and other correspondence concerning activities performed during the RFI, will be directed through Ms. Cheryl Hiatt and Mr. Suresh Thomas.

Conestoga-Rovers & Associates (CRA), 11100 Metro Airport Center Drive, Suite 160, Romulus, Michigan, 48174, will be REALM's environmental consultant for the RFI. CRA's Project Manager is Mr. Glenn Turchan. CRA's Project Coordinator and Project Engineer is Mr. Randy Moore. CRA's Quality Assurance (QA) Officer is Mr. Paul Wiseman. CRA's Health and Safety Officer is Ms. Karen Sud. CRA will supervise all field activities of the RFI and assure that they are performed in accordance with the RFI Work Plan. CRA will compile and assess all data collected as part of the RFI and will prepare the RFI Report.

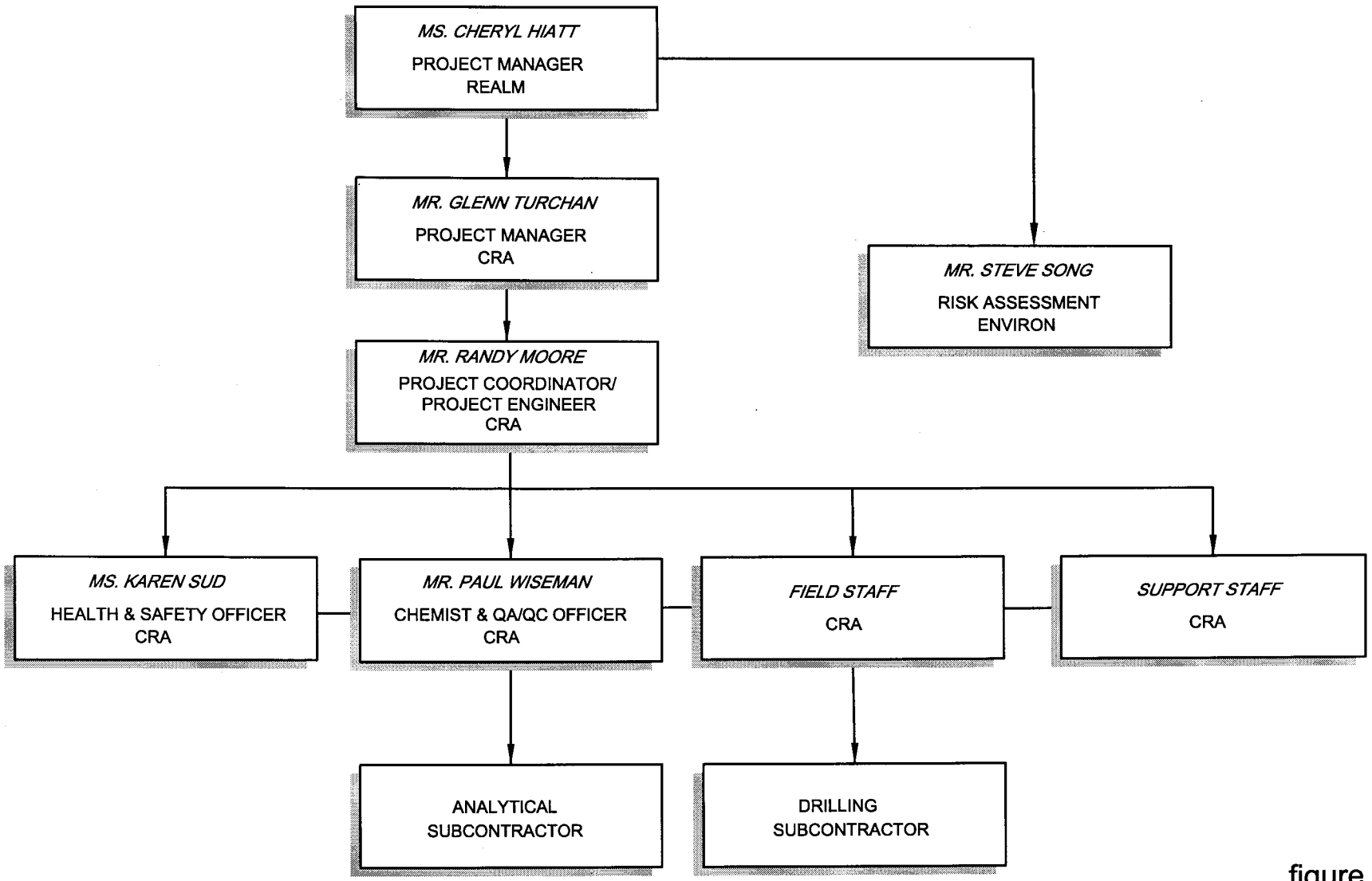

figure B.4.1

PROJECT MANAGEMENT ORGANIZATION
FORMER PEREGRINE (US) INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA005 MAY 15/2000

Environ (Environ) will be REALM's risk assessment consultant for the RFI. Mr. Steve Song is Environ's Risk Assessment Manager. Environ will perform human health and ecological risk assessments based on the RFI data (as required).

Severn Trent Laboratories, Inc. (Severn Trent) will be the analytical subcontractor. Severn Trent will perform all chemical analyses for samples collected as part of the RFI.

The drilling subcontractors will be determined following competitive bidding protocols. The firms selected by REALM for the analytical and drilling activities will be identified to MDEQ-WMD prior to commencement of field activities.

All firms will provide project management as appropriate to their responsibilities. CRA will provide subcontractor administrative oversight.

5.0 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.

5.1 KEY PERSONNEL

As identified in Section 4.0, CRA 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, and curricula vitae are included in Attachment B-1.

5.1.1 PROJECT MANAGEMENT

Mr. Glenn Turchan: Mr. Turchan will act as CRA's Project Manager. He will oversee all aspects of the project, participate in technical meetings with the MDEQ-WMD, and will be actively involved in the direction of the project. Mr. Turchan has extensive experience in projects of this nature. He will ensure that technical quality and scheduling are maintained throughout all activities. The CRA Project Coordinator will brief him regularly regarding all activities.

Mr. Randy Moore: Mr. Moore will act as CRA's Project Coordinator. He will ensure that all day-to-day tasks are conducted in a professional and timely manner. Mr. Moore will keep the CRA 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.

5.1.2 TECHNICAL RESOURCE PERSONNEL

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

Project Engineer: Mr. Randy Moore, an Engineer with CRA, will review and evaluate data pertaining to the RFI.

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: Ms. Karen Sud will be CRA's Site Health and Safety Officer. Ms. Sud will supervise the CRA Site Representative (responsibilities defined in Health and Safety Plan, Appendix E), 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.

6.0 ENVIRON QUALIFICATIONS

Environ is fully qualified to perform human health and ecological risk assessments in accordance with U.S. EPA's Risk Assessment Guidance for Superfund (RAGS).

Environ will provide a qualifications package, if requested by MDEQ.

7.0 LABORATORY QUALIFICATIONS

Severn Trent is fully qualified to conduct analyses in accordance with U.S. EPA/MDEQ analytical methods specified in the QAPP which is presented as Appendix C of the RFI Work Plan.

Severn Trent will provide a qualifications package, if requested by MDEQ.

8.0 DRILLING SUBCONTRACTOR QUALIFICATIONS

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

1. Satisfy applicable 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 by MDEQ.

9.0 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 MDEQ-WMD, as requested.

ATTACHMENT B-1

CURRICULA VITAE

GLENN T. TURCHAN, M.A.Sc., P. Eng.

EDUCATION

B.A.Sc.	University of Waterloo, Geological Engineering
M.A.Sc.	University of Waterloo, Civil Engineering
Other Courses:	Organic Contaminants in Groundwater, June 1988 (sponsored by MOE and WCGR)
	40-Hour OSHA Personal Protection and Safety Training (as per CFR 1910-120), September 1988 (hazardous waste site safety training)
	8-Hour OSHA Site Supervisors and Managers Course Training (as per 29 CFR 1910-120), March 1989 (hazardous waste site managers health and safety training)
	8-Hour Refresher in Personal Protection and Safety (as per 29 CFR 1910-120) (yearly update as required)

EMPLOYMENT

1995-Present	Project Manager/Principal/Principal-In-Charge (Detroit Office) Conestoga-Rovers & Associates
1994-95	Associate, Project Manager/Coordinator, Conestoga-Rovers & Associates
1989-93	Project Manager/Coordinator, Conestoga-Rovers & Associates
1987-89	Project Engineer, Conestoga-Rovers & Associates
1985-86	Engineering Assistant, Conestoga-Rovers & Associates
1984	Engineering Assistant, Atomic Energy of Canada Limited
1984	Engineering Assistant, Acres Consulting Services Limited

AFFILIATIONS

Professional Engineers Ontario (PEO #90212960)

PROFILE OF PROFESSIONAL ACTIVITIES

- Professional Engineer/Hydrogeologist
- Project Engineer/Coordinator or Project Manager responsible for environmental due diligence/environmental site assessments/environmental compliance audits/investigations/site characterizations/remedial investigations (RI)/feasibility studies (FS)/remedial design (RD)/remedial action (RA)/LUST closures/permitting/contract documents/supervision and contractor oversight
- Remedial Investigation/Feasibility Studies (RI/FS) at the following sites:
  - Willow Run Sludge Lagoon, Washtenaw County, Michigan
  - Willow Run Creek Site, Washtenaw County, Michigan
  - WDE Sanitary Landfill, Andover, Minnesota

- Nepera Inc. Plant Site, Harriman, New York
- Nepera Inc. Former Lagoon NPL Site, Maybrook, New York
- Fons Landfill Site, Washtenaw County, Michigan
- Old Wayne Landfill Site, Washtenaw County, Michigan
- Textile Road Site, Rawsonville, Michigan
- RCRA RFI/CMS at GM Pontiac facility, Pontiac, Michigan
- Metamora Landfill Site, Lapeer County, Michigan
- Highway 48 Site, Stouffville, Ontario
- Sterling Drug Site, Cincinnati, Ohio
- National Airport Site, Westland, Michigan

- Overview and Critique of U. S. EPA or State Lead RI/FS or RD/RA programs at the following Superfund sites:
  - Pristine Site, Cincinnati, Ohio
  - G & H Landfill Site, Utica, Michigan
  - Metamora Landfill Site, Lapeer County, Michigan
  - MIG/Dewane Landfill Site, Belvidere, Illinois
  - Michigan Chrome and Chemical, Detroit, Michigan
- Remedial Design/Remedial Action (RD/RA) at the following sites:
  - Metamora Landfill Site, Lapeer County, Michigan
  - G&H Landfill Site, Utica, Michigan
  - Willow Run Creek Site, Washtenaw County, Michigan
  - Fons Landfill Site, Washtenaw County, Michigan
  - Old Wayne Landfill Site, Washtenaw County, Michigan
  - SMDA Sites 9/9A Macomb Township, Michigan
  - Pontiac Sites (various), Pontiac, Michigan
  - Meritor Automotive Site, Allegan, Michigan
  - Middleground Landfill, Bay City, Michigan
- Environmental Site Assessments/Environmental Compliance Audits
  - Completed over 250 ESA and ECA projects in Canada and the United States involving industrial and commercial facilities
- Acquisition/Divestiture Due Diligence
  - Environmental due diligence support related to purchase of five commercial laundry plants
  - Environmental due diligence support related to acquisition of 14 gravel pit operations
  - Environmental due diligence support on purchase of 2 radio stations
  - Environmental due diligence support in divestiture of a shopping mall located in Bloomfield Township
  - Environmental due diligence support to GM related to various properties at the Centerpoint Business Campus and Clark Street Technology Park

- Environmental due diligence support to GM in divestiture of two Delphi plants in Livonia and Flint to Peregrine Inc.
- Environmental due diligence support related to purchase of 11 cemeteries
- Environmental due diligence support to GM in divestiture of five plants in Grand Rapids, Auburn Hills and Warren to Lear Corporation
- Environmental due diligence support to GM in divestiture of three Delphi plants in Oshawa, St. Catherine's and New Brunswick, New Jersey
- Environmental due diligence support to Oxford Automotive in acquisition of two plants in Ohio and two plants in Michigan from Howell Industries Inc.
- Environmental due diligence support to Oxford Automotive in acquisition of one plant in Indiana and two plants in Ontario from Eaton-Yale
- Environmental due diligence support to Delphi Automotive Systems in leasing facilities in Rochester Hills, Michigan and Duncan, South Carolina
- Environmental due diligence support to Ferrous Processing and Trading in acquisition of scrap yard facilities in Windsor, Ontario (3), Detroit (2), Warren, and Flint
- Environmental due diligence support to Trident Properties on acquisitions of properties throughout Michigan

- Baseline Environmental Assessments
  - Northline L.L.C., Romulus, Michigan
  - Equipment Supply, Romulus, Michigan
  - Schlafer Iron and Steel Company, Detroit Michigan
  - TBS Industrial Recycling, Inc., Pontiac, Michigan
  - Mason Iron and Metal, Detroit, Michigan
  - Grand Traverse Overall Supply, NPL Site, Traverse City, Michigan
  - Ferrous Processing Inc., Detroit, Michigan
  - Former New Holland Site, Troy, Michigan
  - St. Marys Cement Plant, Detroit, Michigan
- Brownfield Redevelopment Projects
  - Clark Street Technology Park, Detroit, Michigan
  - Centerpoint Business Campus, Pontiac, Michigan
  - Northline L.L.C., Romulus, Michigan
  - Former New Holland Site, Troy, Michigan
  - Textile Road Site, Ypsilanti Township, Michigan
- Environmental Decommissioning
  - Removal action at Chrysan Industries in Detroit, Michigan
  - Support to GM in development of standard guidelines and specifications for plant decommissioning
  - Building decommissioning assessments at the following sites:
    - GM Brea, California
    - GM Moraine facility, Moraine, Ohio

  - Oldsmobile Site, Lansing, Michigan
    - 500,000 square feet
  - Oldsmobile Site, Lansing, Michigan
    - 1,000,000 square feet
  - GM Desert Proving Grounds, Mesa, Arizona
    - 500,000 square feet on 7500 acres
  - GM Metal Fabrication Division, Kalamazoo, Michigan
    - 1,500,000 square feet on 350 acres
  - Delco Electronics, Kokoma, Indiana
    - 750,000 square feet
  - GM Metal Fabrication Buildings 2, 5, 12, Pontiac, Michigan
    - 750,000 square feet
  - Delphi, Rochester, New York
    - 350,000 square feet
- Risk Assessment Evaluations
- Landfill Gas Management/Recovery/Utilization Projects
  - Brock West Site, Pickering, Ontario
  - Keele Valley Site, Maple, Ontario
- Supervision of drilling and groundwater monitoring well installation
- Preparation and representation of technical information to representatives of State and Federal Governments
- Litigation Support
- Support to various clients on a variety of litigation matters

PUBLICATIONS

- "Landfill Gas Collection and Utilization", paper presented at *Canadian Geotechnical Society, Southern Ontario Section, Solid Waste Symposium*. Toronto, Ontario, March 9, 1988 (with A.J. Crutcher, A.W. Van Norman)
- "Overview on Problems of Pond Construction", paper presented at *Prevention & Treatment of Groundwater and Soil Contamination in Petroleum Exploration and Production* Calgary, Alberta, May 10, 1989 (with D.H. Haycock, S.T. Ruminsky)
- "Soil Remediation Issues", presented at *Third Annual National Meeting and Conference of the Academy of Certified Hazardous Materials Managers*, July 25 - 28, 1989, Indianapolis, Indiana (with A.W. Van Norman)
- "Groundwater Cutoff Walls: Application at Hazardous Waste Sites", paper presented at *Focus Conference on Eastern Regional Groundwater Issues*, National Water Well Associations, Kitchener, Ontario, October 18, 1989 (with A.W. Van Norman, I.K. Richardson)
- "Remediation Approaches and Technologies for Potential Application to PCB Contaminated Sludge Sites", paper presented at *SUPERFUND XIV*, December 1993, Washington, D.C., (with J. Daniel, E. Peterson, K. Waskiewicz, A.W. Van Norman)

- Carey, G.R., M.G. Mateyk, G.T. Turchan, E.A. McBean, and J.R. Murphy, 1996, "Intrinsic Remediation as an Effective Alternative to Groundwater Pump-and-Treat: A Case Study", submitted for publication in NWWA/API Conference on Proceedings of Petroleum Hydrocarbons & Organic Chemicals in Ground Water, Houston, Texas.
- Carey, G.R., M.G. Mateyk, E.A. McBean, G.T. Turchan, and F.A. Rovers, 1996, "Application of a Technical Protocol for Evaluating Intrinsic Remediation at Landfill Sites", submitted for publication in Proceedings of the Madison Waste Conference, Madison, Wisconsin.
- Carey, G., M. Mateyk, E. McBean, G. Turchan, J. Campbell and F. Rovers (1996): "Multiple Lines of Evidence for Evaluating Intrinsic Remediation at a Landfill Site", presented at the 19th Annual Madison Waste Conference, Madison, Wisconsin, September 1996
- Carey, G., Michael Mateyk, Glenn Turchan, Edward McBean, Frank Rovers and Richard Murphy (1996): "Application of an Innovative Visualization Method for Demonstrating Intrinsic Remediation at a Landfill Site", presented at the 1996 Petroleum Hydrocarbons Exposition, Houston, Texas, November 1996
- "Modelling Landfill Cap Influence on Natural Attenuation", presented at Sixth International Landfill Symposium, Cagliari, Sardinia, Italy, October 13-17, 1997 (with G. Carey, P. Van Geel, E. McBean, G. Turchan)
- "Site Reuse/Brownfield Redevelopment in the Detroit Empowerment Zone" technical paper presented (by K. West) at Superfund XVIII Conference, Washington, D.C., December 4, 1997 (co-authors(K. West, B. Pitlock, K. Richards, G. Turchan, S. Anderson)

Reports:

- "Groundwater Cutoff Walls: Applicability, Design and Implementation at Hazardous Waste Sites", *in partial fulfillment of the requirements for the degree of M.A.Sc.*, Department of Civil Engineering, University of Waterloo, March 1, 1990

Presentations:

- "Environmental Engineering in the 90's", presentation at Professional Engineers Ontario - Undergraduate Engineering Students Conference 1994, January 28, 1994 (with J. Daniel, G. O'Neill)
- "Center Point Business Campus Redevelopment", presentation at Industrial Site Recycling Conference – Phoenix Award Finalist, Pittsburgh, Pennsylvania, April 14, 1998 (presented by K. West)

W. RANDY MOORE, P. Eng.

EDUCATION

B.A.Sc.	Civil Engineering (with options in Water Resources and Management Science), May 1989, University of Waterloo, Waterloo, Ontario
B.Sc.	(Honours) Biology, May 1981, University of Waterloo, Waterloo, Ontario
Other Courses	Annual 8-Hour Refresher Program for Hazardous Waste Work, 1990 to 1996
	40-Hour General Site Worker Program (in accordance with 29 CFR 1910.120), July 1989, University of Illinois, Urbana - Champaign, Illinois
	Graduate courses in the M.Sc. Biology program, January 1983, University of Waterloo, Waterloo, Ontario

EMPLOYMENT

1992-Present	Project Coordinator/Project Engineer Conestoga-Rovers & Associates, Waterloo, Ontario
1989-91	Project Engineer, Conestoga-Rovers & Associates, Waterloo, Ontario
1988	Environmental Engineering (work terms), Conestoga-Rovers & Associates, Waterloo, Ontario

AFFILIATIONS

Professional Engineers of Ontario

PROFILE OF PROFESSIONAL ACTIVITIES

- Project Coordinator for a Remedial Investigation/Feasibility Study at a 30-acre former lagoon Superfund site located in Orange County, New York
- Project Coordinator for a Phase II Environmental Site Assessment at a 31-acre former fiberglass reinforced plastics manufacturing facility located in Cornwall, Ontario
- Project Coordinator for a Phase II Environmental Site Assessment at an urea and melamine-based resins manufacturing facility located in Toledo, Ohio
- Project Coordinator for a Remedial Investigation/Feasibility Study including preparation of a Remedial Pre-Design Work Plan at a 24-acre inactive waste disposal site in Cheektowaga, New York
- Project Coordinator for an environmental monitoring program, leachate migration study, gas migration study and environmental monitoring system upgrades at a 320-acre closed landfill site in Stouffville, Ontario
- Project Coordinator for a Remedial Investigation/Feasibility Study at a closed 5-acre municipal landfill site located in Tecumseth, Ontario
- Project Coordinator for an Additional Site Characterization Program at a pyrotechnic, propellant and explosives manufacturing and research facility located in Milpitas, California

- Project Coordinator for the evaluation of a Gas Control System at a closed municipal landfill site located in Elmira, Ontario
- Project Engineer/Report Coordinator for a Remedial Investigation/Feasibility Study at a 50-acre municipal landfill Superfund site located in Niagara County, New York
- Project Engineer/Report Coordinator for a Remedial Investigation/Feasibility Study at a synthetic coating manufacturing facility located in Schenectady, New York
- Project Engineer/Report Coordinator for a Feasibility Study at a former electrical products manufacturing facility located in Wyoming, Illinois
- Project Engineer/Site Coordinator for a Remedial Investigation/Feasibility Study at a former 3,000 drum disposal site in Chenango County, New York
- Project Engineer/Site Coordinator for a Remedial Investigation at a former 7-acre paint-related waste disposal site in Erie County, New York
- Project Engineer/Site Coordinator for a Remedial Investigation/Feasibility Study at a former 900 drum disposal site in Staten Island, New York
- Project Engineer/Site Coordinator for a Remedial Investigation/Feasibility Study including preparation of a Remedial Pre-Design Work Plan at a 21-acre former chemical plant in Lewiston, New York
- Project Engineer/Site Coordinator for a Remedial Investigation at a former 37-acre copper smelting and refining facility on Long Island, New York
- Project Engineer for a drum delineation and removal program at a former disposal site in Syracuse, New York
- Project Engineer for a Subsurface Soil and Groundwater Investigation at a gas station located in London, Ontario

PAUL WISEMAN, B.S.

EDUCATION

B.S. Chemistry and Biology, Northern Michigan University, Marquette, Michigan, 1980

EMPLOYMENT

1994 - Present	Environmental Chemist Conestoga-Rovers & Associates
1993-94	Laboratory Director, Great Lakes Environmental Laboratories
1992	Director - Laboratory Services, DiHydro Analytical Services
1990-92	Laboratory Manager, Core Laboratories
1985-90	Laboratory Manager, Environmental Testing and Certification Corp.
1983-85	Laboratory Coordinator, O.H. Materials Corporation
1981-83	Project Chemist, O.H. Materials Corporation
1980-81	Chemist, O.H. Materials Corporation

AFFILIATIONS

American Chemical Society

Association of Official Analytical Chemists

Michigan Environmental Laboratory Association

PROFILE OF PROFESSIONAL ACTIVITIES

- Responsibility for the development and management of several environmental testing laboratories providing field and analytical services in support of groundwater monitoring and assessments; subsurface soil investigations and excavations; tank closures and removals; waste characterization and disposal; and wastewater treatment and discharge
- Development and establishment of laboratory quality assurance/quality control programs
- Designed, supervised construction and developed an environmental testing laboratory
- Participation in and completion of U.S. Army Corp. of Engineers laboratory validation process and various state certifications
- Qualified as an expert witness regarding a Superfund drum Site in Cantonment, Florida
- Responsibility for the management of a petroleum testing laboratory performing various ASTM and API tests assessing petroleum product quality
- Laboratory chemist completing organic and inorganic analyses on environmental samples using a variety of analytical instrumentation and methods
- Coordination of on-site laboratory activities providing analysis in support of groundwater investigation, monitoring and treatment; building decontaminations; lagoon closures; and drum excavation, removal and disposal projects

- Supervision of and participation in all aspects of drum handling procedures including: field testing and monitoring of containerized waste; compatibility testing; laboratory test bulking; field consolidation monitoring; labeling and manifesting; lab-pack chemical handling procedures
- On-site chemist for numerous Superfund and private-sector projects performing sampling and analyses, overseeing cleanup/treatment of spilled hazardous materials and operating water treatment systems
- Validation and assessment of polychlorinated dibenzo-p-dioxin and polychlorinated dibenzofuran analytical data using high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS) methods
- Validation and assessment of U.S. EPA CLP, SW-846 and non-standard analytical data
- QA/QC officer for numerous Superfund and RCRA investigation and remedial action projects
- Development of Superfund and RCRA quality assurance project plans (QAPPs)
- Review of data generated from sampling and analysis programs to evaluate its usability for RI/FS and RD/RA purposes
- Analytical data quality assessment and liaison work with project laboratories in support of various investigative and remedial projects
- Development of sampling and analysis plans for numerous Superfund and private sector projects
- Development of sampling and analysis plans for numerous facility building decommissioning assessments

KAREN E. SUD, M.S.

EDUCATION

M.S	McGill University, Occupational Health, 1997
B.S	Ryerson Polytechnic University, Environmental Health, 1996
Other Courses:	40-Hour OSHA Health and Safety Training, 1995
	8-Hour Standard First Aid and Adult CPR Training, 1999

EMPLOYMENT

1999-Present	Industrial Hygienist Conestoga-Rovers & Associates; Romulus, Michigan
Sept. 98-Sept. 99	Health and Safety Specialist, EnSafe ; Memphis, Tennessee
Sept. 97	Noise Analyst Specialist
Aug. 98	James Anderson and Associates; Lansing, Michigan

AFFILIATIONS

Member, American Society for Safety Engineers

Member, American Industrial Hygiene Association

Member, American Public Health Association

PROFILE OF PROFESSIONAL ACTIVITIES

Industrial Hygienist at Conestoga-Rovers & Associates Romulus, Michigan Office

Industrial Hygiene

- Conducted Noise Exposure Assessments for various industrial sites across North America
- Determined how chemicals would be stored for an industrial client to meet NFPA and local fire code requirements
- Conducted Chemical Inventories at industrial sites
- Have investigated complaints related to chemical exposures
- Have conducted indoor air quality assessments of industrial buildings
- Have performed qualitative and quantitative chemical exposure assessments for various clients
- Have performed lighting surveys

Health and Safety

- Prepared project-specific health and safety plans
- Have acted as a health and safety officer at various sites

- Prepared and delivered OSHA training courses
- Have written OSHA compliance programs for industrial clients

APPENDIX C

QUALITY ASSURANCE PROJECT PLAN (QAPP) FOR THE RCRA FACILITY INVESTIGATION (RFI) AT FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY GENESEE TOWNSHIP, MICHIGAN

U.S. EPA ID NUMBER MIR 000020743

REVISION NUMBER 0

APRIL 28, 2000

QUALITY ASSURANCE PROJECT PLAN (QAPP)

FOR THE RCRA FACILITY INVESTIGATION (RFI)
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

U.S. EPA ID NUMBER – MIR 000020743
REVISION NUMBER 0
MAY 15, 2000

Prepared By: Conestoga-Rovers & Associates (CRA)

Prepared For: Remediation and Environmental Liability Management Company, Inc. (REALM)

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Opal Johnson

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QAPP DISTRIBUTION LIST

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1.0 PROJECT DESCRIPTION

The following outlines the overall scope of a RCRA Facility Investigation (RFI) to be performed in accordance with the requirements of the permit/consent reference issued to Remediation and Liability Management Company, Inc. (REALM) for the Former Peregrine (US), Inc. Coldwater Road Facility Genessee County, Michigan. 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 this Facility. Specific protocols for sampling, sample handling and storage, chain-of-custody and laboratory and field analyses are described or specifically referenced to related RFI documents. All QA/QC procedures are structured in accordance with applicable technical standards, United States Environmental Protection Agency (U. S. EPA) requirements, regulations, guidance and technical standards. This QAPP has been prepared in accordance with the U.S. EPA Region 5 QAPP policy as presented in "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.

1.1 INTRODUCTION

This QAPP has been prepared on behalf of REALM by Conestoga-Rovers and Associates (CRA). A Project Management Plan, this QAPP, a Data Management Plan, a Health and Safety Plan, and a Community Relations Plan have been appended to the RFI Work Plan. A Field Sampling Plan (FSP) has also been prepared and appended to the RFI Work Plan and has been entirely incorporated into the QAPP through specific reference.

1.1.1 OVERALL PROJECT OBJECTIVES AND DECISION STATEMENT

One purpose of this RFI is to gather sufficient information to quantify risk in the event that environmental contamination is determined to be present. The objectives of the RFI are to determine the nature and extent of contamination at or migrating off-Site from the facility.

Overall objectives of the data collection will be as follows:

- Verify and further define the nature and extent of contamination in previously identified on-site and off-site areas (soil, groundwater, sediment and surface water matrices). Data quality must be sufficient to allow comparison with established action levels or regulatory standards (screening levels).
- Determine the nature and extent of contamination in previously uninvestigated areas. Laboratory data will eventually be compared to established human health and ecological target decision levels. The target decision levels for all project parameters are summarized in the Work Plan.
- Collect sufficient data for all contaminated media and/or biota to support a human health baseline risk assessment, preliminary ecological risk assessment, and feasibility study for corrective measures.

The Decision Statement for this investigation is as follows: What is the nature, risk and extent of identified contamination in onsite or offsite soil, sediment, surface water and groundwater media that presents unacceptable risks, which would therefore warrant remedial action?

Associated specific objectives for field and laboratory data collection are tabulated in Section 1.4 of this QAPP.

1.1.2 PROJECT STATUS/PHASE

CRA will utilize an integrated and phased approach for the RFI. During the RFI, data collection will be conducted in phases, with the results of the initial sampling event that exceed risk based or generic criteria used as determining factors in decisions regarding the necessity for additional phases of investigation.

1.1.3 QAPP PREPARATION GUIDELINES

As previously described, this QAPP has been prepared in accordance with the "U.S. EPA Region 5 RCRA QAPP Instructions" dated April 1998.

1.2 SITE/FACILITY DESCRIPTION

This section is addressed in the RFI Work Plan, which is herein incorporated into this QAPP through reference, and in the drawings which have been submitted along with the RFI Work Plan.

1.3 SITE/FACILITY HISTORY

This section is addressed in the RFI Work Plan, which is herein incorporated into this QAPP through reference, and the drawings and tables which have been submitted along with the RFI Work Plan.

1.4 PROJECT OBJECTIVES AND INTENDED DATA USAGES

For the RFI, it will be necessary to gather sufficient information to evaluate the nature and extent of releases from 12 Areas of Interest (AOI) and also to determine whether unreasonable health risks are associated with actual or potential receptors within and beyond the facility property boundaries.

The specific objectives of the data collection at the Facility follow:

Field monitoring will be utilized to determine appropriate sample points, to determine when monitoring wells have stabilized prior to sampling and for health and safety purposes. These types of data include those generated on-site through the use of photoionization, pH/temperature and specific conductance real-time monitoring equipment at the site. The field data requirements are summarized in Section 1.4.1.1.

Laboratory data will be compared to the specific criteria or action levels defined in the RFI Work Plan for determining unreasonable health risks. The laboratory parameters are summarized in Section 1.4.1.2.

1.4.1 PROJECT TARGET PARAMETERS

The following sections present the field and laboratory parameters. The sample matrices, field and laboratory parameters and frequencies of quality control sample collection are also summarized in Table 1.1.

TABLE C1.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

AOI Number	*AOI Description*	*Sample Matrix*	*Field Parameters*	*Laboratory Parameters*	*Investigative Samples* [7]	*Quality Control Samples* [1]			*Total*	
						Field Blanks [2]	*Field Duplicates*	*MS/MSD MS/DUP* [3]		
AOI 2	Former Waste Pile Pad	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Inorganics & Hexavalent Chromium	6	1/10	1/10	1/20	9	[4]
		Groundwater	pH, Temperature & Specific Conductance	TCL VOC	1	1/10	1/10	1/20	4	[4]
AOI 8	Former Scrap Metal Management System	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Inorganics & Hexavalent Chromium	10	1/10	1/10	1/20	9	[4]
AOI 9	Former Plating Areas	Soil	PID VOC Screen	TCL VOC, TAL Inorganics & Hexavalent Chromium	58	1/10	1/10	1/20	73	[4]
				Modified Appendix IX [5]	2	--	--	--	2	[4]
AOI 11	Former Coal Storage Area	Soil	PID VOC Screen	TCL SVOC, TCL PCB, TAL Inorganics & Hexavalent Chromium	10	1/10	1/10	1/20	13	[4]
AOI 12	Former Caustic Tanks	Soil	PID VOC Screen	TCL VOC, TAL Inorganics, pH & Hexavalent Chromium	4	1/10	1/10	1/20	7	[4]
AOI 13	Former Headliner Adhesive Tanks	Soil	PID VOC Screen	TCL VOC	4	1/10	1/10	1/20	7	[4]

TABLE C1.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

AOI Number	*AOI Description*	*Sample Matrix*	*Field Parameters*	*Laboratory Parameters*	*Investigative Samples* [7]	*Quality Control Samples* [1]			*Total*	
						Field Blanks [2]	*Field Duplicates*	*MS/MSD MS/DUP* [3]		
AOI 14	Former Incinerator	Soil	PID VOC Screen	TCL SVOC, TCL PCB, TAL Inorganics & Hexavalent Chromium	4	1/10	1/10	1/20	7	[4]
AOI 17	Substation Drainage Sump	Soil	PID VOC Screen	TCL SVOC & TCL PCB	4	1/10	1/10	1/20	7	[4]
AOI 18	Former IERY	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Inorganics & Hexavalent Chromium	8	1/10	1/10	1/20	11	[4]
		Groundwater	pH, Temperature & Specific Conductance	Lead	1 [6]	1/10	1/10	1/20	4	[4]
AOI 19	Former Press Yard	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Inorganics & Hexavalent Chromium	15	1/10	1/10	1/20	18	[4]
				Modified Appendix IX [5]	1	--	--	--	1	[4]
AOI 20	Former Tool Room	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TCL PCB, TAL Inorganics & Hexavalent Chromium	7	1/10	1/10	1/20	10	[4]
				Modified Appendix IX [5]	1	--	--	--	1	[4]

TABLE C1.1

SUMMARY OF SAMPLING AND ANALYSIS PROGRAM
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

AOI Number	*AOI Description*	*Sample Matrix*	*Field Parameters*	*Laboratory Parameters*	*Investigative Samples* [7]	*Quality Control Samples* [1] *Field Blanks* [2]	*Field Duplicates*	*MS/MSD MS/DUP* [3]	*Total*
AOI 21	Power Fuel Oil Storage Tanks	Soil	PID VOC Screen	TCL VOC, TCL SVOC, TAL Inorganics & Hexavalent Chromium	4	1/10	1/10	1/20	7 [4]
Other	Non-specific	Water	pH, Temperature & Specific Conductance	TCL VOC	1	1/10	1/10	1/20	4 [4]
				Lead	3 [6]	1/10	1/10	1/20	6 [4]
--	Background	Soil	PID VOC Screen	TCL VOC, TAL Inorganics & Hexavalent Chromium	8	1/10	1/10	1/20	11 [4]

Notes:

[1] - A laboratory trip blank will be submitted with each group of multiple aqueous sampleS for TCL VOC analysis.

[2] - Field blank samples will not be required if dedicated or disposable sampling equipment is used.

[3] - Matrix Spike/Matrix Spike Duplicate (MS/MSD) and MS/Matrix Duplicate (MS/DUP) analyses are required for samples submitted for organic and inorganic analyses respectively are to be analyzed at a frequency of one per group of twenty (20) or fewer investigative samples.

[4] - The frequency of field blanks and field duplicates will be a minimum of one per ten investigative samples from combined AOIs. The total will vary depending on which AOI the QC samples are collected from.

[5] - Modified Appendix IX parameters include Appendix IX VOC, SVOC, PCB and Inorganics.

[6] - Analyzed lead will be determined as filtered/dissolved.

[7] - The number of investigative samples represents a maximum of two soil samples to be collected from each borehole. The first sample will be collected at the 0 to 2 foot bgs horizon. The second sample will be collected at the interval exhibiting the greatest degree of staining or positive response on the PID. If no staining or PID response is encountered, the lower soil sample may not be collected.

TCL = Target Compound List
SVOC = Semi-volatile Organic Compounds
TAL = Target Analyte List
VOC = Volatile Organic Compounds
PCB = Polychlorinated Biphenyls
AOI = Area of Interest

TABLE C1.2

TCL/TAL PARAMETER LIST
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Compound	*Targeted [1] Quantitation Limits*	
	Water (µg/L)	*Soil (µg/kg)*
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	2	10
1,1-Dichloroethane	1	5
1,2-Dichloroethane	1	5
1,1-Dichloroethene	1	5
cis-1,2-Dichloroethene	0.5	5
trans-1,2-Dichloroethene	0.5	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
Styrene	1	5
1,1,2,2-Tetrachloroethane	1	5
Tetrachloroethene	1	5
Toluene	1	5

TABLE C1.2

TCL/TAL PARAMETER LIST
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Compound	*Targeted [1] Quantitation Limits*	
	Water (µg/L)	*Soil (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	10
TCL Semi-Volatile Organic Compounds (SVOC)		
Acenaphthene	5	330
Acenaphthylene	5	330
Acetophenone	10	330
Anthracene	5	330
Atrazine	5	330
Benzaldehyde	10	330
Benzo(a)anthracene	5	330
Benzo(a)pyrene	5	330
Benzo(b)fluoranthene	5	330
Benzo(g,h,i)perylene	5	330
Benzo(k)fluoranthene	5	330
1,1'-Biphenyl	10	330
4-Bromophenylphenyl ether	5	330
Butylbenzylphthalate	5	330
di-n-Butyphthalate	5	330
Caprolactam	10	330
Carbazole	10	330
4-Chloroaniline	10	1,300
bis(2-Chloroethoxy)methane	5	330
bis(2-Chloroethyl)ether	5	330
bis(2-Chloroisopropyl)ether	5	330
4-Chloro-3-methylphenol	5	330
2-Chloronaphthalene	5	330
2-Chlorophenol	5	330
4-Chlorophenyl phenyl ether	5	330
Chrysene	5	330
Dibenz(a,h)anthracene	5	330
Dibenzofuran	5	330
3,3'-Dichlorobenzidine	20	2,000

TABLE C1.2

TCL/TAL PARAMETER LIST
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Compound	*Targeted [1] Quantitation Limits*	
	Water (µg/L)	*Soil (µg/kg)*
TCL Semi-Volatile Organic Compounds (cont'd)		
2,4-Dichorophenol	10	330
Diethylphthalate	5	330
2,4-Dimethylphenol	5	330
Dimethylphthalate	5	330
4,6-Dinitro-2-methylphenol	20	1,700
2,4-Dinitrophenol	20	1,700
2,4-Dinitrotoluene	5	330
2,6-Dinitrotoluene	5	330
bis(2-Ethylhexyl)phthalate	5	330
Fluoranthene	5	330
Fluorene	5	330
Hexachlorobenzene	5	330
Hexachlorobutadiene	5	330
Hexachlorocyclopentadiene	5	330
Hexachloroethane	5	100
Indeno(1,2,3-cd)pyrene	5	330
Isophorone	5	330
2-Methylnaphthalene	5	330
2-Methylphenol	5	330
4-Methylphenol	5	330
Naphthalene	5	330
2-Nitroaniline	20	1,700
3-Nitroaniline	20	1,700
4-Nitroaniline	20	1,700
Nitrobenzene	5	330
2-Nitrophenol	5	330
4-Nitrophenol	20	1,700
N-Nitroso-di-n-propylamine	5	330
N-Nitrosodiphenylamine (diphenylamine)	5	330
di-n-Octylphthalate	5	330
Pentachlorophenol	5	3,400
Phenanthrene	5	330
Phenol	5	330
Pyrene	5	330
2,4,5-Trichlorophenol	5	1,700
2,4,6-Trichlorophenol	5	330

TABLE C1.2

TCL/TAL PARAMETER LIST
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Compound	*Targeted [1] Quantitation Limits*	
	Water (µg/L)	*Soil (µg/kg)*
TCL Polychlorinated Biphenyls (PCB)		
Aroclor 1016	0.2	33
Aroclor 1221	0.2	33
Aroclor 1232	0.4	33
Aroclor 1242	0.2	33
Aroclor 1248	0.2	33
Aroclor 1254	0.2	33
Aroclor 1260	0.2	33
TAL Inorganics		
Aluminum	100	10,000
Antimony	2	200
Arsenic	10	500
Barium	10	1,000
Beryllium	1	100
Cadmium	1	100
Calcium	200	20,000
Chromium	2	200
Cobalt	10	100
Copper	2	200
Iron	100	10,000
Lead	3	100
Magnesium	200	20,000
Manganese	10	1,000
Mercury	0.2	33
Nickel	2	100
Potassium	3,000	300,000
Selenium	5	500
Silver	5	100
Sodium	5,000	500,000
Thallium	1	100
Vanadium	10	1,000
Zinc	20	1,000
Cyanide (total)	10	500
Hexavalent Chromium	0.5	500

Note:

[1] - Please note that these are targeted 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 C1.3

MODIFIED APPENDIX IX PARAMETER LIST
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Compound	*Targeted Quantitation Limit (1) Soil (µg/kg)*
Appendix IX Volatile Organic Compounds (VOC)	
Acetone	20
Acetonitrile	100
Acrolein	100
Acrylonitrile	100
Allyl chloride (3-Chloropropene)	10
Benzene	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	10
2-Butanone	20
Carbon disulfide	5
Carbon tetrachloride	5
Chlorobenzene	5
Chloroethane	10
Chloroform	5
Chloromethane	10
Chloroprene (2-Chloro-1,3-butadiene)	5
Dibromochloromethane	5
1,2-Dibromo-3-chloropropane	10
1,2-Dibromoethane	5
Dibromomethane	5
1,2-Dichlorobenzene	5
1,3-Dichlorobenzene	5
1,4-Dichlorobenzene	5
trans-1,4-Dichloro-2-butene	5
Dichlorodifluoromethane	10
1,1-Dichloroethane	5
1,2-Dichloroethane	5
1,1-Dichloroethene	5
1,2-Dichloroethene (total)	5
1,2-Dichloropropane	5
cis-1,3-Dichloropropene	5
trans-1,3-Dichloropropene	5
1,4-Dioxane	500
Ethylbenzene	5
Ethyl methacrylate	5
Iodomethane	5
Isobutanol	2,000
2-Hexanone	20

TABLE C1.3

MODIFIED APPENDIX IX PARAMETER LIST
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Compound	*Targeted Quantitation Limit (1) Soil (µg/kg)*
Methacrylonitrile	50
Methylene chloride	5
Appendix IX Volatile Organic Compounds (Cont'd)	
Methyl methacrylate	50
4-Methyl-2-pentanone	20
Propionitrile	2,000
Styrene	5
1,1,1,2-Tetrachloroethane	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	10
1,2,3-Trichloropropane	5
Vinyl acetate	10
Vinyl chloride	10
Xylenes (total)	10
Appendix IX Semi-Volatiles Organic Compounds (SVOC)	
Acenaphthene	330
Acenaphthylene	330
Acetophenone	330
2-Acetylaminofluorene	3,300
4-Aminobiphenyl	1,600
Aniline	330
Anthracene	330
Aramite	660
Benzo(a)anthracene	330
Benzo(b)fluoranthene	330
Benzo(k)fluoranthene	330
Benzo(g,h,i)perylene	330
Benzo(a)pyrene	330
Benzyl alcohol	330
4-Bromophenyl phenyl ether	330
Butyl benzyl phthalate	330
4-Chloroaniline	330
Chlorobenzilate	330
bis(2-Chloroethoxy)methane	330
bis(2-Chloroethyl)ether	330

TABLE C1.3

MODIFIED APPENDIX IX PARAMETER LIST
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Compound	*Targeted Quantitation Limit (1) Soil (µg/kg)*
bis(2-Chloroisopropyl)ether	330
4-Chloro-3-methylphenol	330
2-Chloronaphthalene	330
2-Chlorophenol	330
Appendix IX Semi-Volatiles Organic Compounds (cont'd)	
4-Chlorophenyl phenyl ether	330
Chrysene	330
Diallate	660
Dibenz(a,h)anthracene	330
Dibenzofuran	330
Di-n-butyl phthalate	330
3,3'-Dichlorobenzidine	1,600
2,4-Dichlorophenol	330
2,6-Dichlorophenol	330
Diethyl phthalate	330
Dimethoate	660
p-Dimethylaminoazobenzene	330
7,12-Dimethylbenz(a)-anthracene	660
3,3'-Dimethylbenzidine	1,600
a,a-Dimethylphenethyl-amine	1,600
2,4-Dimethylphenol	330
Dimethyl phthalate	330
1,3-Dinitrobenzene	330
4,6-Dinitro-2-methylphenol	1,600
2,4-Dinitrophenol	1,600
2,4-Dinitrotoluene	330
2,6-Dinitrotoluene	330
Di-n-octyl phthalate	330
Diphenylamine	330
bis(2-Ethylhexy) phthalate	330
Ethyl methanesulfonate	330
Fluoranthene	330
Fluorene	330
Hexachlorobenzene	330
Hexachlorobutadiene	330
Hexachlorocyclopentadiene	1,600
Hexachloroethane	330
Hexachlorophene	3,300
Hexachloropropene	3,300
Indeno (1,2,3-cd)pyrene	330
Isophorone	330

TABLE C1.3

MODIFIED APPENDIX IX PARAMETER LIST
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Compound	*Targeted Quantitation Limit (1) Soil (µg/kg)*
Isosafrole	660
Methapyrilene	1,600
3-Methylcholanthrene	660
Methyl methanesulfonate	330
2-Methylnaphthalene	330
2-Methylphenol	330
Appendix IX Semi-Volatiles Organic Compounds (cont'd)	
3/4-Methylphenol	330
Naphthalene	330
1,4-Naphthoquinone	1,600
1-Naphthylamine	330
2-Naphthylamine	330
2-Nitroaniline	1,600
3-Nitroaniline	1,600
4-Nitroaniline	1,600
Nitrobenzene	330
2-Nitrophenol	330
4-Nitrophenol	1,600
4-Nitroquinoline-1-oxide	3,300
N-Nitroso-di-n-butylamine	330
N-Nitrosodiethylamine	330
N-Nitrosodimethylamine	330
N-Nitrosodiphenylamine	330
N-Nitroso-di-n-propylamine	330
N-Nitrosomethylethylamine	330
N-Nitrosomorpholine	330
N-Nitrosopiperidine	330
N-Nitrosopyrrolidine	330
5-Nitro-o-toluidine	330
Pentachlorobenzene	330
Pentachloroethane	1,600
Pentachloronitrobenzene	1,600
Pentachlorophenol	330
Phenacetin	660
Phenanthrene	330
Phenol	330
4-Phenylenediamine	3,300
2-Picoline	660
Pronamide	660
Pyrene	330
Pyridine	660

TABLE C1.3

MODIFIED APPENDIX IX PARAMETER LIST
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Compound	*Targeted Quantitation Limit (1) Soil (µg/kg)*
Safrole	330
1,2,4,5-Tetrachlorobenzene	330
2,3,4,6-Tetrachlorophenol	1,600
2-Toluidine	660
1,2,4-Trichlorobenzene	330
2,4,5-Trichlorophenol	330
2,4,6-Trichlorophenol	330
1,3,5-Trinitrobenzene	1,600
Appendix IX Polychlorinated Biphenyls (PCB)	
Aroclor 1016	330
Aroclor 1221	330
Aroclor 1232	330
Aroclor 1242	330
Aroclor 1248	330
Aroclor 1254	330
Aroclor 1260	330
Appendix IX Inorganics	
Antimony	200
Barium	1,000
Beryllium	100
Cadmium	100
Chromium	200
Cobalt	100
Copper	200
Nickel	100
Silver	100
Tin	1,000
Vanadium	1,000
Zinc	1,000
Arsenic	500
Lead	1,000
Mercury	33
Thallium	100
Cyanide	500
Selenium	500
Sulfide	1,000

TABLE C1.3

MODIFIED APPENDIX IX PARAMETER LIST
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Compound	*Targeted Quantitation Limit (1) Soil (µg/kg)*

Notes:

(1) - Actual sample quantitation limits are highly matrix dependent. Targeted quantitation presented are for guidance only. Targeted quantitation limits may be affected by matrix interferences, QA/QC problems and high concentrations of target and non-targ analytes.

1.4.1.1 FIELD PARAMETERS

The field parameters are listed in Table 1.1. The field soil parameters include headspace screening with a photoionization detector (PID). The rationale for choosing this soil parameter is to assist in identifying the appropriate depth or location of soil sample collection. Groundwater field parameters include pH/temperature, and conductivity, in order to access stability of groundwater during monitor well purging prior to sample collection.

1.4.1.2 LABORATORY PARAMETERS

The project-specific laboratory parameters will consist of target compound list (TCL) volatile organic compounds (VOC), TCL semi-volatile organic compounds (SVOC), polychlorinated biphenyls (PCB), and target analyte list (TAL) inorganics (metals and cyanide). Select samples will be analyzed for Appendix IX VOC, SVOC, PCBs, and metals. Table 1.2 and Table 1.3 present the targeted quantitation limits (TQL) for each compound or analyte. The basis for the selected parameters list is provided in RFI Work Plan. The TQLs and/or laboratory method detection limits (MDL) meet the recommended target method detection limits established by the State of Michigan with the exception of atrazine in water; potassium, sodium, aluminum, and cyanide in soil.

1.5 SAMPLING LOCATIONS

The intended soil and groundwater monitoring well sampling locations are described in the RFI Work Plan, which is fully incorporated into this QAPP through reference. It is possible, however, that depending on the nature of encountered field conditions, sampling locations may be changed. The person who shall be responsible for making such decisions will be the Field QA Officer whose responsibilities are described in Section 2 of this QAPP. Locations of monitoring and residential wells to be sampled, with associated screen depths, are indicated on Figures provided in the RFI Work Plan. Any changes to this sampling strategy will only be implemented after receipt of approval from the REALM Project Manager.

1.5.1 RATIONALE FOR SELECTED SAMPLING LOCATIONS

The rationale for the selected sampling locations (and depths) at each area of interest are fully described in the Section 5.0 of the RFI Work Plan.

1.5.2 SAMPLE NETWORK BY TASK AND MATRIX

Sample matrices, analytical parameters and frequencies of sample collection are presented in Table 1.1.

1.6 PROJECT SCHEDULE

The sampling and analysis schedule is presented in the RFI Work Plan.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

The REALM Project Manager shall have overall responsibility for all phases of the RFI. CRA will perform and be responsible for the field investigation and preparation of the RFI Report. Severn Trent Laboratories (formerly Quanterra) in North Canton, Ohio (STL-NC) with the support of STL Denver, Colorado and Richland, Washington has been selected as the project laboratory providing all laboratory analysis. Project management will also be provided by REALM and CRA. The various quality assurance, field, laboratory and management responsibilities of key project personnel are defined below.

2.1 PROJECT ORGANIZATION CHART

The lines of authority for this specific project are presented on Figure 2.1.

2.2 MANAGEMENT RESPONSIBILITIES

Cheryl Hiatt – REALM Project Manager

The REALM Project Manager is responsible for implementing the project, and has the authority to commit the resources necessary to meet project objectives and requirements. The Project Manager's primary function is to ensure that technical, financial and scheduling objectives are achieved successfully. The REALM Project Manager will provide the major point of contact and control for matters concerning the project. The REALM Project Manager will:

i) be the point of contact for all agency communication;

ii) define project objectives and develop a detailed work plan schedule;

iii) establish project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task;

iv) acquire and apply technical and corporate resources as needed to ensure performance within budget and schedule constraints;

v) orient all field leaders and support staff concerning the projects special considerations;

vi) monitor and direct the field leaders;

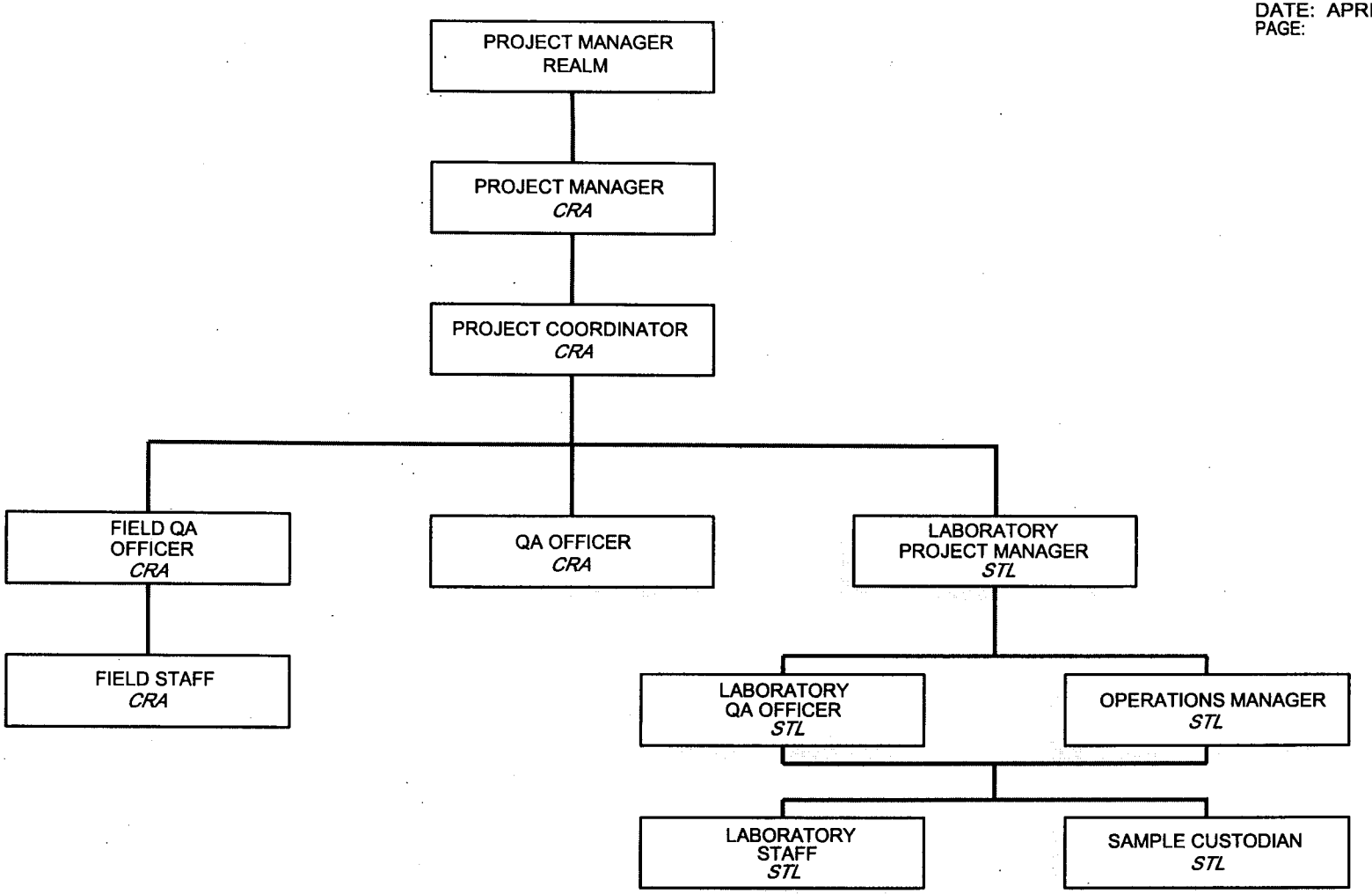


figure 2.1

PROJECT QA/QC ORGANIZATION
FORMER PEREGRINE (US) , INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

vii) develop and meet ongoing project and/or task staffing requirements, including mechanisms to review and evaluate each task product;

viii) review the work performed on each task to ensure its quality, responsiveness and timeliness;

ix) review and analyze overall task performance with respect to planned requirements and authorizations;

x) approve all reports (deliverables) before their submission to U.S. EPA Region V;

xi) ultimately be responsible for the preparation and quality of interim and final reports;

xii) represent the project team at meetings and public hearings; and

xiii) approval of the QAPP.

Glenn Turchan - CRA Project Manager

The CRA Project Manager has overall responsibility for ensuring that the project meets U.S. EPA's objectives and CRA quality standards. The CRA Project Manager will provide assistance to the REALM Project Manager in terms of writing and distributing the QAPP to all those parties connected with the project (including the laboratories). The CRA Project Manager will report directly to the REALM Project Manager and is responsible for technical quality control and project oversight. The CRA Project Manager will provide approval of the QAPP.

Randy Moore – CRA Project Coordinator

The CRA Project Coordinator will assist the CRA Project Manager in day-to-day project management. He is responsible for coordinating the procurement of project subcontractors. The CRA Project Coordinator will:

i) assist the CRA Project Manager in monitoring RFI progress and quality;

ii) provide overview of field activities;

iii) prepare and review RFI reports;

iv) provide technical representation of project activities; and

v) coordinate CRA's technical group.

2.3 QUALITY ASSURANCE RESPONSIBILITIES

Paul Wiseman - CRA QA Officer

The CRA QA Officer reports directly to the CRA Project Manager and will be responsible for ensuring that all QA/QC procedures for this project are being followed. The QA Officer will be responsible for the data validation of all sample results from the analytical laboratories. Additional responsibilities include:

i) laboratory system audits;

ii) overview and review field QA/QC; ;

iii) coordinate supply of performance evaluation samples and review results from performance audits;

iv) review laboratory QA/QC;

v) advise on data corrective action procedures;

vi) preparation and review of reports;

vii) QA/QC representation of project activities; and

viii) approval of the QAPP.

2.4 LABORATORY RESPONSIBILITIES

STL-NC will perform all analytical services in support of the RFI Work Plan.

Amy McCormick - Project Manager – STL-NC

The Project Manager will report directly to the CRA QA Officer and will be responsible for the following:

i) ensuring all resources of the laboratory are available on an as-required basis;

ii) advise CRA's QA Officer of laboratory status;

iii) review and approval of final analytical reports; and

iv) approval of the QAPP.

Stacey Campbell - Operations Manager – STL-NC

The operation manager will report to the Project Manager and will be responsible for:

i) coordinating laboratory analyses;

ii) supervising in-house chain-of-custody;

iii) scheduling sample analyses;

iv) overseeing data review; and

v) overseeing preparation of analytical reports.

Opal Johnson - QA Officer – STL-NC

The QA Officer has the overall responsibility for data after it leaves the laboratory. The QA Officer will be independent of the laboratory but will communicate data issues through the Project Manager. In addition, the QA Officer will:

i) overview laboratory quality assurance;

ii) overview QA/QC documentation;

iii) conduct data review;

iv) determine whether to implement laboratory corrective actions, if required;

v) define appropriate laboratory QA procedures; and

vi) approval of the QAPP.

Lois Ezzo - Sample Custodian – STL-NC

The Sample Custodian will report to the Operations Manager. Responsibilities of the Sample Custodian will include:

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 Laboratory Manager and Laboratory Supervisor of sample receipt and inspection;

vi) assigning a unique identification number and customer number, and entering each into the sample receiving log;

vii) with the help of the Operations Manager, initiating transfer of the samples to lab sections; and

viii) controlling and monitoring access/storage of samples and extracts.

Final responsibility for project quality rests with CRA's Project Manager. Independent quality assurance will be provided by each laboratory's Project Manager and QA Officer prior to release of all data to CRA and REALM.

Project Laboratory - Technical Staff

The technical staff will be responsible for sample analysis and identification of corrective actions. The staff will report directly to the operations manager.

2.5 FIELD RESPONSIBILITIES

Ken Vander Meulen - CRA Field QA Officer

The REALM and CRA Project Managers will be supported by the CRA Field QA Officer who is responsible for leading and coordinating the day-to-day activities of the field team. The CRA Field QA Officer will report directly to the CRA Project Manager. Specific responsibilities include:

i) provision of day-to-day coordination with the CRA Project Manager on technical issues in specific areas of expertise;

ii) developing and implementing of field-related work plans, assurance of schedule compliance, and adherence to management-developed study requirements;

iii) coordinating and managing of field staff including sampling and drilling;

iv) performing field system audits;

v) overseeing QC for technical data provided by the field staff including field measurement data;

vi) adhering to work schedule;

vii) authoring, writing and approving of text and graphics required for field team efforts;

viii) coordinating and overseeing of technical efforts of subcontractors assisting the field team;

ix) identifying problems at the field team level, resolving difficulties in consultation with the CRA Project Manager, implementing and documenting corrective action procedures and provision of communication between team and upper management;

x) approving the QAPP; and

xi) participating in preparation of the final report.

CRA Field Technical Staff

The field team for this project will be drawn from CRA's pool of corporate resources. The field team will be utilized to gather and analyze data and to prepare various task reports and support materials.

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

The overall QA objective is to develop and implement procedures for field sampling, chain-of-custody, laboratory analyses and reporting that will provide results which are legally defensible in a court of law. This section will provide in greater detail specific project objectives and intended data usages mentioned in Section 1.0 of this QAPP. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal QC, audits, preventative maintenance of field/laboratory equipment, and corrective action are described in other sections of this QAPP.

3.1 PRECISION

3.1.1 DEFINITION

Precision is a measure of degree to which two or more measurements are in agreement.

3.1.2 FIELD PRECISION OBJECTIVES

Field precision is assessed through the collection and measurement of field duplicates at a rate of one duplicate per 10 investigative samples. The total number of duplicates for this project is found in Table 1.1.

3.1.3 LABORATORY PRECISION OBJECTIVES

Precision in the laboratory is assessed through the calculation of relative percent differences (RPD). The equation to be used to determine precision in this project is presented in Section 12.3.1 of this QAPP. Precision control limits are presented in Table 3.1.

3.2 ACCURACY

3.2.1 DEFINITION

Accuracy is the degree of agreement between an observed or measured value and an accepted reference or true value.

3.2.2 FIELD ACCURACY

Accuracy in the field is assessed through the use of field and trip blanks and through the adherence to all sample handling procedures, sample preservation requirements and holding time periods.

3.2.3 LABORATORY ACCURACY OBJECTIVES

Laboratory accuracy is assessed through the analysis of matrix spikes (MS) or standard reference materials (SRM) or laboratory control samples and the determination of percent recoveries. The equation to be used to determine accuracy in this project is presented in Section 12.3.1 of this QAPP. Accuracy control limits are presented in Table 3.1 and the applicable Standard Operating Procedures (SOPs) as referenced in Section 7.0 of this QAPP.

The accuracy of the organics 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 sample preparation and analysis. These compounds are not expected to be found naturally occurring in the samples but behave analytically similar to the compounds of interest. Consequently, surrogate compound percent recoveries will provide information on the effect of the sample matrix on the accuracy of the analyses. Table 3.2 provides surrogate compound control limits.

TABLE C3.1

PRECISION AND ACCURACY CONTROL LIMITS
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Analyses	*MS/MSD Percent Recover Control Limits*[1]		*Laboratory Control Sample Percent Recovery Control Limits*[1]	
	Water	*Soil*	*Water*	*Soil*
VOC[2]				
Benzene	81-116 (18)	84-121 (13)	84-112 (14)	85-120 (13)
Chlorobenzene	82-117 (18)	76-122 (22)	89-109 (10)	88-119 (22)
1,1-Dichloroethene	75-126 (25)	75-131 (17)	81-121 (20)	76-128 (17)
Toluene	77-120 (21)	79-129 (23)	85-112 (13)	86-118 (23)
Trichloroethene	69-125 (28)	78-121 (17)	86-110 (12)	86-116 (17)
SVOC[2]				
Acenaphthene	41-101 (27)	32-110 (33)	42-107 (27)	50-100 (33)
4-Chloro-3-methylphenol	38-109 (30)	29-115 (43)	39-110 (30)	50-107 (43)
2-Chlorophenol	36-96 (32)	29-104 (40)	36-101 (32)	52-97 (40)
1,4-Dichlorobenzene	32-84 (31)	23-100 (50)	33-87 (31)	50-93 (50)
2,4-Dinitrotoluene	47-110 (19)	30-117 (38)	45-121 (19)	47-115 (38)
4-Nitrophenol	25-126 (33)	10-136 (58)	12-138 (33)	31-129 (58)
N-Nitroso-di-n-propylamine	35-98 (31)	26-105 (39)	37-103 (31)	46-96 (39)
Pentachlorophenol	29-126 (32)	10-118 (85)	33-111 (32)	17-106 (85)
Phenol	23-99 (33)	24-102 (43)	16-109 (33)	44-98 (43)
Pyrene	42-118 (26)	18-133 (41)	50-124 (26)	45-121 (41)
1,2,4-Trichlorobenzene	35-94 (29)	30-109 (40)	39-94 (29)	52-101 (40)
PCB				
Aroclor 1016	56-119 (20)	26-144 (39)	61-118 (20)	49-122 (39)
Aroclor 1260	31-138 (27)	37-138 (33)	61-124 (27)	51-127 (33)
Inorganics				
Aluminum	80-120 (20)	80-120 (20)	87-115 (14)	80-113 (20)
Antimony	80-120 (20)	80-120 (20)	87-108 (11)	80-104 (15)
Arsenic	80-120 (20)	80-120 (20)	80-120 (20)	80-120 (20)
Barium	80-120 (20)	80-120 (20)	87-110 (12)	80-109 (15)
Beryllium	80-120 (20)	80-120 (20)	85-110 (12)	80-105 (14)
Cadmium	80-120 (20)	80-120 (20)	89-115 (13)	80-112 (16)
Calcium	80-120 (20)	80-120 (20)	86-109 (12)	80-109 (16)
Chromium	80-120 (20)	80-120 (20)	86-112 (13)	81-116 (17)
Cobalt	80-120 (20)	80-120 (20)	83-107 (12)	80-104 (14)
Copper	80-120 (20)	80-120 (20)	84-112 (14)	80-113 (18)
Iron	80-120 (20)	80-120 (20)	80-120 (20)	80-120 (20)
Lead	80-120 (20)	80-120 (20)	80-120 (20)	80-120 (20)

TABLE C3.1

PRECISION AND ACCURACY CONTROL LIMITS
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Analyses	*MS/MSD Percent Recover Control Limits*[1]		*Laboratory Control Sample Percent Recovery Control Limits*[1]	
	Water	*Soil*	*Water*	*Soil*
Inorganics (cont'd)				
Magnesium	80-120 (20)	80-120 (20)	88-112 (12)	80-109 (17)
Manganese	80-120 (20)	80-120 (20)	88-117 (14)	80-114 (17)
Mercury	80-120 (20)	80-120 (20)	80-120 (20)	80-120 (20)
Nickel	80-120 (20)	80-120 (20)	85-116 (15)	80-112 (16)
Potassium	80-120 (20)	80-120 (20)	87-106 (9)	80-103 (12)
Selenium	80-120 (20)	80-120 (20)	80-120 (20)	80-120 (20)
Silver	80-120 (20)	80-120 (20)	93-120 (14)	81-120 (20)
Sodium	80-120 (20)	80-120 (20)	88-107 (9)	80-107 (14)
Thallium	80-120 (20)	80-120 (20)	80-120 (20)	80-120 (20)
Vanadium	80-120 (20)	80-120 (20)	86-111 (12)	80-111 (17)
Zinc	80-120 (20)	80-120 (20)	83-120 (24)	80-112 (21)
Cyanide	70-130 (20)	70-130 (20)	70-130 (20)	70-130 (20)
Hexavalent Chromium	90-110 (20)	75-125 (35)	90-110 (20)	90-110 (20)

Notes:

[1] Values in parenthesis are the maximum relative percent difference (RPD) values allowed for MS/MSD, sample/DUP or LCS/LCD analyses. Laboratory control limits are updated on a periodic basis and the control limits in effect when the samples are analyzed, identified in the laboratory's report will be used for data validation purposes.

[2] The laboratory may prepare and analyze an "all-analyte" spike; however, the control analytes presented are utilized for QC batch control.

3.3 COMPLETENESS

3.3.1 DEFINITION

Completeness is a measure of the amount of valid (usable) data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions.

3.3.2 FIELD COMPLETENESS OBJECTIVES

Completeness is a measure of the amount of valid measurements obtained from all measurements taken in this project. The equation for completeness is presented in Section 12.1 of this QAPP. Field completeness for this project will be 90 percent or greater.

3.3.3 LABORATORY COMPLETENESS OBJECTIVES

Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. The equation for completeness is presented in Section 12.0 of this QAPP. Laboratory completeness for this project will be 95 percent or greater.

3.4 REPRESENTATIVENESS

3.4.1 DEFINITION

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition or an environmental condition within a defined spatial and/or temporal boundary.

3.4.2 MEASURES TO ENSURE REPRESENTATIVENESS OF FIELD DATA

Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the [FSP] is followed and that proper sampling

TABLE C3.2

SURROGATE CONTROL LIMITS
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Analyses	*Control Limits* [1]	
	Water Recovery (%)	*Soil Recovery (%)*
VOC		
4-Bromofluorobenzene	75-122	60-137
Dibromofluoromethane	77-127	70-135
1,2-Dichloroethane-d4	64-140	75-117
Toluene-d8	84-112	86-122
SVOC		
2-Fluorobiphenyl	29-103	21-108
2-Fluorophenol	19-101	26-108
Nitrobenzene-d5	34-106	24-115
Phenol-d5	10-110	25-105
Terphenyl-d14	10-160	29-134
2,4,6-Tribromophenol	27-120	10-123
Pesticides/PCB		
Decachlorobiphenyl	61-118	23-141
Tetrachloro-meta-xylene	61-124	31-127

Note:

[1] Laboratory control limits are updated on a periodic basis and the control limits in effect when the samples are analyzed will be used for data validation purposes.

techniques are used. In designing the sampling program, media of concern have been specified.

3.4.3 MEASURES TO ENSURE REPRESENTATIVENESS OF LABORATORY DATA

Representativeness in the laboratory is ensured by using the proper analytical procedures, meeting sample holding times and analyzing and assessing field duplicate samples. The sampling network was designed to provide data representative of facility conditions. During development of this network, consideration was given to past waste disposal practices, existing analytical data, physical setting and processes and constraints inherent to the RCRA program. The rationale of the sampling network has been provided in Section 5.0 of the RFI Work Plan.

3.5 DECISION RULES

3.5.1 DEFINITION

A Decision Rule is a statement which allows for a course of action or non-action to be taken, based on assumptions made to draw out and test its logical or empirical consequences.

3.5.2 DECISION RULE OBJECTIVE

The Decision rule objectives address the following:

- Define statistical parameter(s) characterizing the population (e.g., mean, maximum, percentile) and incorporate the scale of decision-making (e.g., solid waste management unit (SWMU)). For this project, samples shall be collected discretely without compositing to provide greater indication of locally contaminated zones and hot spots.
- Identify action level(s) (e.g., Soil Screening Level; Maximum Contaminant Level for drinking water; Ecological Data Quality Levels (EDQL) or a reference-based standard).

- Develop "if/then" statements defining conditions that would cause the decision maker to choose among alternative actions (e.g., remediation or no remediation).

Decision levels, expressed as human health or ecological target decision levels, for each critical measurement parameter are specified in the Work Plan of this QAPP.

The Decision rules for the RFI at this facility can be stated as follows:

1. Detectable concentrations of the compounds or analytes presented in Table 1.2 will be evaluated and compared to the Michigan Department of Environmental Quality (MDEQ) Generic Industrial and Residential Criteria as defined by Part 201 of Act 451 to determine the nature and preliminary extent of contamination at the facility.

3.6 COMPARABILITY

3.6.1 DEFINITION

Comparability is an expression of the confidence with which one data set can be compared with another.

3.6.2 MEASURES TO ENSURE COMPARABILITY OF FIELD DATA

Comparability is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the RFI Work Plan is followed and that proper sampling techniques are used.

3.6.3 MEASURES TO ENSURE COMPARABILITY OF LABORATORY DATA

The analytical data to be obtained during the RFI will be comparable to existing data by using similar analytical methods. Comparability is also dependent on similar QA objectives.

3.7 LEVEL OF QUALITY CONTROL EFFORT

Field blank, trip blank, method blank, field duplicate, laboratory duplicate, laboratory control sample, SRM or QC check samples and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling and laboratory analytical programs.

Field and trip blanks will be submitted to the project laboratory to provide the means to assess the quality of the data resulting from the field sampling program. Field blank samples (equipment rinsate blanks) are analyzed to check for contamination introduced during sampling at the Facility which may cause sample contamination. Trip blanks are used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage.

Method blank samples are generated within the laboratory and are used to assess contamination resulting from laboratory procedures. Field duplicate samples are analyzed to check for sampling and analytical reproducibility. Matrix spikes provide information about the effect of the sample matrix on the preparation and measurement methodology. Matrix spikes are performed in duplicate. One matrix spike/matrix duplicate (MS/MSD) or matrix spike/matrix duplicate (MS/DUP) will be collected/designated for every 20 or fewer investigative samples per sample matrix (i.e., groundwater, soil).

MS/MSD samples are prepared from investigative samples. Soil MS/MSD samples require the collection of triple sample volume for VOC samples. However, aqueous MS/MSD samples must be collected at double the volume for all parameters.

The general level of the QC effort will be one field duplicate and one field blank sample for every 10 or fewer investigative samples. Field blanks will be collected for every 10 or fewer aqueous investigative samples. One VOC trip blank consisting of purchased reagent water that has been purged with an inert gas will be included along with each shipment of aqueous VOC samples. Trip blank samples will be prepared by the laboratory and will remain unopened during the sampling event. Trip blank samples will be preserved by the laboratory in the same manner as the investigative samples.

The number of field duplicate, field blank, MS/MSD and MS/DUP samples to be collected are presented in Table 1.1. Sampling procedures are specified in the Field Sampling Plan (FSP) and Section 4.0 of this QAPP.

4.0 SAMPLING PROCEDURES

The sampling procedures for the RFI will be consistent for the objectives of this project. The FSP, which is presented as Appendix G to the RFI Work Plan, provides the sampling procedures for all sampling activities to be conducted during this investigation. The samples will be collected from areas of least contamination to most contamination, if known.

Refer to the following sections of the FSP for the following field procedures:

Equipment Cleaning/Decontamination	Section G.2.2
Soil Sampling Protocols	Section G.3.0
Monitoring Well Installation Procedures	Section G.4.1
Well Development Procedures	Section G.4.2
Groundwater Sampling Procedures	Section G.5.0

Additional information is provided in the following sections.

4.1 SAMPLE CONTAINERS

The appropriate sample containers will be provided by the project laboratory. The required sample container type and volume, number of containers per analysis, preservation method maximum holding times, packaging and shipping requirements are presented in Table 4.1. Specific packaging procedures are presented in Section 5.1.2.

4.1.1 OBTAINING CONTAMINANT-FREE SAMPLE CONTAINERS

The sample containers for the parameters detailed in Table 1.1 will beprovided by STL-NC. STL-NC will purchase I-CHEM certified ™200 series or equivalent containers. These containers will be cleaned by the manufacture 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-.5A. Certificates of analysis will be maintained by the laboratory for each container lot to document conformance to U.S. EPA specifications.

TABLE C4.1

**CONTAINER, PRESERVATION, SHIPPING AND PACKAGING REQUIREMENTS
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN**

Analyses[1]	*Sample Containers*[2]	*Preservation*	*Maximum Holding Time from Sample Collection*[3]	*Volume of Sample*	*Shipping*	*Normal Packaging*
WATER (Groundwater)						
VOC	Three 40 mL teflon-lined septum vials per analysis	HCl to pH < 2 Iced, ≤10° C	14 days for analysis	Fill completely, no air bubbles	Overnight or Hand Deliver	Foam Liner or Bubble-wrap
SVOC & PCB	Two 1 liter amber glass bottles per analysis	Iced, ≤10° C	7 days for extraction 40 days after extraction for analysis	Fill to neck of bottle	Overnight or Hand Deliver	Bubble Pack or Foam Chips
Metals	One 1 liter plastic bottle	HNO3 to pH < 2 Iced, ≤10° C	180 days (mercury-28 days) for analysis	Fill to neck of bottle	Overnight or Hand Deliver	Bubble Pack or Foam Chips
Total Cyanide	One 500 ml plastic bottle	NaOH to pH>12 ascorbic acid[4] Iced, ≤10° C	14 days for analysis	Fill to neck of bottle	Overnight or Hand Deliver	Bubble Pack or Foam Chips
Hexavalent Chromium	One 500 ml plastic bottle	Iced, ≤10° C	24 hours for analysis	Fill to neck of bottle	Overnight or Hand Deliver	Bubble Pack or Foam Chips
SOLID (Soil/Sediment)						
VOC	Three 5g En Core Sampler™ per analysis	Iced, ≤10° C	48 hours for extraction 14 days for analysis	Fill completely	Overnight or Hand Deliver	Bubble Pack or Foam Chips

TABLE C4.1

CONTAINER, PRESERVATION, SHIPPING AND PACKAGING REQUIREMENTS
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Analyses [1]	*Sample Containers* [2]	*Preservation*	*Maximum Holding Time from Sample Collection* [3]	*Volume of Sample*	*Shipping*	*Normal Packaging*
SOLID (continued)						
SVOC & PCB	One 4-ounce glass jar per analysis	Iced, ≤10° C	14 days for extraction 40 days after extraction for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Bubble Pack or Foam Chips
Metals	One 4-ounce glass jar	Iced, ≤10° C	180 days (mercury & hexavalent chromium 28 days) for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Bubble Pack or Foam Chips
Hexavalent Chromium	One 4-ounce glass jar	Iced, ≤10° C	28 days for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Bubble Pack or Foam Chips
Cyanide	One 4-ounce glass jar	Iced, ≤10° C	14 days for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Bubble Pack or Foam Chips
Sulfide	One 4-ounce glass jar	Iced, ≤10° C	28 days for analysis	Fill to shoulder of jar	Overnight or Hand Deliver	Bubble Pack or Foam Chips
pH	One 4-ounce glass jar	Iced, ≤10° C	analyze immediately	Fill to shoulder of jar	Overnight or Hand Deliver	Bubble Pack or Foam Chips

Notes:

[1] - Parameter groups identified are inclusive of both TCL/TAL and Modified Appendix IX parameter lists.

[2] - Multiple parameters on a single sample may be combined into one single 16 ounce glass jar.

[3] - These are technical holding times, i.e., are based on time elapsed from time of sample collection.

[4] - If sulfides are present as determined by lead acetate field test, ascorbic acid will be added.

4.2 SAMPLE LABELING

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:

Example: S-12636-MMDDYY-XX-001

S	- designates types of sample (S-soil, SD-sediment, GW-groundwater, SW-surface water)
12636	- site reference number
MMDDYY	- designates date of collection presented as month/day/year
XX	- sampler's initials
001	- sequential number starting with 001 at the start of the project

Field 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 with a wire. The tag will provide information concerning the requested analyses and sample preservation in addition to the information provided on the sample label. An example of a sample tag and directions for its completion are provided in Attachment 1.

4.3 FIELD QC SAMPLE COLLECTION

4.3.1 FIELD BLANK SAMPLE COLLECTION

Field blank samples will be collected when non-dedicated sampling equipment are used to collect investigative samples. Field blanks consist of distilled 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., VOCs first, SVOCs second which are followed by the containers for the remaining analyses).

4.3.2 FIELD DUPLICATE SAMPLE COLLECTION

4.3.2.1 WATER SAMPLES

Field duplicate samples will be collected using the following procedure:

1. The bailer will be removed from the well and placed over the containers to be filled. If a pump is used (for semi-volatile compounds and inorganics only) the tubing will be placed over the containers to be filled.
2. The groundwater sample is poured from the bailer (or pumped) with minimal agitation into the first sample container until it is one-half full. The procedure is repeated for the second sample container.
3. The first sample container is filled to the proper level and sealed. The procedure is repeated for the second sample container.
4. The samples are properly labeled as specified in Section 4.2.
5. Steps 1 through 4 are repeated for the bottles for each analysis. The samples are collected in order of decreasing analyte volatility as detailed in Section 4.3.1.
6. Chain-of-custody documents are executed (Section 5.0).
7. The samples are packaged as specified in Table 4.1.

4.3.2.2 SOIL SAMPLES

Soil/sediment field duplicates will be collected as specified in the following:

1. The split-spoon sampler or trowel is retrieved from the sampling point.
1. Soil for VOC analysis is removed from the sampling device utilizing a specialized zero headspace sampling tool (EnCore Sampler™) as specified in Table C.4.1. The specific EnCore sampling procedures are presented in Section G.3.0 of the FSP.
2. Soil 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 requires that double the volume of sample is collected for aqueous samples. The sampling procedure is specified in Section 4.3.2.1 is used to collect aqueous MS/MSD.

Soil VOC samples will require triple the volume for MS/MSD analysis. No extra volume of soil is required for remaining parameter MS/MSD samples.

5.0 CUSTODY PROCEDURES

Custody is one of several factors which is 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:

i) the item is in actual possession of a person;

ii) the item is in the view of the person after being in actual possession of the person;

iii) the item was in actual possession but is locked up to prevent tampering; or

iv) the item is in a designated and identified secure area.

5.1 FIELD CUSTODY PROCEDURES

Field logbooks will provide the means of recording data collecting activities performed. As such, entries will be described in as much detail as possible so that persons going to the Facility could reconstruct a particular situation without reliance on memory.

Field logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel and will be stored with the project files when not in use. Each logbook will be identified by the project number which is 12636.

The title page of each logbook will contain the following:

i) person to whom the logbook is assigned;

ii) logbook number;

iii) project name;

iv) project start date; and

v) end date.

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors to the Facility during the RFI and the purpose of their visit will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. All entries will be made in ink with no erasures. If an incorrect entry is made, the information will be crossed out with a single strike mark and initialed. Whenever a sample is collected, or a measurement is made, a detailed description of the location of the sampling point, which includes compass direction and distance taken from a reference point will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the sampling procedures documented in the FSP as referenced in Section 4.0 and the additional procedures in Section 4.2 and 4.3 of this QAPP. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth from which the sample was collected, volume and number of containers. Sample identification number will be assigned during sample collection. Field QC samples, which will receive an entirely separate sample identification number, will be submitted blind to avoid laboratory bias of field QC samples.

The sample packaging and shipment procedures summarized in the following subsections will ensure that the samples will arrive at the laboratory with the chain-of-custody intact. The protocol for specific sample numbering and other sample designations are included in Section 4.2 of this QAPP. Examples of field custody documents and instructions for completion are presented in Attachment 1.

5.1.1 FIELD PROCEDURES

1. The field sampler is personally responsible for the care and custody of the samples until they are transferred to another individual or secure area or

properly dispatched to the laboratory. As few people as possible should handle the samples.

2. All bottles will be labeled with unique sample numbers and will have a sample tag affixed to the neck. Examples of sample labels are presented in Attachment 1.

3. Sample labels will be completed for each sample using waterproof ink unless prohibited by weather conditions such as freezing weather precluding the use of a ballpoint pen. In such cases, a pencil will be used and the deviation will be documented in the field logbook.

5.1.2 TRANSFER OF CUSTODY AND SHIPMENT PROCEDURES

1. Samples will be accompanied by a properly completed chain-of-custody record. The sample numbers and requested parameters will be listed on the chain-of-custody record. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the form. This form documents the transfer of custody of samples from the sampler to another person, to the laboratory, or to/from a secure storage area.

2. Samples will be packaged for shipment and shipped to the appropriate laboratory for analysis with a separate signed custody record enclosed in each sample cooler. Water VOC samples will be placed in bubble-wrap bags that that contain three containers per bag 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 plastic containers will be placed directly into the sample cooler. All the samples will be placed in an upright position without contacting each other in the cooler and packing will be limited to one layer of samples for water samples. After the containers are placed in the cooler, additional bubble-wrap or styrofoam chips will be added to the void space. Shipping containers will be secured with strapping tape and CRA custody tape for shipment to the laboratory. The procedure includes the use of custody tape attached to the right and left sides of the cooler.

3. Whenever samples are split with MDEQ-WMD or Facility representatives, a separate chain-of-custody record will be prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the Facility or Agency personnel will request the representative's signature acknowledging sample receipt.

4. All shipments will be accompanied by the chain-of-custody record identifying the contents. The original and yellow copies of the chain-of-custody record will accompany the shipment, and the pink and goldenrod copies will be retained by the shipper (if different than the sampler) and the sampler for return to the office where the copies will be kept with the project evidentiary files.

5. If the samples are sent by commercial carrier, a bill of lading will be used. A copy of the bill of lading will be retained as part of the 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.

6. Samples will be picked up by laboratory courier or transported by overnight courier to the laboratories the same day they are collected unless they are collected on a weekend or holiday. In these cases, they will be stored in a secure area until the next business day. Prior to shipment, additional ice will be added to the sample cooler.

5.2 LABORATORY CHAIN-OF-CUSTODY PROCEDURES

The sample custodian will assign a unique number to each incoming sample for use in the laboratory. The unique number and customer number will then be entered into the sample receiving log. The laboratory date of receipt will also be noted.

Laboratory custody procedures and document control for those samples analyzed by the project laboratory will be carried out using the laboratory's standard operating procedure (SOP) provided in Attachment 2 and the custody sequence provided as Figure 1.4. Samples tags will be maintained in the project laboratory's file. Examples of the chain-of-custody document(s) and procedures are provided in Attachment 1.

5.3 STORAGE OF SAMPLES

After the sample custodian has completed the sample receiving log, the chain-of-custody will be checked to ensure that all samples are stored in the appropriate location(s). All samples will be maintained and properly preserved (see Table 4.1) in an access-controlled location (i. e. walk-in-cooler) until completion of all analytical work or, at a minimum, for 30 days after receipt of the final report by CRA. At this time, the

samples will be disposed by the laboratory in accordance with all Federal, State and local requirements.

5.4 FINAL EVIDENCE FILES CUSTODY PROCEDURES

The final evidence file will be the central repository for all documents which constitute evidence relevant to sampling and analysis activities as described in this QAPP. CRA is the current custodian of the evidence file and will maintain the contents of evidence files for the RFI, including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports and data reviews in a secured, limited access area and under custody of CRA's project manager or his designee. The MDEQ-WMD will be offered the final evidence file prior to disposal (after 6 years).

The final evidence file will include at a minimum:

i) project plans and drawings;
ii) field log books;
iii) field data records and deliverables;
iv) sample identification documents and soil boring/monitoring well logs;
v) all chain-of-custody documentation;
vi) correspondence;
vii) references, literature;
viii) laboratory data deliverables;
ix) data validation and assessment reports;
x) progress reports, QA reports; and
xi) final report.

The laboratory will be responsible for maintaining analytical log books, laboratory data and sample tags. Raw laboratory data files and copies of hard copy reports will be inventoried and maintained by the laboratory for a period of six years, at which time the laboratories will notify CRA regarding the need for additional storage.

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 INSTRUMENTS CALIBRATION

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated as specified in the field standard operating procedures (SOPs).

The field instruments include pH meters, specific conductance meters and photoionization detectors. Temperature is measured in conjunction with pH using the same instrument and is pre-calibrated by the manufacturer. General cleaning and maintenance will be performed to ensure that reproducible temperature measurements are maintained.

Field instruments will be calibrated prior to use and the calibration will be verified after a maximum of every 10 investigative samples. Specific information concerning calibration frequency, acceptance criteria and the conditions that require more frequent calibration is contained in the field SOPs in Attachment 2.

6.2 LABORATORY INSTRUMENT CALIBRATION

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.

7.0 ANALYTICAL PROCEDURES

Soil and groundwater samples collected during the RFI will be analyzed for the parameters specified in Section 1.4 of this QAPP by STL-NC, 4101 Shuffel Drive NW, North Canton, Ohio 44720 with the support of STL Denver, Colorado and STL Richland, Washington for metals analyses.

7.1 FIELD ANALYTICAL PROCEDURES

The standardization and QA information for field measurement of VOC headspace screening with a PID and pH/temperature, and conductivity are described in Section 3.0 of this QAPP. Specific procedures for measurement of these parameters are provided as SOPs in Attachment 2. Table 7.1 identifies the Field SOPs and the analytical methods upon which the SOPs were based.

7.2 LABORATORY ANALYTICAL PROCEDURES

The laboratory identified in Section 7.0 will use the SOPs provided in Attachment 2 for the TCL/TAL and Appendix IX parameter analyses. Table 7.1 identifies the laboratory SOPs and the analytical methods upon which the SOPs were based for sample preparation and analysis. These SOPs are based on the methods contained in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA SW-846, 3rd Edition, Final Update III, 1996.

7.2.1 LIST OF PROJECT TARGET COMPOUNDS AND LABORATORY DETECTION LIMITS

A complete listing of project target compounds, and project quantitation limits for each analyte group listed in Table 7.1 are presented in Table 1.2. Method Detection Limits (MDLs) presented have been experimentally determined using the method provided in 40 CFR Part 136 Appendix B. The laboratory's MDL data are provided in Attachment 2. These MDLs are subject to change by the laboratory and the latest MDLs at the time of analysis will be utilized by the laboratory.

7.2.2 LIST OF ASSOCIATED QC

The laboratory SOPs listed in Table 7.1 include a QC section which addresses the minimum QC requirements for the analysis of specific analyte groups. Section 8.0 of this QAPP contains or references a complete listing of the associated QC samples for every analyte group and matrix.

TABLE C7.1

SUMMARY OF ANALYTICAL METHODS
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter[1]	*Preparation Method*	*Laboratory Preparation SOP*	*Analytical Method*	*Laboratory Analytical SOP*
Groundwater and Surface Water Samples				
VOC	SW-846 5030B[2]	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[4]				
ICP Metals	SW-846 3010A/3020A	CORP-IP-0003	SW-846 6010B	CORP-MT-0001DEN
ICP/MS Metals	SW-846 3010A/3020A	CORP-IP-0003	SW-846 6020	DEN-MT-0002
mercury	SW-846 7470A	CORP-MT-0005DEN	SW-846 7470A	CORP-MT-0005NC
Cyanide	SW-846 9012A	NC-WC-0032	SW-846 9012A	NC-WC-0031
Hexavalent Chromium	SW-846 7196A	NC-WC-0024	SW-846 7196A	NC-WC-0024
Soil, Sediment and Sludge 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[4]				
ICP Metals	SW-846 3050A	CORP-IP-0002DEN	SW-846 6010B	CORP-MT-0001DEN
ICP/MS Metals	SW-846 3050A	CORP-IP-0002DEN	SW-846 6020	DEN-MT-0002
mercury	SW-846 7471A	CORP-MT-0007DEN	SW-846 7471A	CORP-MT-0007NC
Cyanide	SW-846 9012A	NC-WC-0031	SW-846 9012A	NC-WC-0032
Hexavalent Chromium[5]	SW-846 3060A	RICH-WC-5005	SW-846 7196A	RICH-WC-5005
Sulfide	SW-846 9030A	NC-WC-0060	SW-846 9030A	NC-WC-0060
pH	SW-846 9045C	NC-WC-00160	SW-846 9045C	NC-WC-0010

Notes:

[1] Refer to Tables 1.2 and 1.3 for the compounds/elements of each parameter group. Methods and SOPs are inclusive of TCL/TAL and Appendix IX parameter lists.

[2] SW-846 - "Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods ", SW-846, 3rd Edition, Final Update III, 1996.

[3] Sediment and sludge samples collected as bulk samples will be prepared in accordance with SW-846 Method 5030B.

[4] Metals by Method (All metals will completed by STL's Denver, Colorado Laboratory)

Water ICP: Aluminum, Arsenic, Barium, Calcium, Cobalt, Iron, Lead, Magnesium, Manganese, Potassium, Selenium, Sodium, Vanadium, Zinc.

Water ICP/MS: Antimony, Beryllium, Cadmium, Chromium, Copper, Nickel, Silver, Thallium

Soil ICP: Aluminum, Barium, Calcium, Iron, Manganese, Magnesium, Potassium, Sodium, Tin, Vanadium

Soil ICP/MS: Arsenic, Antimony, Beryllium, Cadmium, Chromium, Cobalt, Copper, Lead, Nickel, Selenium, Silver, Thallium, Zinc

[5] Hexavalent Chromium analysis of soil samples will be completed by STS's Richland, Washington Laboratory

VOC = Volatile Organic Compounds
SVOC = Semi-Volatile Organic Compounds
PCB = Polychlorinated Biphenyls

8.0 INTERNAL QUALITY CONTROL CHECKS

This section presents the internal quality control checks which will be employed for field and laboratory measurements.

8.1 FIELD QUALITY CONTROL

QC procedures for pH/temperature, and conductivity of water samples and VOC headspace screening with a photoionization detector (PID) of 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 field blanks for laboratory analysis. Collection of the samples will be in accordance with the applicable procedures in Section 4.3 of this QAPP at the frequency indicated in Table 1.1.

8.1.1 FIELD BLANKS SAMPLES

Field blank samples will be collected in the field at a minimum frequency of one per every 10 or fewer aqueous investigative samples. Field blanks will be used to assess the efficiency of sampling equipment decontamination. The presence of analytes in the field blank samples will be indicative of potentially 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 multiple aqueous VOC samples. 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 every 10 or fewer investigative samples by matrix. 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:

- Calibration Standards
- Instrument Performance Checks - Organics
- Initial and Continuing Calibration Checks
- Internal Standard Performance
- Method Blanks Samples
- Laboratory Control Samples and QC Check Samples
- Matrix Spike/Matrix Spike Duplicates – Organic Analysis
- Surrogates
- Matrix Spike/Matrix Spike Duplicates – Inorganic Analyses
- Pesticide/PCB – QC Analyses
- ICP Interference Check Samples (ICS)
- ICP Serial Dilution
- ICP and ICP/MS QC Analyses
- Reagent Checks
- 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 reanalyzed by the laboratory, if sufficient volume is acceptable.

9.0 DATA REDUCTION, VALIDATION AND REPORTING

All data generated in field and laboratory activities will be reduced and validated prior to reporting. No data shall be disseminated by the laboratory until they have been subjected to these procedures which are summarized in the subsections below.

9.1 DATA REDUCTION

9.1.1 FIELD DATA REDUCTION PROCEDURES

Field data reduction procedures will be minimal in scope compared to those implemented for laboratory data. Only direct reading instrumentation will be employed in the field. The use of pH meters, specific conductance meters and photoionization meters will generate measurements directly read from the meters following calibration as outlined in the SOPs in Attachment 2. Such data will be written into field logbooks immediately after measurements are taken. If errors are made, results will be legibly crossed out, initialed by the field sampler and corrected in a space adjacent to the original (erroneous) entry. The Field QA Officer, identified in Section 2.0 of this QAPP, will periodically review the field logbooks to determine whether any transcription errors have been made by the field crew.

9.1.2 LABORATORY DATA REDUCTION PROCEDURES

Laboratory data reduction procedures will be followed according to the following protocol:

1. Raw data produced and checked by the responsible analyst is turned over for independent review by another analyst.
2. The area supervisor or senior chemist reviews the data for attainment of quality control criteria established by the QAPP.
3. Upon completion of all reviews and acceptance of the raw data by the laboratory area supervisor, a report will be generated and sent to the laboratory Project Manager.
4. The laboratory Project Manager will complete a thorough inspection of all reports.

5. The QA Officer and/or area supervisor will decide whether any sample reanalysis is required.

6. Upon acceptance of the preliminary reports by the Project Manager, final reports will be generated and signed by the Project Manager, while the case narrative is reviewed and signed by the QA Officer.

Specific equations used for data reduction are contained in the SOPs in Attachment 2.

9.2 DATA VALIDATION

Data validation procedures will be performed for both field and laboratory operations as described in the following subsections.

9.2.1 PROCEDURES USED TO EVALUATE FIELD DATA

Field data validation will be the responsibility of the Field QA Officer and will include the following:

1. An independent review of field log books to result forms;
2. Field log books will be inspected to ensure that field instrumentation calibration and QC checks are conducted and documented at the appropriate frequency; and
3. Determine the level of compliance of calibration and QC check acceptance criteria.

9.2.2 PROCEDURES TO VALIDATE LABORATORY DATA

Validation of the analytical data will be performed by CRA's QA Officer utilizing the "U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review", EPA-540/R-94-012, February 1994 and "U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review", EPA-540/R-94-013, February 1994 (CLP National Functional Guidelines) as a basis for data review establishing the specific objectives, defining the evaluation process and identifying the actions while incorporating the specific quality control limits presented

in Table 3.1, and Table 3.2 of this QAPP and laboratory SOPs, the calibration requirements defined in Section 6.2 of this QAPP, and the additional measurements present in the analytical procedures and laboratory SOP as outlined in Table 7.1 and Attachment 2 of this QAPP. The specific data qualifiers used will be as presented and defined in the CLP National Functional Guidelines. Validation will be performed, by trained CRA chemists at the direction of the CRA QA Officer. Full data validation will be completed on 50% of the expanded deliverables during Phase I activities. This may be reduced to 25% based on prior laboratory performance during subsequent activities.

The following deliverables will be evaluated in the full data validation:

Organic Analyses

i) technical holding times;
ii) GC/MS instrument performance check;
iii) initial and continuing calibration;
iv) blanks;
v) system monitoring compounds (surrogate spikes);
vi) MS/MSD results;
vii) laboratory control samples;
viii) internal standard performance;
ix) target compound identification and quantitation;
x) tentatively identified compounds;
xi) system performance;
xii) retention time windows (GC analyses); and
xiii) field duplicates.

Inorganic Analyses

i) technical holding times;
ii) initial and continuing calibration;
iii) blanks;
iv) interference check samples;
v) laboratory control samples;
vi) matrix duplicate sample analysis;
vii) matrix spike sample analysis;
viii) ICP serial dilution;
ix) ICP/MS internal standard performance;

x) sample result verification; and
xi) field duplicates.

The following deliverables will be evaluated for the remaining 50 or 75 percent of the data.

Organic Analyses

i) technical holding times;
ii) blanks;
iii) system monitoring compounds (surrogate spikes);
iv) MS/MSD results;
v) laboratory control samples; and
vi) field duplicates

Inorganic Analyses

i) technical holding times;
ii) blanks;
iii) laboratory control samples;
iv) MS/MSD results; and
v) field duplicates.

Method detection limit (MDL) studies have been performed by STL-NC in accordance with the provisions of Federal Register, Volume 49, number 209, October 26, 1984 pp. 198-199, the results of these MDL studies are provided in Attachment 2 (Laboratory Reference Data Summary).

The overall completeness of the data package will also 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, and QC summaries. The review will determine whether all required items are present and request copies of missing deliverables.

9.3 DATA REPORTING

Data reporting procedures will be carried out for field and laboratory operations as described in the following subsections.

9.3.1 FIELD DATA REPORTING

Field data reporting will 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 task of reporting laboratory data (to the MDEQ-WMD) by REALM begins after the validation activity has been concluded. The laboratory Project Manager will perform a final review of the report summaries and case narratives to determine whether the report meets the project requirements. In addition to the record of the chain-of-custody, the 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) QC procedures utilized and also references to the acceptance criteria;
   - vii) laboratory report contents;
   - viii) project name and number;
   - ix) condition of samples "as received";
   - x) discussion of whether or not sample holding times were met;
   - xi) discussion of technical problems or other observations which may have created analytical difficulties;

xii) discussion of laboratory QC checks which failed to meet project criteria; and

xiii) signature of the Laboratory QA Officer or his designee.

2. Chemistry Data Package

i) case narrative for each analyzed batch of samples;

ii) cross referencing of laboratory sample to project sample identification numbers;

iii) description of data qualifiers to be used;

iv) methods of sample preparation and analyses for samples;

v) sample results;

vi) raw data for sample results and laboratory quality control samples;

vii) results of (dated) initial and continuing calibration checks and GC/MS tuning results;

viii) 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);

ix) matrix spike recoveries and matrix duplicate relative percent differences, laboratory control sample, serial dilutions, method blank results, and reagent blank results (inorganics); and

x) labeled and dated chromatograms/spectra of sample results and laboratory quality control checks.

The data package submitted will be a "CLP-like" data package consisting of all the information presented in a CLP data package but not necessarily on CLP forms.

10.0 PERFORMANCE AND SYSTEM AUDITS

Performance and system audits of both field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in the FSP and QAPP. The audits of field and laboratory activities 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 including sampling and field measurements will be conducted by the CRA Field QA Officer. These audits will verify that all established procedures are being followed.

10.1.2 INTERNAL FIELD AUDIT FREQUENCY

These audits will verify that all established procedures are being followed. Internal field audits will be conducted at least once at the beginning of the site sample collection activities.

10.1.3 INTERNAL FIELD AUDIT PROCEDURES

The audits will include the examination of field sampling records, field screening analytical results, field instrument operating records, sample collection, handling and packaging in compliance with the established procedures, maintenance of QA procedures and chain-of-custody. Follow-up audits will be conducted to correct deficiencies and to verify that QA procedures are maintained throughout the RFI. The audits will involve review of field measurement records, instrumentation calibration records and sample documentation. The field audit checklist to be used for the RFI is presented in Attachment 1.

10.1.4 EXTERNAL FIELD AUDIT RESPONSIBILITIES

External audits may be conducted by the REALM Project Manager.

10.1.5 EXTERNAL FIELD AUDIT FREQUENCY

External field audits may be conducted 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 V.

10.1.6 OVERVIEW OF THE EXTERNAL FIELD AUDIT PROCESS

External field audits will be conducted according to the field activity information presented in this QAPP. The external field audit process can include (but not be limited to) sampling equipment decontamination procedures, sample bottle preparation procedures, sampling procedures, examination of field sampling and safety plans, sample vessel cleanliness and QA procedures, procedures for verification of field duplicates, sample preservation and preparation for shipment, as well as field screening practices.

10.2 LABORATORY PERFORMANCE AND SYSTEM AUDITS

10.2.1 INTERNAL LABORATORY AUDIT RESPONSIBILITIES

The internal laboratory audit will be conducted by the CRA QA Officer or General Motors QA Officer.

10.2.2 INTERNAL LABORATORY AUDIT FREQUENCY

The internal laboratory system audits will be performed on an annual basis while the internal laboratory performance audits will be conducted on a semi-annual basis depending on project duration.

10.2.3 INTERNAL LABORATORY AUDIT PROCEDURES

The internal laboratory system audits will include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis and instrument operating records. The

performance audits will involve preparing blind QC samples and submitting them along with project samples to the laboratory for analysis throughout the project. The CRA QA Officer will evaluate the analytical results of these blind performance samples to ensure the laboratory maintains acceptable QC performance. The laboratory audit checklist is provided in this QAPP as Attachment 1.

10.2.4 EXTERNAL LABORATORY AUDIT RESPONSIBILITIES

External audits will be conducted as required, by appropriate QA staff of General Motors Corporation, as part of GM regular audit program.

10.2.5 EXTERNAL LABORATORY AUDIT FREQUENCY

An external laboratory audit may be conducted at least once prior to the initiation of the sampling and analysis activities. Audits may or may not be announced and are at the discretion of REALM.

10.2.6 OVERVIEW OF THE EXTERNAL LABORATORY AUDIT PROCESS

External audits may include any or all of: review of laboratory analytical procedures, laboratory on-site visits, and/or submission of performance evaluation samples to the laboratory for analysis. Failure of any or all audit procedures chosen can lead to laboratory disqualification, and the requirement that another suitable laboratory be chosen.

An external on-site review can consist of: sample receipt procedures, custody and sample security and log in procedures, sample through put tracking procedure, review of instrument calibration records, instrument logs and statistics (number and type), review of QA procedures, log books, sample prep procedures, sample analytical SOP review, instrument (normal or extends quantitation report) reviews, personnel interviews, review of deadlines and glassware prep, and a close out to offer potential corrective action.

It is common practice when conducting an external laboratory audit to review one or more data packages from sample lots recently analyzed by the laboratory. This review will most likely include but not be limited to:

- Comparison of resulting data to the SOP or method, including coding for deviations.
- Verification of initial and continuing calibrations within control limits.
- Verification of surrogate recoveries and instrument timing results where applicable.
- Review of extended quantitation reports for comparisons of library spectra to instrument spectra, where applicable.
- Recoveries on control standard runs.
- Review of run logs with run times, ensuring proper order of runs.
- Review of spike recoveries/QC sample data.
- Review of suspected manually integrated GC data and its cause (where applicable).
- Review of GC peak resolution for isolated compounds as compared to reference spectra (where applicable).
- Assurance that samples are run within holding times.

Ideally, the data should be reviewed while on the premises, so that any data called into question can be discussed with the staff.

11.0 PREVENTIVE MAINTENANCE

11.1 FIELD INSTRUMENT PREVENTATIVE MAINTENANCE

The field equipment for this project includes pH meters, specific conductance meters and photoionization meters. Specific preventative maintenance procedures to be followed for field equipment are those recommended by the manufacturer. Field instruments will be checked and calibrated daily before use. The maintenance schedule and trouble-shooting procedures for field instruments are presented in Table 11.1.

11.2 LABORATORY INSTRUMENT PREVENTATIVE MAINTENANCE

As part of their QA/QC program, a routine preventative maintenance program is conducted by the laboratories to minimize the occurrence of instrument failure and other system malfunctions. Designated laboratory employees will regularly perform routine scheduled maintenance and repair of (or coordinate with the instrument manufacturer for the repair of) all instruments. All maintenance that is performed shall be documented in the laboratory's maintenance logbooks. All laboratory instruments are maintained in accordance with manufacturer's specifications.

Table 11.1 provides the frequency which components of key analytical instruments or equipment will be serviced.

TABLE C11.1

**ROUTINE MAINTENANCE PROCEDURES AND SCHEDULES
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN**

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 C11.1

ROUTINE MAINTENANCE PROCEDURES AND SCHEDULES
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

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 C11.1

ROUTINE MAINTENANCE PROCEDURES AND SCHEDULES
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

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 contai[illegible] free oil,	1. Standard solution 2. Spare electrodes

TABLE C11.1

ROUTINE MAINTENANCE PROCEDURES AND SCHEDULES
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Instrument/Equipment	*Maintenance Procedures/Schedule*	*Spare Parts in Stock*
	wash the electrode in soap and rinse thoroughly with water.	
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.	

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 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 for organic analyses. The relative percent difference (RPD) will be calculated for each pair of duplicate analyses.

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 (%R)

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:

$$\%R = \frac{SSR - SR}{SA} \quad X \ 100$$

where:

SSR = Spiked Sample Result
SR = Sample Result or Background
SA = Spike Added

12.3.2 RELATIVE PERCENT DIFFERENCE (RPD)

The relative percent difference 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:

R1 = value of first result
R2 = value of second result

13.0 CORRECTIVE ACTION

Corrective action is the process of identifying, recommending, approving and implementing measures to counter unacceptable procedures or out of quality control performance which can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation and data assessment. All corrective action proposed and implemented will be documented in the regular quality assurance reports to management. Corrective action should only be implemented after approval by the REALM or CRA Project Manager or his designee. If immediate corrective action is required, approvals secured by telephone from the REALM or CRA Project Manager will be documented in an additional memorandum or field logbook.

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the CRA or REALM Project Manager. If the problem is analytical in nature, information on these problems will be promptly communicated to REALM QA Officer and CRA's QA Officer. Implementation of corrective action will be confirmed in writing through the same channels.

Any nonconformance with the established quality control procedures in the QAPP or FSP will be identified and corrected in accordance with the QAPP. The REALM or CRA Project Manager, or his designee, will include a nonconformance report for each nonconformance condition in the quality assurance reports to management.

13.1 FIELD CORRECTIVE ACTION

Corrective action in the field may be necessary when the sample network is changed (i.e., more/less samples, sampling locations other than those specified in the QAPP), sampling procedures and/or field analytical procedures require modification, due to unexpected conditions. In general, the field team may identify the need for corrective action. The field team, in consultation with the Field QA Officer, will recommend a corrective action. The Field QA Officer will approve the corrective action which will be implemented by the field team. It will be the responsibility of the Field QA Officer to ensure the corrective action has been implemented.

If the corrective action will supplement the existing sampling plan (i.e., additional soil borings) using existing and approved procedures in the QAPP, corrective action

approved by the Field QA Officer will be documented. If corrective actions resulting in less samples (or analytical fractions) or alternate locations which may cause project quality assurance objectives not be achieved, it will be necessary that all levels of project management including the Field QA Officer concur with the proposed action.

Corrective action resulting from internal field audits will be implemented immediately if data may be adversely affected due to unapproved or improper use of approved methods. CRA's Field QA Officer will identify deficiencies and recommended corrective action to CRA's Project Manager. Implementation of corrective actions will be performed by CRA's Field QA Officer and field team. Corrective action will be documented in quality assurance reports to management.

Corrective actions will be implemented and documented in the field logbook. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective action are insufficient, work may be stopped by the REALM Project Manager.

13.2 LABORATORY CORRECTIVE ACTION

Corrective action in the laboratory may occur prior to, during and after initial analyses. A number of conditions such as broken sample containers, multiple phases, low/high pH readings, potentially high concentration samples may be identified during sample log-in or just prior to analysis. Following consultation with analysts and section leaders, it may be necessary for the laboratory QA Officer to approve the implementation of corrective action. The submitted SOPs specify some conditions during or after analysis that may automatically trigger corrective action or optional procedures. These conditions may include dilution of samples, additional sample extract cleanup, automatic reinjection/reanalysis when certain QC criteria are not met.

The bench chemist will identify the need for corrective action. The Laboratory Project Manager, in consultation with the laboratory supervisor and staff, will approve the required corrective action to be implemented by the laboratory staff. The laboratory QA Officer will ensure implementation and documentation of the corrective action. If the nonconformance causes project objectives not be achieved, it will be necessary to inform all levels of project management including REALM Project Manager to concur with the corrective action.

These corrective actions are performed prior to release of the data from the laboratory. The corrective action will be documented in both the laboratory's corrective action report and the narrative data report sent from the laboratory to CRA's QA Officer. If corrective action does not rectify the situations, the laboratory will contact CRA's QA Officer.

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. Potential types of corrective action may include resampling by the field team or re-injection/reanalysis of samples by the laboratory.

These actions are dependent upon the ability to mobilize the field team, whether the data to be collected is necessary to meet the required quality assurance objectives (e.g., the holding time for samples is not exceeded). When CRA's QA Officer identifies a corrective action situation, REALM Project Manager will be responsible for approving the implementation of corrective action, including resampling, during data assessment. All corrective actions of this type will be documented by CRA Project Manager.

14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The CRA Project Manager and REALM Project Manager may receive reports on the performance of the measurement system and data quality following each sampling round and at the conclusion of the project.

Minimally, these reports will include:

1. assessment of measurement quality indicators, i.e., data accuracy, precision and completeness;
2. results of system and performance audits; and
3. QA problems, corrective action taken and resolutions.

The CRA QA Officer will be responsible within the organizational structure for preparing these reports for submission the progress reports generated upon completion of each round of sampling activities. The final report for the project will also include a separate QA section which will summarize data quality information contained in the periodic QA reports to management, and details an overall data assessment and validation in accordance with the data quality objectives outlined in Section 1.4.3 of this QAPP.

15.0 REFERENCES

1. United States Environmental Protection Agency "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 "U.S. EPA RCRA QAPP Instructions", U.S. EPA Region V, Revision: April 1998.

3. United States Environmental Protection Agency "Preparation Aids for the Development of Category I Quality Assurance Project Plans", EPA/600/8-91/003, Risk Reduction Engineering Laboratory, Office of Research and Development, Cincinnati, Ohio, February 1991.

4. United States Environmental Protection Agency "Data Quality Objectives Process for Superfund, Interim Final Guidance", EPA 540/R-93-071, Office of Solid Waste and Emergency Response, September 1993.

5. United States Environmental Protection Agency "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", EPA 540/R-94-012.

7. United States Environmental Protection Agency "U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review", EPA 540/R-94-013.

ATTACHMENT 1

EXAMPLE FORMS AND CHECKLISTS

1.	Sample Label and Custody Tape	Figure ATT-1.1
2.	Example Sample Tag	Attachment - 1.2
3.	Chain-of-Custody Sequence	Figure ATT-1.3
4.	Laboratory Custody Sequence	Figure ATT-1.4
5.	Field Audit Checklist	Attachment -1.5
6.	Laboratory Audit Checklist	Attachment -1.6

CRA

Conestoga Rovers & Associates

JOB NAME : DATE :

JOB No. : SAMPLE I.D. :

SAMPLERS NAME : _____

ANALYSIS REQUEST : _____

NOTES: _____

_____

SAMPLE LABEL

NOTES:

1) SAMPLE LABELS TO BE FIRMLY AFFIXED TO SAMPLE CONTAINERS.

2) ALL SAMPLE LABELS TO BE COMPLETED USING WATER INSOLUBLE INK.

CRA

CONESTOGA-ROVERS & ASSOCIATES

11100 METRO AIRPORT CENTER DR. STE 160

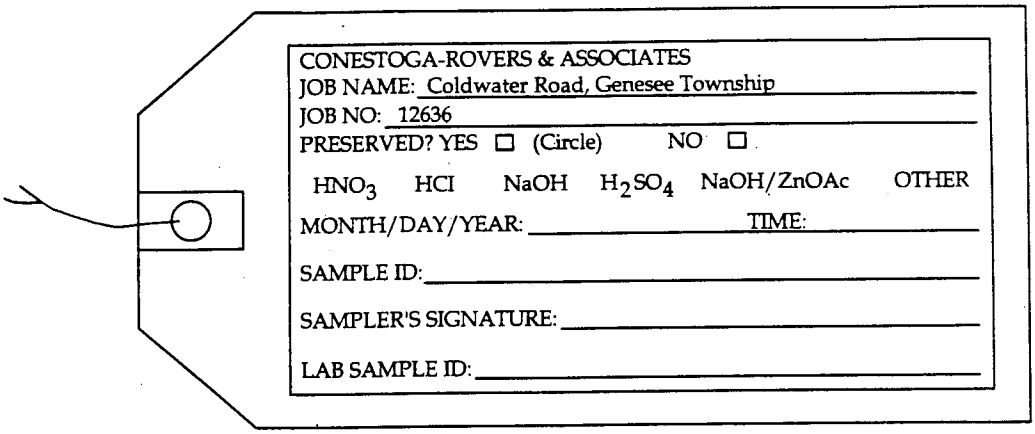
ROMULUS, MI 48174

CUSTODY TAPE

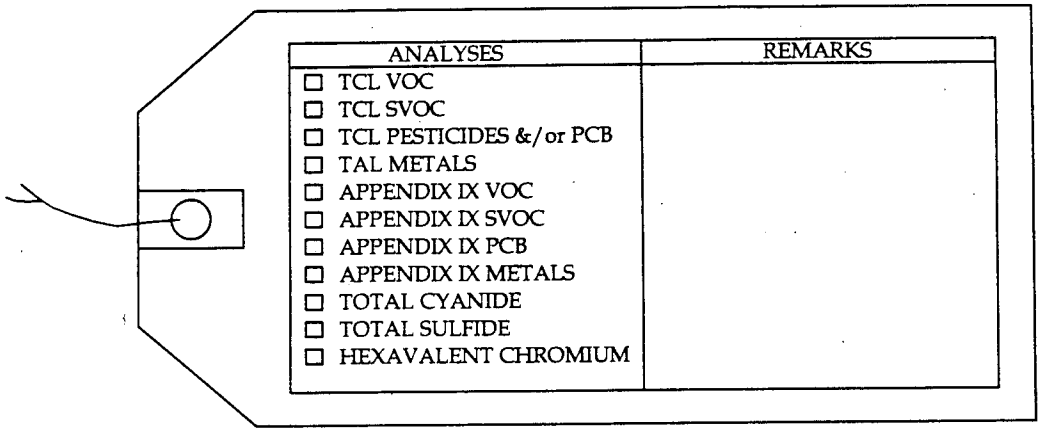
figure ATT-1.1

EXAMPLES OF SAMPLE LABEL AND CUSTODY TAPE
FORMER PEREGRINE (US) , INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

SAMPLE TAG



FRONT



BACK

NOTES

1) SAMPLE TAG TO BE AFFIXED TO SAMPLE CONTAINER TOP (BELOW CAP) WITH WIRE.

2) ALL SAMPLE TAGS TO BE COMPLETED USING WATER INSOLUABLE INK.

figure ATT-1.2

EXAMPLE SAMPLE TAG
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

CRA
CONESTOGA–ROVERS & ASSOCIATES, INC.
11100 Metro Airport Center Drive – Suite 160
Romulus, MI 48174 (734)942–0909

CHAIN OF CUSTODY RECORD

SHIPPED TO (Laboratory Name):

REFERENCE NUMBER:

PROJECT NAME:

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:

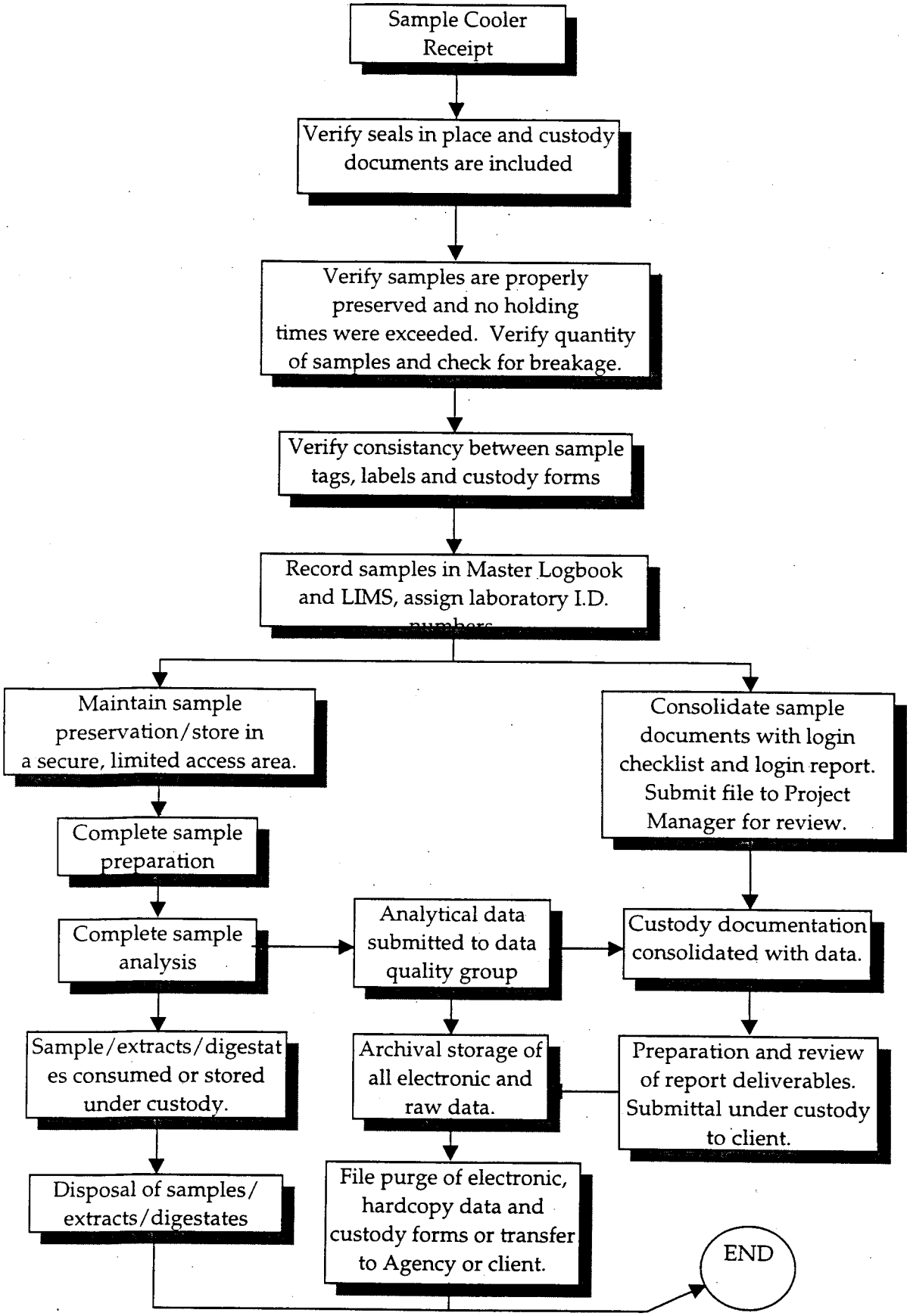
RECEIVED FOR LABORATORY BY:

DATE:_____ TIME:_____

figure ATT-1.3

EXAMPLE OF CHAIN-OF-CUSTODY RECORD
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

FIGURE ATT-1.4
STL-NC CUSTODY SEQUENCE



ATTACHMENT 1.5

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?

_____

_____

_____

ATTACHMENT 1.5

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?

_____

_____

_____

ATTACHMENT 1.5

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.

ATTACHMENT 1.5

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 1.6

LABORATORY AUDIT CHECKLIST

The attached laboratory audit checklist is designed for the completion of an on-site laboratory audit primarily in support of an ongoing Conestoga-Rovers & Associates (CRA) project or Master Agreement. The checklist consists of a number of charts that will be used to audit the laboratory against the project Quality Assurance Project Plan (QAPP) standard operating procedures (SOP) and/or standard U.S.EPA Methods. Only those checklists that pertain to the project or program of interest shall be covered during the on-site laboratory audit.

The laboratory audit checklist covers the 12 sections detailed below:

1.0 General Laboratory Information

2.0 Personnel

3.0 Laboratory Environment/Facilities

4.0 Equipment and Instrumentation

5.0 Field Sampling

6.0 Sample Receipt and Storage

7.0 Organic Analyses by Gas Chromatography (GC)

8.0 Organic Analyses by GC/Mass Spectrometry

9.0 Metals Analysis

10.0 Other Classical Analyses

11.0 Data Reduction, Review and Reporting

12.0 Performance and System Audits

The attached charts (CHARTS I – BB) contain lists on which the availability of written operating procedures is to be checked. These lists cover instrument maintenance and calibration, all aspects of sampling, and the quality control program of the laboratory. The laboratory should be asked to show supporting documents to the auditor(s) during the on-site inspection and to discuss them with the auditor(s).

These checklists are not a demand or request for written procedures (for example, a Quality Control Program) in a particular standard format. The key point is that the principal laboratory controls should be documented in a permanent way. Some procedures may be brief or may not include all of the items to be checked.

GENERAL LABORATORY INFORMATION

1. Laboratory Name: _____

2. Street Address: _____

   _____

   Mailing Address: _____

   _____

3. Telephone No.: _____ FAX No.: _____

4. Number of Employees: _____ Years in Operation: _____

5. Name of Laboratory Director: _____

   Name of Laboratory Project Manager: _____

   Name of Quality Assurance (QA) Officer: _____

6. Project Details:

   Project Number: _____ Project Name: _____

   Laboratory analyses required for project or program (attach pages if necessary)

Parameters	Matrices	Methods

Completed by: _____ _____ Date: _____
NAME TITLE

1.0 AUDIT CHECKLIST – GENERAL LABORATORY INFORMATION

CHART I

ITEM	YES	NO	COMMENT
Does the lab maintain a Quality Assurance Manual?	___	___	_____
Does the manual address the important elements of a QC program, including the following:			
a. Project Organization and Responsibility?	___	___	_____
b. Organization Chart?	___	___	_____
c. Personnel Training/Certification?	___	___	_____
d. Facilities and Equipment?	___	___	_____
e. Quality Assurance Objectives?	___	___	_____
f. Sampling Procedures?	___	___	_____
g. Sample Custody?	___	___	_____
h. Sample Receiving and Disposal?	___	___	_____
i. Analytical Procedures?	___	___	_____
j. Calibration Procedures and Frequency?	___	___	_____
k. Preventive Maintenance?	___	___	_____
l. Certification of Reagents and Standards?	___	___	_____
m. Internal Quality Control Checks?	___	___	_____
n. Data Reduction, Review and Reporting?	___	___	_____
o. Reliability of Data?	___	___	_____
p. Feedback and Corrective Action?	___	___	_____
q. Recordkeeping and Storage?	___	___	_____
r. Performance and System Audits?	___	___	_____
s. Quality Control Reports?	___	___	_____

1.0 AUDIT CHECKLIST – GENERAL LABORATORY INFORMATION (Continued)

CHART I

ITEM	YES	NO	COMMENT
Is the manual available to all laboratory personnel?	___	___	_____
Is the manual updated regularly with appropriate document control procedures?	___	___	_____
Is line authority for all referenced responsibilities explained by inclusion in an organization chart(s)?	___	___	_____
Is the organizational structure appropriate to accomplish the project QA objectives?	___	___	_____
Are QA/QC responsibilities and reporting relationships clearly defined?	___	___	_____
Are all staff aware of QA/QC and its application?	___	___	_____
Is the QA Officer a full-time function?	___	___	_____
Does the QA Officer operate independently of the analyses?	___	___	_____
Does the QA Officer report directly to senior management?	___	___	_____
Are internal QA reviews conducted at least annually and recorded including any corrective action taken?	___	___	_____
QA Objectives and Criteria:			
a. Are the terms and definitions for precision, accuracy, comparability, representativeness and completeness properly used?	___	___	_____
b. Have the following been defined for each matrix and parameter			
1) Level of QA effort (frequency of QC, etc.)?	___	___	_____

CHART I

ITEM	YES	NO	COMMENT
2) Accuracy (matrix spikes, surrogate spikes, reference samples, etc.)?	___	___	_____
3) Precision (replicate samples)?	___	___	_____
4) Sensitivity or MDL?	___	___	_____
5) Statistical reporting limits?	___	___	_____
c. Are quantitation limits established for each analytical parameter?	___	___	_____
d. Are field and lab analyses both covered?	___	___	_____
e. If appropriate, are completeness objectives quantitatively stated?	___	___	_____
f. Are representativeness and comparability appropriately addressed?	___	___	_____
Do SOPs include or refer to SOPs addressing the following areas:			
a. Interferences?	___	___	_____
b. Safety Considerations?	___	___	_____
c. Apparatus and Equipment?	___	___	_____
d. Reagents and Supplies?	___	___	_____
e. Sample Preservation and Storage?	___	___	_____
f. Sample Preparation?	___	___	_____
g. Instrument Calibration?	___	___	_____
h. Quality Control Procedures?	___	___	_____
i. Detailed Step-by-Step Procedure?	___	___	_____
j. Sample Calculations?	___	___	_____

1.0 AUDIT CHECKLIST – GENERAL LABORATORY INFORMATION (Continued)

CHART I

ITEM	YES	NO	COMMENT
k. Method Performance Criteria (accuracy and precision)	___	___	_____
Are there SOPs for:			
a. Sample collection, preservation, reception storage and handling?	___	___	_____
b. Documenting all analytical procedures?	___	___	_____
c. Operation/calibration of instrument?	___	___	_____
d. Preventive maintenance?	___	___	_____
e. Quality Control for each type of test?	___	___	_____
f. Quality Control Charting?	___	___	_____
g. Recordkeeping and storage?	___	___	_____
h. Data reduction, review and reporting?	___	___	_____
i. Personnel training/certification?	___	___	_____
j. Procurement and inventory procedures?	___	___	_____
k. Glassware cleaning?	___	___	_____
l. Waste disposal?	___	___	_____
Is at least the following minimum QC practiced in the lab?	___	___	_____
a. For Inorganic/Wet Chemistry:	___	___	_____
1) Minimum 3 concentrations of standards plus blank, and one check standard in10; the lab shall repeat all samples if check standard is outside ±10%. (±20% for mercury.)	___	___	_____

1.0 AUDIT CHECKLIST – GENERAL LABORATORY INFORMATION (Continued)

CHART I

ITEM	YES	NO	COMMENT
2) One method blank in 20 or per batch.	___	___	_____
3) One matrix spike in 20 or per batch.	___	___	_____
4) One lab duplicate or matrix spike duplicate in 20 or per batch.	___	___	_____
5) One laboratory control sample (LCS) (consists of a control matrix spiked with analytes representative of the target analytes) in 20 or per batch.	___	___	_____
b. For Organic Chemistry:			
1) Minimum 5 concentrations of standards plus blank and one check standard in10; if any are outside control limits repeat all samples.	___	___	_____
2) One method blank in 20 or per batch.	___	___	_____
3) One matrix spike in 20 or per batch.	___	___	_____
4) One lab duplicate or matrix spike duplicate in 20 or per batch.	___	___	_____
5) Surrogates for all samples.	___	___	_____
Is a QC batch defined as a single event (i.e. one day or less)?	___	___	_____
Are there any exceptions to the above minimum QC practiced in the lab?	___	___	_____
Has the lab established control limits for all the above types of QC samples?	___	___	_____
Are control limits at least as tight as those stated in the methods?	___	___	_____

1.0 AUDIT CHECKLIST – GENERAL LABORATORY INFORMATION (Continued)

CHART I

ITEM	YES	NO	COMMENT
Are quality control data (e.g., standard curve, results of duplicates and spikes) accessible for all analytical results?	___	___	_____
Are method detection limits determined and documented?	___	___	_____
Are control charts maintained for each routine analysis?	___	___	_____
Do lab records indicate what corrective action has been taken when results fail to meet QC criteria?	___	___	_____
Is a program of initial and continuing calibration established for each method?	___	___	_____
Does QA Plan include calibration documentation requirements:	___	___	_____
a. Date(s) of calibration?	___	___	_____
b. Identification of standards used?	___	___	_____
c. Personnel performing calibration?	___	___	_____
d. Results of calibration (raw data and summary statistics)?	___	___	_____
e. Corrective actions taken?	___	___	_____
Are primary reference standards used for calibration only?	___	___	_____
Are all working standards versus primary standards verified and documented?	___	___	_____
Are reference materials (lab standards) traceable?	___	___	_____
Are reagent grade or higher purity chemicals used to prepare standards?	___	___	_____

CHART I

ITEM	YES	NO	COMMENT
Are fresh analytical standards prepared at a frequency consistent with good QC?	___	___	_____
Are reference materials/reagents properly labeled with concentrations, dates of receipt, preparation/expiration and the identity of the person preparing the reagent?	___	___	_____
Are updated equipment operating instructions available?	___	___	_____
Are all analytical procedures written as SOPs available for review?	___	___	_____
Are SOPs provided and maintained in the laboratory or area of use?	___	___	_____
Are all procedural steps and options described in SOPs?	___	___	_____
Are procedures in place for making and controlling revisions to SOPs?	___	___	_____
Are the criteria of method selection included (e.g., in order to obtain a specific Data Quality Objective?)	___	___	_____
If method choice is governed by regulatory requirement (e.g., NPDES, SDWA, RCRA) have the appropriate methods been chosen?	___	___	_____
Are approved methods being used as specified?	___	___	_____
Are procedures documented for data handling, reporting and record keeping?	___	___	_____
Are documented validation procedures applied at appropriate levels of all measurement procedures?	___	___	_____

1.0 AUDIT CHECKLIST – GENERAL LABORATORY INFORMATION (Continued)

CHART I

ITEM	YES	NO	COMMENT
Are documented procedures available for checking the validity of reported analysis values?	___	___	_____
Are predetermined limits available for data acceptability beyond which corrective action is required?	___	___	_____
Are documented procedures available for correcting erroneously reported results?	___	___	_____
Does the lab have a LIMS currently in use? If yes, manufacturer.: _____ Model No.: _____	___	___	_____

Brief Description:_____

_____

_____

_____

_____

_____

Does the LIMS have an audit trail feature?	___	___	_____

Completed by: _____ _____ Date: _____
NAME TITLE

2.0 AUDIT CHECKLIST – PERSONNEL:

CHART J

ITEM	YES	NO	COMMENT
Are there documented protocol for training in quality control methods?	___	___	_____
Do personnel assigned to this project have the appropriate background to successfully accomplish the objectives of this project?	___	___	_____
Does each analyst accountable for performing tasks in any of the following areas meet the specified minimum experience:	___	___	_____
a. General chemistry and instrumentation — 6 months.	___	___	_____
b. Sample preparation — 6 months.	___	___	_____
c. Gas chromatography — 1 year.	___	___	_____
d. Mass spectrometry — 1 year.	___	___	_____
e. Atomic absorption — 1 year.	___	___	_____
f. Atomic emission — 1 year.	___	___	_____
g. Spectral/chromatography interpretation — 2 years.	___	___	_____
h. Pesticide residue analysis — 2 years.	___	___	_____
Is backup provided for technical staff?	___	___	_____
Are qualifications, training and experience recorded?	___	___	_____
Is there documented evidence of analyst proficiency for each test method performed?	___	___	_____
Is the QA Officer familiar with all test procedures and QC requirements?	___	___	_____
Does the lab have an in-house training program or send staff to training school?	___	___	_____

Completed by: _____ _____ Date: _____
NAME TITLE

3.0 AUDIT CHECKLIST – LABORATORY ENVIRONMENT/FACILITIES:

CHART K

ITEM	YES	NO	COMMENT
Does the lab building have a security system?	___	___	_____
Is access to the laboratory area controlled?	___	___	_____
Is equipment protected and environment monitored as needed?	___	___	_____
Does the lab have adequate work space, ventilation, light and access to stable power sources at workstations?	___	___	_____
Is the lab free of dust, drifts and temperature extremes?	___	___	_____
Is water free of trace contamination available for preparation of standards and blanks?	___	___	_____
Is the pH and conductivity of water routinely checked and recorded on a daily basis?	___	___	_____
Are exhaust hoods provided to allow contamination-free work with volatile and hazardous materials?	___	___	_____
Is the air flow of the hoods periodically checked and recorded?	___	___	_____
Are adequate facilities, including cold storage, provided for separate storage of samples, extracts, reagents, solvents, reference materials and standards to preserve their identity, concentration, purity and stability?	___	___	_____
Is adequate chemical storage space available and are chemicals properly segregated according to hazard class?	___	___	_____
Are solvent storage cabinets properly vented as appropriate for the prevention of possible lab contamination?	___	___	_____

CHART K

ITEM	YES	NO	COMMENT
Does the lab have adequate safety devices such as eye wash stations, spill control stations, showers, first-aid stations, etc.)?	___	___	_____
Are these safety devices checked routinely and documented to ensure they are still working properly?	___	___	_____
Are special facilities provided for handling extremely toxic materials such as dioxin (e.g. glove box, controlled air)?	___	___	_____
Are chemical waste disposal policies and procedures well-defined and followed by the lab?	___	___	_____

Completed by: _____ _____ Date: _____
NAME TITLE

4.0 AUDIT CHECKLIST – EQUIPMENT AND INSTRUMENTATION:

CHART L

ITEM	YES	NO	COMMENT
Is appropriate equipment/instrumentation available for use in accordance with required methodology?	___	___	_____
Is equipment adequately maintained with sufficient spare parts and are maintenance instructions available?	___	___	_____
Is out-of-service equipment clearly labeled?	___	___	_____
Are instrument maintenance logs kept?	___	___	_____
Are standard curves prepared to adequately cover the expected concentration ranges of samples?	___	___	_____
Are calibration logs maintained?	___	___	_____
Is a new curve prepared annually (or more frequently as specified by the method) or whenever new reagents are prepared, whichever is more frequent?	___	___	_____
Are calibration labels used as applicable?	___	___	_____
Is proper backup equipment available?	___	___	_____
General Equipment Requirements:			
Analytical and Pan Balances:	___	___	_____
a. Are analytical balances capable of weighing 0.1 mg in use?	___	___	_____
b. Is a record of balance calibration kept for at least two ranges with Class S weights? (Please specify the ranges.)	___	___	_____
c. Do records shows daily functional and calibration checks for analytical balances and weekly for pan balances?	___	___	_____

CHART L

ITEM	YES	NO	COMMENT
d. Are the balances serviced and calibrated at least annually?	___	___	_____
pH Meter:			
a. Is a clean pH meter and appropriate electrode with scale graduations at least 0.1 pH units in use?	___	___	_____
b. Is a thermometer or a temperature sensor for automatic compensation in use?	___	___	_____
c. Do records show daily, or before each use, calibration, whichever is less frequent?	___	___	_____
e. Are aliquots of standard pH 4 & pH 7, or pH 7 & pH 10 used only once?	___	___	_____
Conductivity Meter:			
a. Are a conductivity meter and probe of sufficient sensitivity in use?	___	___	_____
b. Do records show a daily, or before each use, calibration check, whichever is less frequent?	___	___	_____
c. Do records show cell constant is determined annually?	___	___	_____
Glassware:			
a. Is glassware cleaned in a manner appropriate for the analytical procedures for which it is used?	___	___	_____
b. Are glassware cleaning SOPs available where cleaning occurs?	___	___	_____
c. Is glassware maintained separately for each department?	___	___	_____

4.0 AUDIT CHECKLIST – EQUIPMENT AND INSTRUMENTATION: (continued)

CHART L

ITEM	YES	NO	COMMENT
Refrigeration:			
a. Is a thermometer in each refrigerator with bulb immersed in liquid?	___	___	_____
b. Are thermometers graduated in increments no larger than 1°C?	___	___	_____
c. Are thermometers calibrated against NIST (NBS) - traceable thermometer and documented?	___	___	_____
d. Do records show that refrigerator temperatures are maintained in the range of 2° to 6°C?	___	___	_____
e. Are temperatures for each refrigerator recorded daily?	___	___	_____
f. Are samples stored in separate refrigerators from standards?	___	___	_____
Ovens:			
a. Are thermometers graduated in increments no larger than 1°C?	___	___	_____
b. Are thermometers calibrated against NIST (NBS) - traceable thermometer and documented?	___	___	_____
c. If the oven temperature cannot be read without opening the door, is the bulb of the thermometer in a sand bath?	___	___	_____
d. Is oven temperature adequately monitored (e.g., beginning and end of each use cycle)?	___	___	_____
Thermometers:			
a. Does lab have access to a NIST (NBS) traceable thermometer?	___	___	_____

4.0 AUDIT CHECKLIST – EQUIPMENT AND INSTRUMENTATION: (continued)

CHART L

ITEM	YES	NO	COMMENT
b. Is the calibration of each working mercury-in-glass thermometer checked at least annually against a NIST(NBS) treaceable certified thermometer?	___	___	_____
c. Is the calibration of each dial-type thermometers checked at least quarterly against a NIST (NBS)-traceable thermometer?	___	___	_____

Completed by: _____ _____ Date: _____
NAME TITLE

5.0 AUDIT CHECKLIST - FIELD SAMPLING: (Complete if this lab will conduct field sampling for CRA.)

CHART M

ITEM	YES	NO	COMMENT
Is the Site Specific Quality Assurance Project Plan (QAPP) available to field personnel?	___	___	_____
Are field personnel familiar with the QC requirements of the Site specific QAPP?	___	___	_____
Do field documentation procedures:			
a. Document source of reagents/supplies?	___	___	_____
b. Include procedures/forms for recording the exact location and specific considerations associated with sample collection?	___	___	_____
c. Document specific preservative methods?	___	___	_____
d. Include labels containing all necessary information?	___	___	_____
e. Include forms for tracking sample custody?	___	___	_____
Do sampling procedures follow contract specifications?	___	___	_____
Is there a unique identification number on each sample?	___	___	_____
Is sampling information properly recorded such as sample ID, type (grab vs composite), preservatives, analytes, location, date and time of collection and name of sampler?	___	___	_____
Are written chain-of-custody procedures available for review? Are they in accordance with EPA and QAPP guidelines?	___	___	_____

5.0 AUDIT CHECKLIST - FIELD SAMPLING (continued)

CHART M

ITEM	YES	NO	COMMENT
Are there written sampling procedures covering sampling plans, sampling equipment, sample collection, preservation, identification, storage and lab handling?	___	___	_____
Are there written descriptions of the chain-of-custody of samples? (Attach a copy of chain-of-custody form.)	___	___	_____
Are there written procedures for field measurement of flow, dissolved oxygen, residual chlorine, etc.	___	___	_____
Are there written SOPs for monitoring water supply, effluent, ambient air, stacks, mobile vehicles, pesticides, radiation, etc.?	___	___	_____
Are proper preservation techniques being used for the analytical methods and sample types concerned?	___	___	_____
Are provisions made for the collection of QA/QC split samples?	___	___	_____
Are provisions made for field blanks and duplicate samples at an appropriate percentage (normally 10% or minimum of one per matrix type, whichever is greater, or as specified in Contract?)	___	___	_____
Are adequate facilities available to do compatibility testing?	___	___	_____

Completed by: _____ _____ Date: _____
NAME TITLE

6.0 AUDIT CHECKLIST - SAMPLE RECEIPT/STORAGE:

CHART N

ITEM	YES	NO	COMMENT
Are appropriate chain-of-custody procedures documented and followed in the lab?	___	___	_____
Are there adequate written procedures for receipt and storage of samples to ensure that holding times are met?	___	___	_____
Do lab custody procedures: a. Identify sample custodian? Name of custodian: _____	___	___	_____
b. Provide for custody record within the lab?	___	___	_____
c. Specify procedures for sample handling, storage, dispersement for analysis, and disposal?	___	___	_____
Does a permanent record exist for sample log-in?	___	___	_____
Are samples assigned an unambiguous sample number when logged in?	___	___	_____
Are there sample preservation records?	___	___	_____
Are the pH values of aqueous samples checked and adjusted in a hood for trace metals, phenols, cyanide and TRPH during sample log-in?	___	___	_____
Is the temperature of sample cooler/shuttle recorded upon receipt?	___	___	_____
Is client notified of receipt temperature outside of 4° ± 2°C?	___	___	_____
Are discrepencies between sample labels and chain-of-custody documents recorded?	___	___	_____
Is client notified of any discrepancies following sample receipt and log-in?	___	___	_____

6.0 AUDIT CHECKLIST - SAMPLE RECEIPT/STORAGE: (continued)

CHART N

ITEM	YES	NO	COMMENT
Are there adequate facilities for sample receipt and storage of samples?	___	___	_____
Are samples stored in such a way as to maintain their identity, integrity, stability, and concentration?	___	___	_____
How long will the lab store samples before disposing?	___	___	_____

Completed by: _____ _____ Date: _____

NAME TITLE

7.0 AUDIT CHECKLIST - ORGANIC ANALYSES BY GAS CHROMATOGRAPHY (GC):

A. Summary of GC Staff:

CHART O

Position Title	Name of Employee	Years of Experience[1]	Education in Chemistry	Appropriate Training in Analytical Testing Area[2]
GC Group Leader[3] (Experience: 2 years minimum required)				
GC Operators[3] (Experience: 1 year minimum required)				
GC Interpretation Experts[3] (Experience: 2 years minimum required)				
PCB and Pesticide Residue Analysis Experts[3] (Experience: 2 years minimum required)				
Purge & Trap Experts[3] (Experience: 1 year minimum required)				

7.0 AUDIT CHECKLIST - ORGANIC ANALYSES BY GC: (continued)

CHART O

Position Title	Name of Employee	Years of Experience[1]	Education in Chemistry	Appropriate Training in Analytical Testing Area[2]
Extraction and Concentration Experts[4] (Experience: 6 months minimum required)				

1. Requirements for experience as listed are minimal and may be increased for specific projects involving difficult samples or unusual matrices.
2. Manufacturer sponsored class, EPA symposiums, etc.
3. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.
4. Minimum of high school diploma and knowledge of general chemistry.

7.0. AUDIT CHECKLIST - ORGANIC ANALYSES BY GC: (continued)

B. Summary of GC Instrumentation:

CHART P

No.	Manufacturer	Model Number	Year Acquired	Used for Method[1]	Comments[2]	CRA Use Only S	CRA Use Only U
1							
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15							
16							
17							
18							
19							
20							

1 State EPA method from SW-846 8000 or 40 CFR 136 600 series.
2 Detectors, condition, autosamplers, modifications, etc.

7.0. AUDIT CHECKLIST - ORGANIC ANALYSES BY GC: (continued)

C. Questions about the GC instruments described above. (If one answer does not apply to all of the instruments listed, use the instrument number (1-20) to designate answers as appropriate.

CHART Q

ITEM	YES	NO	COMMENT
Are the following detectors available and are they used appropriately?			
a. Flame ionization (603, 604, 609, 8015, 8030, 8040, 8060, 8090, 8100)?	___	___	_____
b. Photoionization (602, 8020)?	___	___	_____
c. Electron capture (604, 606, 608, 609, 612, 8040, 8060, 8080, 8090, 8120, 8150)?	___	___	_____
d. Electrolytic conductivity (601, 611, 8010, 8080, 8140)?	___	___	_____
e. Microcoulometric (601, 611, 8140)?	___	___	_____
f. Thermal Energy Analyzer (607)?	___	___	_____
g. Nitrogen/Phosphorus (607, 8140)?	___	___	_____
h. Flame photometric (8140)?	___	___	_____
Are the column ovens, at a minimum, capable of control within +0.2° C to 220° C?	___	___	_____
Are the injection ports glass lined?	___	___	_____
Are purge and trap systems subjected to periodic bake-out and cleaning process and are these actions documented?	___	___	_____
Will the system accept 5 mL samples?	___	___	_____
Is the gaseous headspace less than 15 mL?	___	___	_____
Is the trap a minimum of 25 cm long?	___	___	_____
Will the system accept 25 mL samples?	___	___	_____

7.0. AUDIT CHECKLIST - ORGANIC ANALYSES BY GC: (continued)

CHART Q

ITEM	YES	NO	COMMENT
Are appropriate confirmation columns or GC/MS analyses in use?	___	___	_____
Is methanol purge and trap quality or equivalent?	___	___	_____
Is calibration by internal standards?	___	___	_____
If not, is calibration by external standards?	___	___	_____
Is the calibration curve or calibration factor verified every 10 samples?	___	___	_____
Is there a calibration protocol available to the operator?	___	___	_____
Are calibration results kept in a permanent logbook?	___	___	_____
Are SOPs available for each analytical method?	___	___	_____
Are 5-point calibrations used?	___	___	_____
Are surrogate recoveries determined for each sample?	___	___	_____
Is corrective action taken if surrogate recoveries exceed QC limits?	___	___	_____
Does laboratory use EPA CLP protocols to evaluate internal QC data?	___	___	_____
If not, what are the laboratory's criteria for evaluation of internal QC data?	___	___	_____
Are EPA check samples analyzed as appropriate?	___	___	_____
Is a matrix spike and a matrix spike duplicate run with each batch?	___	___	_____

7.0. AUDIT CHECKLIST - ORGANIC ANALYSES BY GC: (continued)

CHART Q

ITEM	YES	NO	COMMENT
Is corrective action taken if matrix spike recoveries exceed QC limits?	___	___	_____
Are method blanks run with each batch?	___	___	_____
Has method proficiency (acceptable accuracy and precision) been established for all procedures?	___	___	_____
Are method matrix-specific accuracy criteria established and maintained?	___	___	_____
Have laboratory-specific surrogate percent recovery QC limits been established?	___	___	_____
Are manufacturer's operating manuals readily available to the operator?	___	___	_____
Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?	___	___	_____
Have any instruments been modified in any way?	___	___	_____
Are the instruments properly vented?	___	___	_____

Completed by: _____ _____ Date: _____

NAME TITLE

8.0 AUDIT CHECKLIST - ORGANIC ANALYSES BY GC/MASS SPECTROMETRY

A. Summary of GC/MS Staff:

CHART R

Position Title	Name of Employee	Years of Experience[1]	Education in Chemistry	Appropriate Training in Analytical Testing Area[2]
GC/MS Group Leader[3] (Experience: 2 years minimum required)				
GC/MS Operators[3] (Experience: 1 year minimum required)				
GC/MS Spectral Interpretation Experts[3] (Experience: 2 years minimum required)				
Purge & Trap Experts[3] (Experience: 1 year minimum required)				

8.0 AUDIT CHECKLIST - ORGANIC ANALYSES BY GC/MS: (continued)

CHART R

Position Title	Name of Employee	Years of Experience[1]	Education in Chemistry	Appropriate Training in Analytical Testing Area[2]
Extraction and concentration Experts[4] (Experience: 6 months minimum required)				

1. Requirements for experience as listed are minimal and may be increased for specific projects involving difficult
2. Manufacturer sponsored class, EPA symposiums, etc.
3. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.
4. Minimum of high school diploma and knowledge of general chemistry.

8.0 AUDIT CHECKLIST - ORGANIC ANALYSES BY GC/MS: (continued)

B. Summary of GC/MS Instrumentation:

CHART S

No.	Manufacturer	Model Number	Year Acquired	Used for Method[1]	Comments[2]	CRA Use Only S	CRA Use Only U
1							
2							
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11							
12							
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14							
15							
16							
17							
18							
19							
20							

1 State EPA methods from SW-846 8000 or 40 CFR 136 600/1600 series.
2 Condition, autosamplers, modifications, etc.

8.0 AUDIT CHECKLIST - ORGANIC ANALYSES BY GC/MS: (continued)

C. Questions about GC/MS instruments described above. (If one answer does not apply to all of the instruments listed, use the instrument number (1-20) to designate answers as appropriate

CHART T

ITEM	YES	NO	COMMENT
Are any GCs not programmable? If so, list the instrument number?	___	___	_____
Is a split/splitless capillary injection in place?	___	___	_____
Is the MS capable of scanning from 35 to 500 amu every one second or less?	___	___	_____
Is tuning compound, PFTBA, used to verify mass calibration?	___	___	_____
Do the mass spectra of BFP and DFTPP meet all criteria before each batch is run?	___	___	_____
Are standards containing all of the analytes of interest analyzed to verify response factors and update retention time?	___	___	_____
Is glassware heated at 400°C in a muffle furnace after careful cleaning?	___	___	_____
Is glassware sealed and stored in a clean environment?	___	___	_____
Are magnetic media stored in a secure area?	___	___	_____
Are extensive in-house replacement parts available?	___	___	_____
Are manufacturer's operating manuals readily available to the operator?	___	___	_____
Is there a calibration protocol available to the operator?	___	___	_____
Are calibration results kept in a permanent logbook?	___	___	_____
Are SOPs available for each analytical method?	___	___	_____

8.0 AUDIT CHECKLIST - ORGANIC ANALYSES BY GC/MS: (continued)

CHART T

ITEM	YES	NO	COMMENT
Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?	___	___	_____
Has the instrument been modified in any way?	___	___	_____
Are the instruments properly vented?	___	___	_____
Are 5-point calibrations used?	___	___	_____
Are continuing calibration checks done on a 12-hour basis?	___	___	_____
Are system performance response factors checked on a 12-hour basis?	___	___	_____
Are BFB and DFTPP tuning checks done on a 12-hour basis?	___	___	_____
Are DDT, pentachlorophenol and benzidine included in the DFTPP tuning solution?	___	___	_____
Are low-level methods routinely used for environmental soil/sediment samples?	___	___	_____
Is the most recent version of the EPA/NIST Mass Spectral Library available?	___	___	_____
For tentatively identified compounds, are library searches done for the 10 volatile organics and the 20 semivolatile organics of highest concentration?	___	___	_____
Are surrogate recoveries determined for each sample?	___	___	_____
Is a corrective action taken if surrogate recoveries exceed QC limits?	___	___	_____
Is a matrix spike and a matrix spike duplicate run with each batch?	___	___	_____

8.0 AUDIT CHECKLIST - ORGANIC ANALYSES BY GC/MS: (continued)

CHART T

ITEM	YES	NO	COMMENT
Is a corrective action taken if matrix spike recoveries exceed QC limits?	___	___	_____
Is a method blank run with each batch?	___	___	_____
Are samples screened by GC prior to GC/MS analysis?	___	___	_____
Has the laboratory demonstrated initial method proficiency?	___	___	_____
Are method matrix-specific accuracy criteria established and maintained?	___	___	_____
Have laboratory-specific surrogate percent recovery QC limits been established?	___	___	_____

Completed by: _____ _____ Date: _____
NAME TITLE

9.0 AUDIT CHECKLIST - METALS ANALYSIS:

A. Summary of AA/ICP Staff:

CHART U

Position Title	Name of Employee	Years of Experience[1]	Education in chemistry	Appropriate Training in Analytical Testing Area[2]
Metals Group Leader[3] (Experience: 2 years minimum required)				
ICP Spectroscopists[3] (Experience: 1 year minimum required)				
Flameless AA Spectroscopists[3] (Experience: 1 year minimum required)				
Flame and Cold Vapor AA Spectroscopists[4] (Experience: 1 year minimum required)				

CHART U

Position Title	Name of Employee	Years of Experience[1]	Education in Chemistry	Appropriate Training in Analytical Testing Area[2]
Inorganic Sample Preparation Experts[4] (Experience: 6 months minimum required)				
Wet Lab Manager[3] (Experience: 2 years minimum required)				
UV/VIS spectroscopist[3] (Experience: 6 months minimum required)				

1. Requirements for experience as listed are minimal and may be increased for specific projects involving difficult samples or unusual matrices.
2. Manufacturer sponsored class, EPA symposiums, etc.
3. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.
4. Minimum of high school diploma and knowledge of general chemistry.

9.0 AUDIT CHECKLIST - METALS ANALYSIS: (continued)

B. Summary of AA/ICP Instrumentation:

CHART V

No.	Manufacturer	Model Number	Year Acquired	Used for Method[1]	Comments[2]	CRA Use Only S	CRA Use Only U
1							
2							
3							
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11							
12							
13							
14							
15							
16							
17							
18							
19							
20							

1 State EPA method SW-846 6000/7000 or 40 CFR 136 200 series.
2 Condition, autosamplers, modifications, etc.

9.0 AUDIT CHECKLIST - METALS ANALYSIS: (continued)

C. Questions about AA/ICP instruments described above. (If one answer does not apply to all of the instruments listed, use the instrument number (1-20) to designate answers as appropriate

CHART W

ITEM	YES	NO	COMMENT
Atomic absorption spectrophotometer:			
a. Are fuels and oxidants commercial grade?	___	___	_____
b. Is there a filter moisture trap between the air source and the spectrometer?	___	___	_____
c. Is nitrous oxide reagent grade?	___	___	_____
d. Are flash-back arrestors and heaters in use where needed?	___	___	_____
e. Are all lamps dated when received and first put into use?	___	___	_____
f. Are lamps available for all elements analyzed?	___	___	_____
g. Does the AA have a deuterium background correction system?	___	___	_____
h. Does the AA have a Zeeman background correction system?	___	___	_____
i. Does the AA have a Smith-Hieftje background correction system?	___	___	_____
ICP-Atomic emission spectrometer:			
a. Is a background correction technique in use and documented according to sample matrix at least quarterly?	___	___	_____
b. Has the instrument detection limit for each element been established and documented at least annually?	___	___	_____
c. Where required, has the effect of high dissolved solids and/or acid concentration been controlled?	___	___	_____

9.0 AUDIT CHECKLIST - METALS ANALYSIS: (continued)

CHART W

ITEM	YES	NO	COMMENT
d. Has salt build-up on the nebulizer been controlled?	___	___	_____
f. When a new matrix is encountered, is a serial dilution, spike addition, or an alternate method technique in use to eliminate potential interference?	___	___	_____
g. Is the spectrometer equipped with an argon gas supply?	___	___	_____
h. Are ultra high purity grade nitric acid, hydrochloric acid and deionized or distilled water used for sample processing and preparation?	___	___	_____
Are manufacturer's operating manuals readily available to the operator?	___	___	_____
Is there a calibration protocol available to the operator?	___	___	_____
Are calibration results kept in permanent logbooks?	___	___	_____
Are SOPs available for each analytical method?	___	___	_____
Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?	___	___	_____
Is ICP calibration checked using a blank and three standards prior to sample analysis?	___	___	_____
Is ICP calibration verified every 10 samples and at the end of the analytical run, using a calibration blank and a check standard?	___	___	_____
Does the result of check standard agree within ±10% of expected value?	___	___	_____

9.0 AUDIT CHECKLIST - METALS ANALYSIS: (continued)

CHART W

ITEM	YES	NO	COMMENT
Are interelement and background correction factors checked at the beginning and end of an analytical run or twice during every 8-hour work shift, whichever is more frequent?	___	___	_____
Does the result of interelement check sample agree within ±20% of expected value?	___	___	_____
Are AA calibrated with a blank and three standards (five standards for mercury)?	___	___	_____
Is the AA calibration verified with a reference standard to within ± 10% of expected value prior to sample analysis?	___	___	_____
Is the AA calibration verified every tenth sample with a mid-range of reference standard?	___	___	_____
Does the results of these check standards agree within ± 20% of expected value?	___	___	_____
Is a matrix spike duplicate or internal QC duplicate run with each batch?	___	___	_____
Is corrective action taken if matrix spike recoveries exceed QC limits?	___	___	_____
Is a corrective action taken if internal QC duplicate or MS/MSD RPDs exceed QC limits?	___	___	_____
Is an analytical (post-digestion) spike analyzed after each sample?	___	___	_____
Is the method of standard addition in use where needed?	___	___	_____
Has the instrument been modified in any way?	___	___	_____
Are the instruments properly vented?	___	___	_____

Completed by: _____ _____ Date: _____
NAME TITLE

10.0 AUDIT CHECKLIST - OTHER CLASSICAL ANALYSES:

A. Summary of Classical Analysis Staff:

CHART X

Position Title	Name of Employee	Years of Experience[1]	Education in Chemistry	Appropriate Training in Analytical Testing Area[2]
Cyanide Analyst[4] (Experience: 6 months minimum required)	_____	_____	_____	_____
	_____	_____	_____	_____
IR Spectroscopists[3] (Experience: 6 months minimum required)	_____	_____	_____	_____
	_____	_____	_____	_____
TPH Analyst[3](418.1) (Experience: 6 months minimum required)	_____	_____	_____	_____
	_____	_____	_____	_____
HPLC Analysis Experts[3] (Experience: 1 year minimum required)	_____	_____	_____	_____
	_____	_____	_____	_____
Explosives Analyst[3]	_____	_____	_____	_____
Ion Chromatographic Analysis Experts (Experience: 1 year minimum required)	_____	_____	_____	_____ _____
	_____	_____	_____	_____ _____

10.0 AUDIT CHECKLIST - OTHER CLASSICAL ANALYSES: (continued)

CHART X

Position Title	Name of Employee	Years of[1] Experience	Education in Chemistry	Appropriate Training in[2] Analytical Testing Area
Common Ions Analyst[3] Radionuclides Analysts (Experience: 2 years minimum required)				
Microbiological Analysis Experts (Experience: 1 year minimum required)				

10.0 AUDIT CHECKLIST - OTHER CLASSICAL ANALYSES: (continued)

Other Classical Lab Analysis Experts: (Experience: 6 months minimum required) Please specify position title and analysis.

CHART X

Position Title	Name of Employee	Years of[1] Experience	Education in Chemistry	Appropriate Training in[2] Analytical Testing Area

1. Requirements for experience as listed are minimal and may be increased for specific projects involving difficult samples or unusual matrices.
2. Manufacturer sponsored class, EPA symposiums, etc.
3. Minimum of Bachelor's degree in chemistry or any scientific/engineering discipline.

10.0 AUDIT CHECKLIST - OTHER CLASSICAL ANALYSES: (continued)

CHART Y

No.	Instrument/ Manufacturer	Model Number	Year Acquired	Used for Method[1]	Comments[2]	CRA Use Only S	CRA Use Only U
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							

1 General classical wet lab/water quality analysis methods.
2 Condition, automation, modifications, etc.

10.0 AUDIT CHECKLIST - OTHER CLASSICAL ANALYSES: (continued)

C. Questions about instruments described above. (If one answer does not apply to all of the instruments listed, use the instrument number (1-13) to designate answers as appropriate.

CHART Z

ITEM	YES	NO	COMMENT
Is the wavelength accuracy and repeatability of all spectrophotometers checked at several wavelengths for each batch of samples?	___	___	_____
Is photometric accuracy and repeatability checked and documented with NIST (NBS) traceable standards?	___	___	_____
Is acid washed glassware retained for phosphorus analyses only?	___	___	_____
Is ammonia free water used in preparation of standards and samples for nitrogen analyses?	___	___	_____
Are manufacturer's operating manuals readily available to operator of each instrument?	___	___	_____
Is there a calibration protocol available to the operator?	___	___	_____
Are calibration results kept in permanent logbooks?	___	___	_____
Are SOPs available for each analytical method?	___	___	_____
Is a permanent logbook kept for each instrument that summarizes instrument problems and servicing records?	___	___	_____
Has any instrument been modified in any way?	___	___	_____
Are 4-point calibrations used?	___	___	_____
Are continuing calibration checks done on a regular basis?	___	___	_____

10.0 AUDIT CHECKLIST - OTHER CLASSICAL ANALYSES: (continued)

CHART Z

ITEM	YES	NO	COMMENT
Are internal QC run per method requirements?	___	___	_____
Is a method blank used with each batch?	___	___	_____

Completed by: _____ _____ Date: _____
NAME TITLE

11.0 AUDIT CHECKLIST - DATA REDUCTION, REVIEW AND REPORTING:

CHART AA

ITEM	YES	NO	COMMENT
Are there documented procedures for internal field and lab checks of:			
a. Precision/Accuracy?	___	___	_____
b. Routine duplicates, spikes, and standard samples?	___	___	_____
c. Statistical methods, including control chart and computer methods?	___	___	_____
Is there a written description of the lab record system including data handling/calculation review and audit?	___	___	_____
Is there written description of the lab reporting system?	___	___	_____
Is there a system in place that provides for retrievability and traceability of the sample source, analytical methods, results, person performing analysis, and date?	___	___	_____
Are records and reports adequately secured for the required amount of time to ensure their integrity per regulatory or CRA contract requirements?	___	___	_____
Are all lab notebooks when filled, and raw data, QC data, analytical reports, pertinent documents filed in a secure, controlled archives area?	___	___	_____
Are permanently bound notebooks with consecutively numbered pages being used?	___	___	_____
Is a unique serial number clearly displayed on each notebook cover or spine?	___	___	_____
Have entries been made in permanent fashion and corrections made without obliterating original entries?	___	___	_____

11.0 AUDIT CHECKLIST - DATA REDUCTION REVIEW AND REPORTING: (continued)

CHART AA

ITEM	YES	NO	COMMENT
Are notebooks appropriately controlled?	___	___	_____
Has the supervisor of each individual maintaining a notebook personally examined and reviewed each notebook periodically, signed and dated the review?	___	___	_____
Does the lab's reports accurately, clearly, and unambiguously present results and all other relevant information?	___	___	_____
Does each test report include the following information?			
a. Name and address of laboratory?	___	___	_____
b. Unique identification and page number?	___	___	_____
c. Name and address of client?	___	___	_____
d. Case narrative?	___	___	_____
e. Sample identification and description?	___	___	_____
f. Dates of sample receipt and test performed, as appropriate?	___	___	_____
g. Identify of sample preparation and analysis methods used?	___	___	_____
h. Description of any deviations from test method?	___	___	_____
i. Disclosure of any subcontractor used?	___	___	_____
j. Results including all method required QC data?	___	___	_____
k. Description of any problems or failures identified?	__	__	_____
l. Measurement uncertainty, if relevant?	__	__	_____

Completed by: _____ _____ Date: _____
NAME TITLE

12.0 AUDIT CHECKLISTS - PERFORMANCE AND SYSTEM AUDITS:

CHART BB

ITEM	YES	NO	COMMENT
Are there audits for both field and lab activities?	___	___	_____
Are there designated persons who will conduct the audits?	___	___	_____
Is there a documented protocol which will be used for audit?	___	___	_____
Are acceptance criteria defined?	___	___	_____
Are audit reports prepared and distributed? to whom?	___	___	_____
Are the type and frequency of audit reports specified?	___	___	_____
Do the reports address:			
a. Status of project (time table)?	___	___	_____
b. Results of performance and system audits?	___	___	_____
c. Data quality assessment?	___	___	_____
d. Significant QA problems and proposed corrective action?	___	___	_____

Completed by: _____ _____ Date: _____

NAME TITLE

ATTACHMENT 2

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	SOP Title	SOP ID
C.	Inorganics Preparation and Analysis	
i)	Aqueous ICP, FLAA Acid Digestion of Aqueous Samples and Extracts for Total Metals.	CORP-IP-0003
ii)	Acid Digestion of Sediments, Sludges, and Soils for Metal Analysis by GFAA, FLAA, or ICP.	CORP-IP-0002DEN
iii)	Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrophometric Method for Trace Element Analysis, SW-846 Method 6010B and EPA Method 200.7.	CORP-MT-0001
iv)	Inductively Coupled Plasma Mass Spectrometry, EPA Methods 6020 and 200.8	DEN-MT-0002
v)	Preparation and Analysis of Mercury in Aqueous Samples by Cold Vapor Atomic Absorption, SW-846 7470A and MCA WW 245.1.	CORP-MT-0005DEN
vi)	Preparation and Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption Spectroscopy, SW-846 7471A and MCA WW 245.5.	CORP-MT-0007DEN
vii)	Cyanide Preparation Method	NC-WC-0032
viii)	Cyanide Automated, Pyridine-Barbituric Acid Method	NC-WC-0031
ix)	Sulfide	NC-WC-0060
x)	Determination of Hexavalent Chromium (CR VI) in Solid Matrices with Alkaline Digestion by Method 3060A and Colorimetric Determination by Method 7196A	RICH-WC-5005
xi)	Chromium Hexavalent (Colorimetric)	NC-WC-0024
xii)	pH Electrometric Method	NC-WC-0010

ATTACHMENT 2

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	SOP Title	SOP ID
I.	**FIELD SOPs**	
A.	Groundwater Monitoring	
	i) pH/Temperature	PHT-CRA-98
	ii) Specific Conductance	SC-CRA-98
B.	Soil	
	i) Soil VOC Screening	SVS-CRA-98
II.	**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
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, 8081, 8082, and 8151A, SW-846.	CORP-GC-0001NC

ATTACHMENT 2

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D. Laboratory Reference Data Summary

- Laboratory Report Limits, Method Detection Limits and Quality Control Criteria

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.

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 ^{o}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.

QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: SAMPLE RECEIVING AND SAMPLE CONTROL

(SUPERSEDES: REVISION 5, 04/20/99)

Prepared by: _____ 9-16-99
Date

Reviewed by: _____ 9-16-99
Technology Specialist Date

Approved by: _____ 9-20-99
Quality Assurance Manager Date

Approved by: _____ 9-20-99
Environmental Health and Safety Coordinator Date

Approved by: _____ 9-20-99
Laboratory Director Date

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2. RESPONSIBILITIES *3*
3. SAFETY *4*
4. PROCEDURES *4*
5. DEFINITIONS *12*
6. APPENDICES *12*

1. PURPOSE

1.1. 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.

1.2. 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.

1.3. 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 Sample Receiving and Control personnel to perform the procedures 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 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.

2.3. Analyst

2.3.1. 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.

2.4. Sample Custodian

2.4.1. It is the responsibility of the sample custodian to fill all bottle requests in a timely fashion and document the transfers on the request/return forms (Appendix 13.1.6).

2.4.2. It is the responsibility of the sample custodian to correctly document the bottle information required (Appendix 13.1.6).

2.4.3. It is the responsibility of the sample custodian to ensure that the returned bottle is the same as the one relinquished and to return it to the proper storage area.

3. SAFETY

3.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

3.2. 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.

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

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.

3.4.1. The following materials are known to be **corrosive: Sulfuric Acid, Nitric Acid, Hydrochloric Acid, Sodium Hydroxide.**

3.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

3.6. 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.

4. PROCEDURES

4.1. Any deviations from this procedure must be documented as a nonconformance, with a cause and corrective action described.

4.2. The Sample Control person will open and examine the shipping container, remove the enclosed sample documents, and record the following information on the Cooler Receipt/ Narrative Form (Appendix 13.1.1).

4.2.1. Presence of the custody seals on the outside of the cooler

4.2.2. Presence of the custody papers inside the cooler

4.2.3. The custody papers were properly filled out (ink, signed, match labels)

4.2.4. The custody papers were signed in the appropriate place

4.2.5. Presence of the shipper's packing slip

4.2.6. Presence of packing material information: if yes, type of packing material

4.2.7. Conditions of samples at receipt (chilled, etc.).

4.2.7.1. 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 4.2.7.2.

4.2.7.2. The temperature of the coolant is recorded if a temp vial is not present. This is performed by placing the thermometer probe between the coolant and the sample(s). If a coolant is not present, proceed to section 4.2.7.3.

4.2.7.3. If no coolant is in the cooler, the thermometer probe is placed between two sample bottles and the temperature recorded.

4.2.7.4. If the temperature is outside 4°C ± 2°C, the anomaly is recorded on the cooler receipt form. The project manager is contacted for further instructions from the client when the temperature is >6°C.

4.2.8. Bottles sealed in separate plastic bags

4.2.9. Condition of bottles upon receipt (good condition, broken, etc.)

4.2.10. Complete bottle labels (date, time, client ID)

4.2.11. Information on bottle labels and tags agree with custody papers

4.2.12. Correct bottles used for the tests indicated

4.2.13. VOA bottles were checked for the presence of air bubbles. Any bubbles exceeding 6 mm in diameter are narrated and the PM is contacted.

4.2.14. Sufficient amount of sample sent in each bottle

4.2.15. Samples were received via overnight courier, client drop off, or other means

4.2.16. 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.

4.2.16.1. In determining if the sample were 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.

4.2.17. 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 6 mL Sodium hydroxide and 2 mL Zinc acetate. The pH adjustment is noted on the cooler receipt form.

4.2.18. 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%)

4.2.18.1. 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.

4.3. 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.

4.4. 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).

4.5. 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.

4.6. 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 rectify the situation.

4.7. 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.

4.8. 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.

4.9. 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.

4.9.1. 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 - Quanterra 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

4.9.2. 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.

4.9.3. 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.

4.9.4. 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.

4.9.5 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.

4.9.4. 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:

4.9.5. Organic extractable samples (Semivolatiles, Pesticides/PCBs) are to be placed into the walk-in refrigerators located in Sample Receiving.

4.9.6 Volatile samples are to be stored in the double-door refrigerator located in the Sample Custodian area.

4.9.6.1 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.

4.9.7 Inorganic samples are to be placed into the walk-in refrigerators located in Sample Receiving.

4.9.8 Metals samples are placed in a non-refrigerated room located in the Sample Custodian area. Metals samples for Navy projects are stored in the same

condition as received. Samples received cold are stored in the walk-in refrigerator.

5. SAMPLE CONTROL RECORD

5.1. 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.

5.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

5.3. For clients who request a show of sample transfer from sample receipt to storage, a sample control record is completed (see Appendix 13.1.4 or Appendix 13.1.5). Quanterra 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:

5.3.1. 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.

5.3.2. Transferred by - record name of person making the transfer

5.3.3. Date - record date of sample transfer

5.3.4. Entered - "X" or "√" since samples are already logged into the LIMs.

5.3.5. Removed - leave blank since samples are not being removed

5.3.6. Reason - record "storage"

5.3.7. Date returned - leave blank

5.4. The completed sample control record is attached to the summary package.

5.5. 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.

5.6. Each bottle has an associated lot number, sample workorder number, and bottle suffix number (Appendix 13.1.9).

5.7. When an analyst requests bottles and exits the request program, a requisition (Appendix 13.1.6) 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.

5.8. 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 13.1.6).

5.9. 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 13.1.6).

5.10. 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.

5.11. 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.

5.12. When a sample custodian is not available, a designee gets the samples, and all request paperwork is completed properly.

6. WASTE DISPOSAL

6.1. 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 entered into LIMS.

6.2. To enter Disposal date in QuantIMS, enter the following commands; INQ-I01, 1, enter lot number, F9 enter, CMD 5, CMD 3.

6.3. 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 indicated "SAVE" and client name or reason.

6.4. Solid samples that can be disposed of are boxed and labeled "SOLIDS NOW."

6.5. 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 Co-ordinator or designee.

6.6. 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.

7. SUBCONTRACTING OF SAMPLES

7.1. Samples that are logged but not analyzed at the laboratory are subcontracted to different laboratories for analysis including other Quanterra facilities.

7.2. The LIMs system will automatically print a Sample Analysis Requisition for these samples upon completion of the log-in process (Appendix 13.1.7).

7.3. 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.

8. METHOD PERFORMANCE

8.1. Training Qualifications:

8.1.1. 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

9. DEFINITIONS

9.1. Refer to the glossary in the Quality Assurance Management Plan (QAMP)

10. APPENDICES

10.1. Comments

10.1.1. The only personnel authorized to execute this SOP are the Sample Log-In persons.

10.1.2. 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.

11. POLLUTION PREVENTION

11.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

12. WASTE MANAGEMENT

12.1. Not Applicable

13.1.1

Example

Cooler Receipt/Narrative Form

Quanterra® Cooler Receipt Form/Narrative

North Canton Facility

Client:_____ Project:_____ Quote#:_____

Cooler Received on:_____ Opened on:_____ by: _____

Fedx ☐ Client Drop Off ☐ UPS ☐ Airborne ☐ Other:_____ (Signature)

Cooler ☐ Safe ☐ Foam Box ☐ Client Cooler ☐ Other:_____

Quanterra Shipper No#:_____

1. Were custody seals on the outside of the cooler and intact? Yes ☐ No ☐

   If YES, Quantity_____ Location_____

   Were signature and date correct? 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 ☐ Coolant ☐ Against Bottles ☐

COOLANT: Wet Ice ☐ Blue Ice ☐ Dry Ice ☐ 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 present 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:_____

Check √	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.
	SR2D	Sample(s)_____ were received in unapproved containers.
3. SAMPLE PRESERVATION		
√	SR3A	The temperature of the cooler was _____ ° C
	SR3B	Sample(s) _____ received for Volatile analysis was/were received with headspace.
	SR3C	Sample(s) _____ were received incorrectly preserved and were adjusted accordingly in sample receiving.
	SR3D	Sample(s) _____ were received incorrectly preserved and split off in sample receiving.
	SR3E	Samples (s)_____ were received incorrectly preserved and were unable to be analyzed.
	N/A	See back for other anomalies

Quanterra® Cooler Receipt Form/Narrative

North Canton Facility

Client ID	pH	Date	Initials

Cooler	Temp	Method	Comments

Discrepancies Cont.

Macro Name:

Macro Name:

Macro Name:

Other Anomalies:

13.1.2

Preservative Preparation

Preservative Preparation

4N Sodium Hydroxide	Add 12.0 g NaOH pellets to 800 mL reagent water in a 1000 mL volumetric flask. Mix, cool, and dilute to volume with reagent water. Store in a well labeled plastic bottle.
1N Zinc Acetate	Add 55 g $Zn(C_2H_2O_2) \bullet H_2O$ to 200 mL reagent water in a 250 mL volumetric flask. Mix and dilute to volume with reagent water. Store in a well labeled plastic bottle.
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.
1:4 Nitric Acid (18%):	Slowly add 360 mL concentrated HNO_3 to 1640 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.*

13.1.3

PreservativeRequirements

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
Elemental PO_4	G	None	250 mL	VOC 624	G	HCl	8x40 mL
Orthophosphate	P	None	250 mL	BTEX 8020	G	HCl	3x40 mL

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative[1,2]	Volume
TDS	P	None	250 mL
TSS	P	None	250 mL
Total Solids	P	None	250 mL
TVS	P	None	250 mL
T. Coliform	P	None	125 mL
Settleable Solids	P	None	1L
Silica	P	None	250 mL
Sulfate	P	None	250 mL
Sulfite	P	None	250 mL
Surfactants (MBAS)	P	None	250 mL
Turbidity	P	None	250 mL
TPH-GC	G	None	2 L
BNAs	G	None	2 L
BNA + Dioxin	G	None	2 L
PNA/PAH	G	None	2 L
Pesticides	G	None	2 L
Reactive Cyanide	P	None	1 L
Reactive Sulfide	P	None	1 L
PCB	G	None	2 L
Pesticides + PCBs	G	None	2 L
Herbicides	G	None	2 L

Parameter	Container	Preservative [1,2]	Volume
VOC 8240	G	HCl	3x40 mL
THM/502.2	G	HCl	2x40 mL
502.2	G	HCl & Asc. Acid	2x40 mL
VOC 624	G	HCl	3x40 mL
VOC 602	G	HCl	3x40 mL
465 C & D	G	HCl	4x40 mL
BTEX 8021	G	HCl	3x40 mL
VOC	G	HCl	3x40 mL
VOC 8260	G	HCl	3x40 mL
VOC and VOA	G	HCl	3x40 mL
VOC 8010/8020	G	HCl	3x40 mL
Total Cyanide	P	NaOH[3]	250 mL
Amenable Cyanide	P	NaOH	250 mL
Free Cyanide	P	NaOH	250 mL
Sulfide	P	Zn Acetate & NaOH	1 L
Formaldehyde	G	None	500 mL
Carbonate	P	None	250 mL
Bicarbonate	P	None	250 mL
TPH - Diesel (Ext.)	G	None	2 L
TPH - Gasoline (P&T)	G	HCl	2x40 mL
Glycols 8015	G	None	2x40 mL

PRESERVATIVES, CONTAINERS, AND VOLUMES

Parameter	Container	Preservative[1,2]	Volume	Parameter	Container	Preservative [1,2]	Volume
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 anlayzed for Cyanide should be field-filtered for Residual Chlorine. If Residual Chlorine is detected, ascorbic acid (0.6 g) should be added.

13.1.4

Example

Internal Chain of Custody - CLP

Revision 05, February6, 1997

QUANTERRA® ENVIRONMENTAL SERVICES
Sample Control Record

Client: 375241 Case Number/SDG: DL292 Storage Location: C120 MS

A9D170130

	Laboratory Sample Number	Transferred By	Date	Entered	Removed	Reason	Date Returned
1	CTQ0R	lde	4-16-99	X		Storage	
2	CTQ0T	"	"	"		"	
3	CTQ1L	"	"	"		"	
4	CTQ27	"	"	"		"	
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							

13.1.5

Internal Chain of Custody

Regular Production-Organic

Revision 05, February6, 1997

QUANTERRA® ENVIRONMENTAL SERVICES
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						
11						
12.						
13						
14.						
15						
16						
17						
18.						
19.						
20.						

13.1.6

Example

Sample Custodian Removal Request Form

(LIMS Generated)

PSR024 3/26/99 6:50:06 MT SAMPLE CUSTODIAN REMOVAL REQUEST PAGE 001

REQUESTED BY: NACEMCSU

METHOD: VR Carbon, Total Organic "TOC" (WALKLEY) - Solids

STORAGE LOCATION	WORK ORDER #	PICKED CNTR#	CONTROL #	CLIENT #	ANALYSIS	LOTID	SMP#	SFX	MATRIX DESCRIPTION	QTY RCVD	QTY REQD
C43	CRFKH-1-02	1	453501	357464	A-82-VR	A9C090139	016		SOLID	1	1
C43	CRFKQ-1-09	1	453502	357464	A-82-VR	A9C090139	017		SOLID	1	1
C43	CRFKR-1-02	1	453503	357464	A-82-VR	A9C090139	018		SOLID	1	1
C43	CRFKB-1-0U	1	453504	357464	A-82-VR	A9C090139	015		SOLID	1	1
C58 MS OUT	CRKF0-1-05	5	453531	357464	A-82-VR	A9C120230	003		SOLID	7	1
C58 OUT	CRKFC-1-04	2	453530	357464	A-82-VR	A9C120230	004		SOLID	2	1
C65 OUT	CRN6V-1-0X	1	453533	357464	A-82-VR	A9C170101	004		SOLID	2	1
C65 OUT	CRN72-1-0X	1	453534	357464	A-82-VR	A9C170101	005		SOLID	2	1
C65 OUT	CRN73-1-02	1	453535	357464	A-82-VR	A9C170101	006		SOLID	2	1

RELINQUISHED BY	RECEIVED BY	DATE/TIME
PH	SAM	3/26/99
	PH	3/29/99

13.1.7

Example Sample Analysis Requisition

Quanterra Incorporated
SAMPLE ANALYSIS REQUISITION

LABORATORY: Quanterra Inc - Austin TX Lab
5307 Industrial Oaks Blvd Suite 160
Austin TX 78735,EZZ OL

NEED ANALYTICAL REPORT BY
4/11/99

ATTN:

LAB PURCHASE ORDER: SR015374

CLIENT CODE: 39722 PROJECT MANAGER: Kenneth J. Kuzior

NUMBER OF SAMPLES IN LOT: 0004

SAMPLE I.D.	SAMPLING DATE	ANALYSIS REQUIRED
A9C270115-001 CT364-1-0G	3/25/99	Dissolved Gasses in Water (GCH20GAS) METHOD: SOP-175
A9C270115-002 CT36T-1-08	3/25/99	Dissolved Gasses in Water (GCH20GAS) METHOD: SOP-175
A9C270115-003 CT372-1-02	3/25/99	Dissolved Gasses in Water (GCH20GAS) METHOD: SOP-175
A9C270115-004 CT375-1-0G	3/25/99	Dissolved Gasses in Water (GCH20GAS) METHOD: SOP-175

NEED DETECTION LIMIT AND ANALYSIS DATE INCLUDED IN REPORT.

SHIPPING METHOD: AIRBORNE DATE: 3/31/99

SEND REPORT TO: KEN KUZIOR

SAMPLE RECEIVED BY: _____ DATE: _____

PLEASE SEND A SIGNED COPY OF THIS FORM WITH REPORT AT COMPLETION OF ANALYSIS.

THANK YOU.

Quanterra - North Canton
INT: _____ 3/31/99 13:28:24
Quanterra Inc - Austin TX Lab
5307 Industrial Oaks Blvd Suite 160
Austin TX 78735,EZZ OL

RELINQUISHED BY: _____ DATE/TIME: _____

RELINQUISHED BY: _____ DATE/TIME: _____

RECEIVED FOR LAB BY: _____ DATE/TIME: _____

PLEASE RETURN ORIGINAL SAMPLE ANALYSIS REQUISITION

13.1.8

Example Subcontracted PO Logbook

Sub-Work Tracking

Tracking #	Client Code	Lot#	# Samples	Analysis	Lab Sent To	Date Out	Shipment Method	Initials
SR15179	1	A9C170192	1	TPH1005	Austin	3-18-99	airb	LBC
SR15194	000039	A9C180154	4	T,S:Lica	ATEL	3-18-99	Cour	TB
SR15233	⊥	A9C190151	3	⊥	⊥	3-19-99	⊥	⊥
SR15272	11164	A9C220123	1	Long Prox	REAM+ HAGGER	3-19-99	SHIPPED TO LAB by Client	WRC
SR15234	375241	A9C190154	11	Dioxin	Knoxville	3-19-99	airb	ldo
—	375241	A9C190154	20	VOC, bna, metals	Pitt	3-22-99	airb	ldo
SR15239	375241	A9C190158	10	Dioxin	Knoxville	3-19-99	airb	ldo
—	375241	A9C190158	20	voc, bna, metals	Pitt	3-22-99	airb	ldo
SR15284	375241	A9C230119	4	TPH1005	Austin	3-23-99	airb	LBC
—	101101	C9C180212	5	Metals	Denver	3-23-99	Q.rb	TS
—	101101	C9C190248	2	Metals	Denver	3-23-99	Q.rb	TS
SR15286	375241	A9C230117	25	TPH1005, TS	Austin	3-23-99	airb	LBC
SR15217	5928	A9C190107	3	BNA	Austin	3-19-99	AIRB	TS
SR15296	405673	A9C240120	6	Explosives	KNOX	3-24-99	AIRB	TS
SR15311	375241	A9C240200	1	TPH 1005	austin	3-25-99	airb	LBC
SR15310	39722	A9C240180	13	Diss Gasses	Austin	3-25-99	AIRB	TS
—	1216623	A9C250115	27	organic Pb	Chemtek	3-25-99	airb	ldo
SR15332	39722	A9C250168	12	Diss Gasses	Austin	⊥	⊥	TB
SR15355	39722	A9C260210	16	11	11	3-26-99	⊥	TB
—	1216623	Samples assoc. w A9C250115	27	organic, Pb	Chemtek	3-25-99	airb	ldo
SR15374	39722	A9C270115	4	Diss GASSES	AUSTIN	3-29-99	airb	TS
SR15393	5995	A9C300161	3	502.2	ATEL	3-30-99	Cour	TB

13.1.9

Laboratory Generated Bottle Label

CT656-001
A9C310142-001
3 VIALS
614MW001-4
MS

375241
3/30/99
8:42

QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: GLASSWARE WASHING

(SUPERSEDES: REVISION 1, DATED 12/23/96)

Prepared by:	Mark Bruce	10/27/99 Date
Reviewed by:	Dorothy J. Leeson Technology Specialist	10/27/99 Date
Approved by:	Quality Assurance Manager	10/27/99 Date
Approved by:	Environmental Health and Safety Coordinator	10-29-99 Date
Approved by:	Laboratory Director	11-4-99 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 Quanterra associates.

3.2. 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. {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 Quanterra 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.

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 completing the hot tap water rinse, rinse the glassware once with 1:1 Hydrochloric acid, then three times with deionized water. Appropriate protective equipment should be worn. Place glassware in a clean drying area.

4.3.5. After air drying, the glassware should be spot and stain free. If it is not, then repeat the entire procedure.

4.3.6. 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.

5. DEFINITIONS

5.1. Refer to the glossary in the Quality Assurance Management Plan (QAMP)

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 Type II Water	3 Times Tap Water 1 Time 1:1 Hydrochloric Acid* 3x Type II Water	3 Times Tap Water 1 Time 1:1 Hydrochloric Acid 3x Type II 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

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.

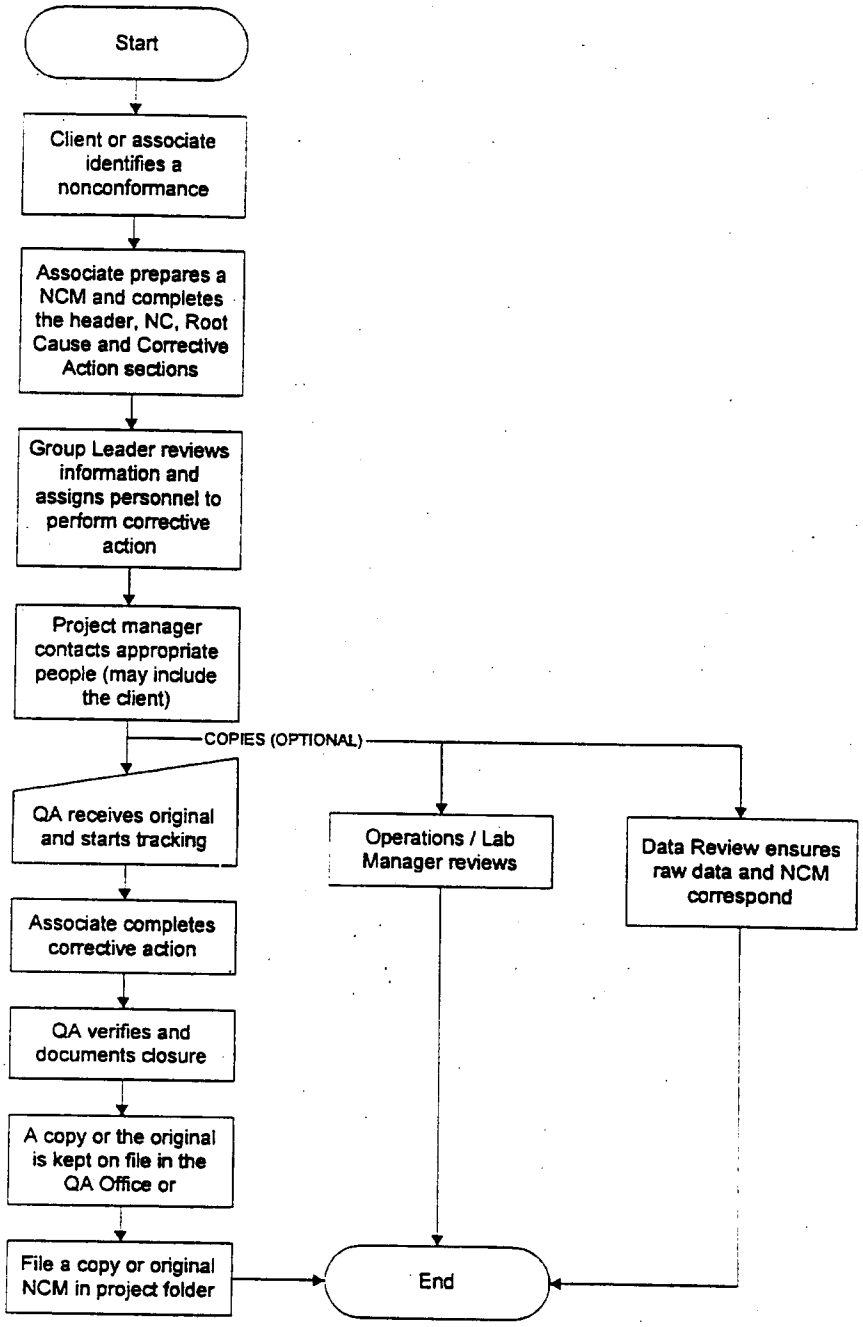
6.2. Appendices

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	❑ GC	❑ Wet chemistry	❑ Data review
❑ Organic preparation	❑ HPLC	❑ Metals	❑ Radiochemistry
❑ Inorganic preparation	❑ GC/MS	❑ Reporting	❑ _____

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: _____

Quality Assurance Review

☐ Audit(s) ☐ Review

☐ Further action required

☐ Notified Ops/Sys Manager (Initials) _____

QA signature

Corrective Action Verification

☐ Verification of required corrective action

☐ Verification complete

☐ Client notified (Specify)

Verified by

Nonconformance Memo Closure

QA signature

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
Nonconformance Memo

Clouseau

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:
Sevda 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
Sevda Aleckson	03/18/99	Quality Assurance

Date Printed: 06/07/99

Page 1 of 1

Controlled Copy
Copy No:_____

Implementation Date: 03/29/00

SOP No. CORP-MS-0002NC
Revision No. 2.1
Revision Date: 03/06/00

STL STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF VOLATILE ORGANICS BY GC/MS BASED ON METHOD 8260B, 624 AND 524.2

(SUPERSEDES: REVISION 2, DATED 12/15/97)

Prepared by: _____ 3/29/00
Date

Approved by: _____ 3/29/00
Technology Specialist — Date

Approved by: _____ 3-13-00
Quality Assurance Manager — Date

Approved by: _____ 3/29/00
Environmental Health and Safety Coordinator — Date

Approved by: _____ 3/29/00
Lab Manager — Date

Approved by: _____ 03/07/00
Corporate Technology and/or Corporate Quality Assurance — Date

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APPENDIX A

APPLICATION OF METHOD 524.2 AND METHOD 8260B TO THE DRINKING WATER ANALYTE LIST 55

APPENDIX B

ANALYSIS OF VOLATILE ORGANICS BY METHOD 624 63

LIST OF TABLES

Table 1 STL Primary Standard and Reporting Limits

Table 2 STL Primary Standard Calibration Levels

Table 3 STL Appendix IX Standard and Reporting Limits

Table 4 STL Appendix IX Standard Calibration Levels

Table 5 Reportable Analytes for STL Standard Tests, Primary Standard

Table 6 Reportable Analytes for STL Standard Tests, Appendix IX Standard

Table 7 Internal Standards

Table 8 Surrogate Standards

Table 9 Matrix Spike / LCS Standards

Table 10 BFB Tune Criteria

Table 11 SPCC Compounds and Minimum Response Factors

Table 12 CCC Compounds

Table 13 Characteristic Ions

Table A-1 STL Drinking Water List Standard and Reporting Limits

Table A-2 Internal Standards, Drinking Water List

Table A-3 Surrogate Standards, Drinking Water List

Table A-4 Calibration Levels, Drinking Water List

Table B-1 Method 624 Analytes and Reporting Limits

Table B-2 Method 624 QC Acceptance Criteria

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. At client request, it may also be used for analysis following method 8240B. Appendices A and B present modifications to the procedures in the main SOP that are necessary for analysis of drinking water by method 524.2 and wastewater by method 624.

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 60 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 satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan) 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 50 µL of 1250 µg/mL solution for a nominal 25 g sample, 10µL 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 50 µL of 1250 µg/mL solution for a nominal 25 g sample, 10µL 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. (50 µL of spike to 25 mL methanol or 10 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 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 50 µL of 1250 µg/mL solution for a nominal 25 g sample, 10µL 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 50 µL of 1250 µg/mL solution for a nominal 25 g sample, 10µL for a nominal 5 g sample.) The addition of spike introduces a slight error, (0.2%) 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. (50 µL of spike to 25 mL methanol or 10 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 (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.

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. It is no longer included 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.

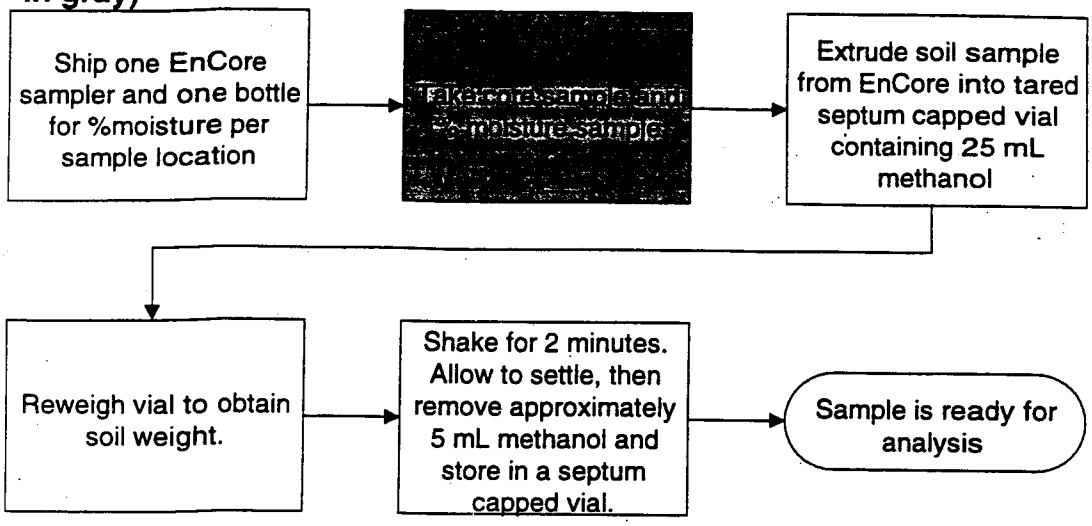
8.10. Medium level solid extracts are aliquoted into 2 - 5 mL glass vials with Teflon lined caps and stored at 4°C +/- 2°C. The extracts are stored with minimum headspace.

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

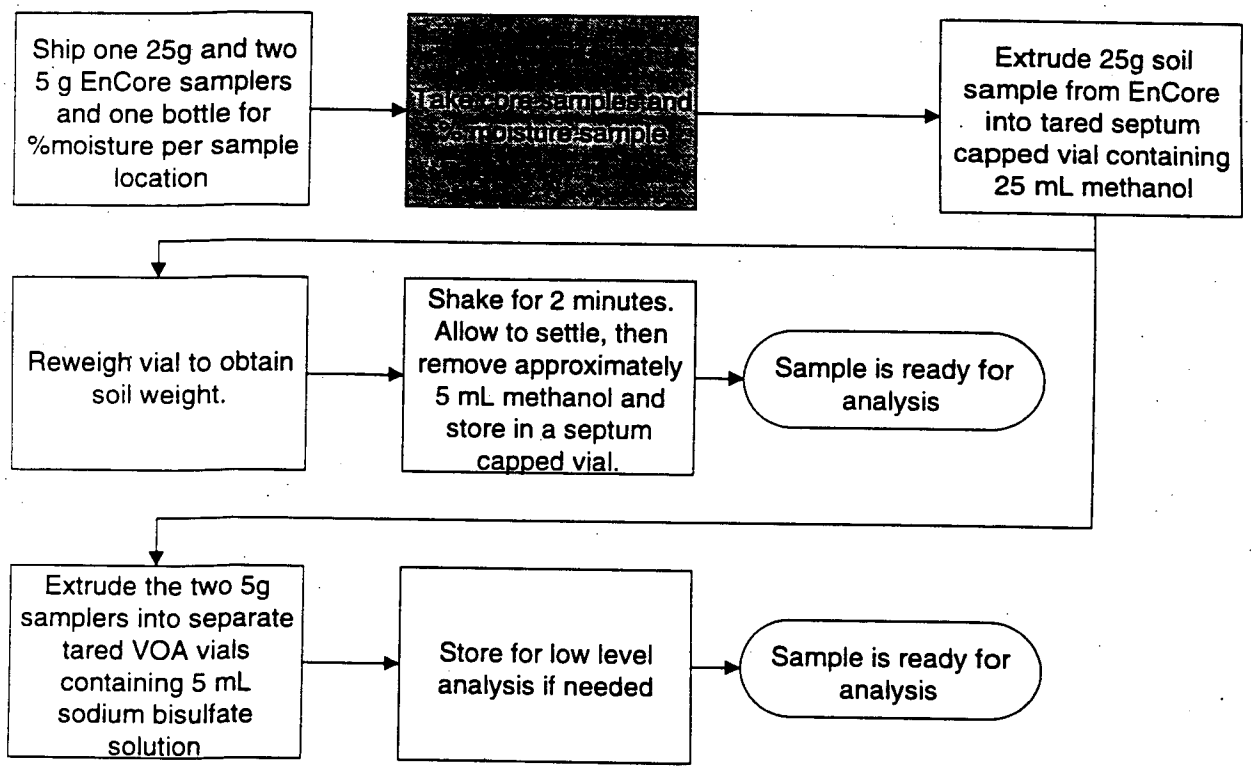
the case narrative). Maximum holding time for the EnCore sampler (before the sample is added to methanol or sodium bisulfate) is 48 hours.

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.

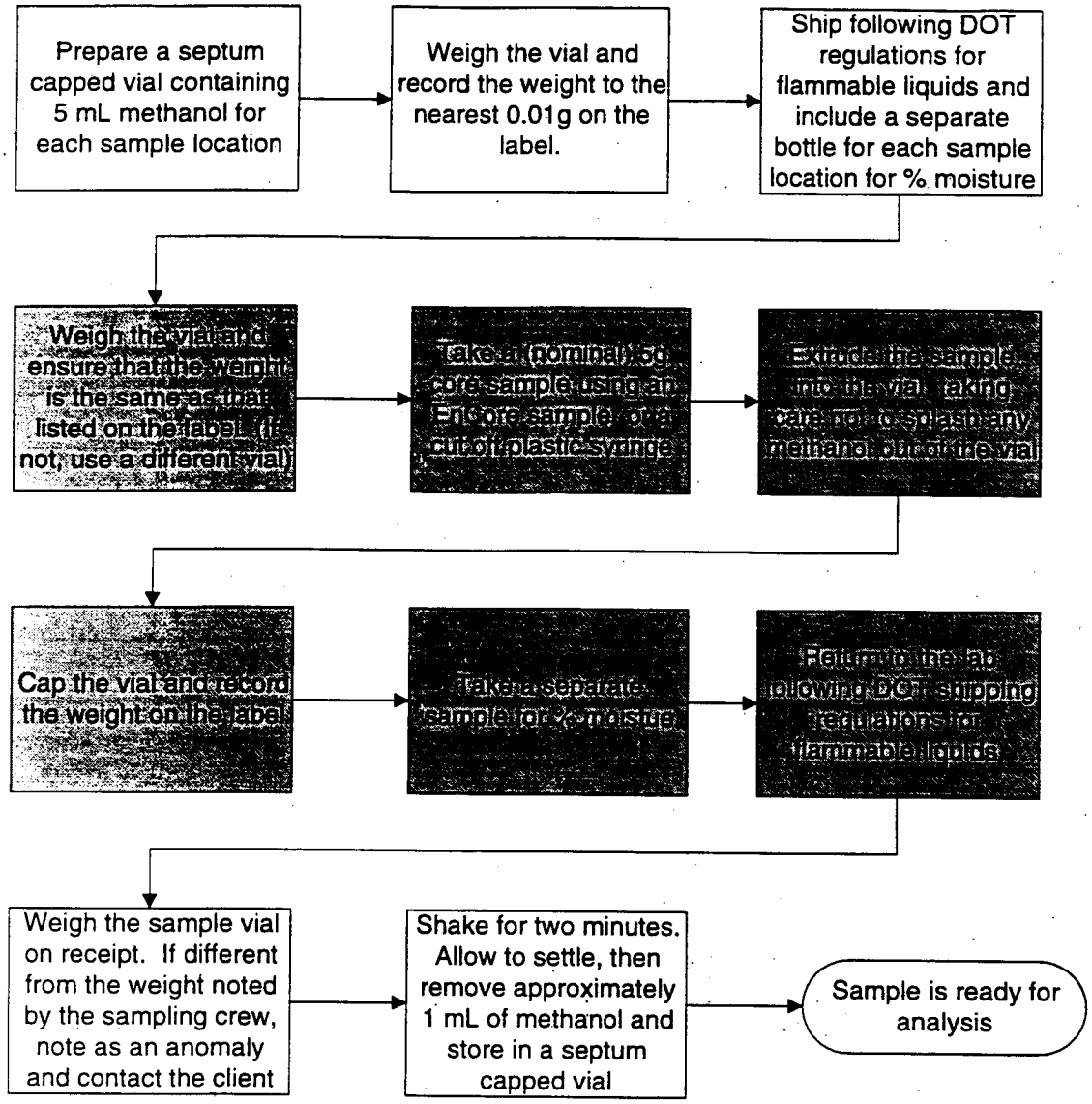
EnCore procedure when low level is not required (field steps in gray)



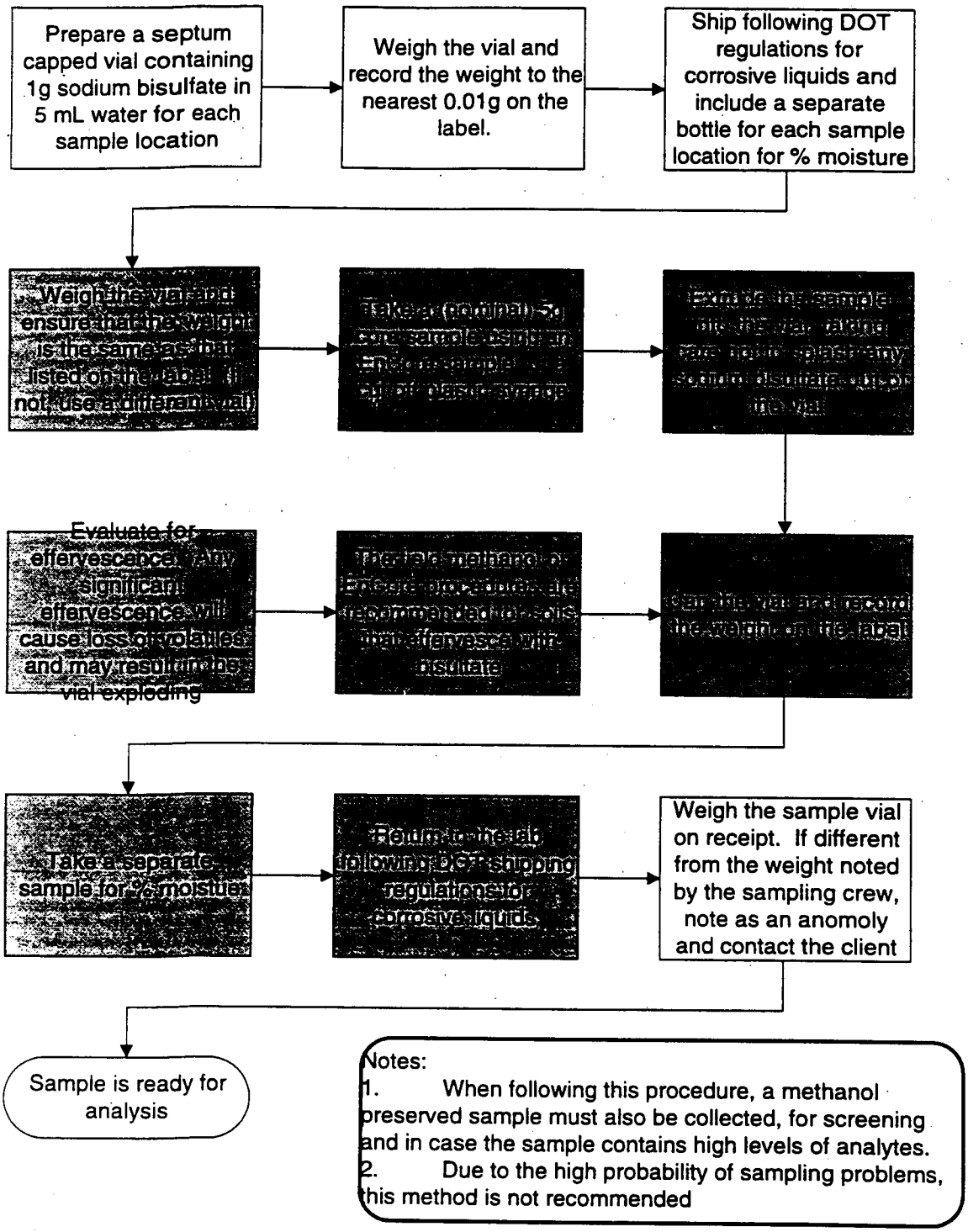
EnCore procedure when low level is required



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

9.5. 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.

9.4.1. 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.

9.4.2. 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.

9.4.3. 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.

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.

- 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. 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, 30, and 60 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. 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 ≥ 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%. 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%. Otherwise the correlation coefficient, (coefficient of determination, r^2 for non-linear curves) must be ≥ 0.990. If the correlation coefficient is < 0.990, then any hit for these compounds must be flagged as estimated.

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. $1/\text{Concentration}^2$ 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.

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).

10.5.4. 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 5-10 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.

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

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 and is not allowed for OhioVAP projects

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. Initial review and corrective actions

11.9.1. 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.

11.9.2. 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.

11.9.2.1. 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.

11.9.3. 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.

11.10. 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

$$\text{Concentration } \mu g / L = \frac{x}{\overline{RF}}$$

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

$$\textbf{RPD} = \frac{|\textbf{MSR - MSDR}|}{\frac{1}{2}(\textbf{MSR + MSDR})} \times 100$$

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

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 $\leq$ 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: If n-Hexane is a compound of interest it must be spiked and reported for both the LCS and MS/MSD.
- Sections 10.4.6: All analytes must have a %RSDs $\leq$ 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

$$\textbf{Concentration } \mu g / L = A + Bx + Cx^2$$

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*.

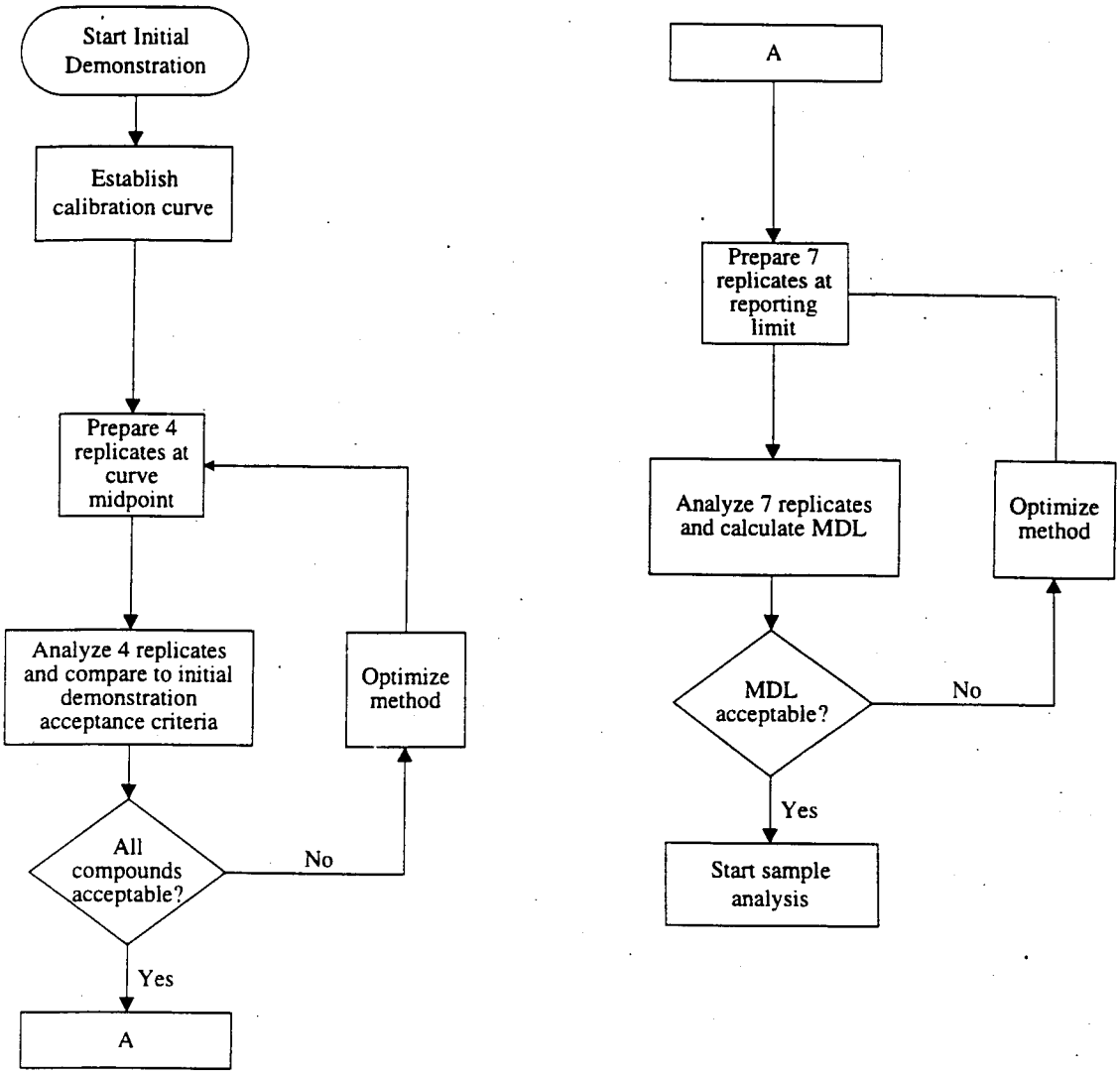

Start Initial Demonstration
Establish calibration curve
Prepare 4 replicates at curve midpoint
Analyze 4 replicates and compare to initial demonstration acceptance criteria
Optimize method
All compounds acceptable?
No
Yes
A
A
Prepare 7 replicates at reporting limit
Analyze 7 replicates and calculate 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 water µg/L	Low soil µg/kg	Med. Soil µg/kg
Dichlorodifluoromethane	75-71-8	10	2	10	500
Chloromethane	74-87-3	10	2	10	500
Bromomethane	74-83-9	10	2	10	500
Vinyl chloride	75-01-4	10	2	10	500
Chloroethane	75-00-3	10	2	10	500
Trichlorofluoromethane	75-69-4	10	2	10	500
Acrolein	107-02-8	100	20	100	5000
Acetone	67-64-1	20	10	20	1000
Trichlorotrifluoroethane	76-13-1	5	1	5	250
Ethanol	64-17-5	500	200	500	25,000
Iodomethane	74-88-4	5	1	5	250
Carbon disulfide	75-15-0	5	1	5	250
Methylene chloride	75-09-2	5	1	5	250
tert-Butyl alcohol	75-65-0	200	50	200	10,000
1,1-Dichloroethene	75-35-4	5	1	5	250
1,1-Dichloroethane	75-34-3	5	1	5	250
trans-1,2-Dichloroethene	156-60-5	5.0	1.0	5.0	250
Acrylonitrile	107-13-1	100	20	100	5000
Methyl *tert*-butyl ether (MTBE)	1634-04-4	20	5	20	1000
Hexane	110-54-3	5	1	5	250
cis-1,2-Dichloroethene	156-59-2	5.0	1.0	5.0	250
1,2-Dichloroethene (Total)	540-59-0	5	1	5	250
Tetrahydrofuran	109-99-9	20	5	20	1000
Chloroform	67-66-3	5	1	5	250
1,2-Dichloroethane	107-06-2	5	1	5	250
Dibromomethane	74-95-3	5	1	5	250
2-Butanone	78-93-3	20	5	20	1000
1,4-Dioxane	123-91-1	500	200	500	25000
1,1,1-Trichloroethane	71-55-6	5	1	5	250
Carbon tetrachloride	56-23-5	5	1	5	250
Bromodichloromethane	75-27-4	5	1	5	250
1,2-Dichloropropane	78-87-5	5	1	5	250
cis-1,3-Dichloropropene	10061-01-5	5	1	5	250
Trichloroethene	79-01-6	5	1	5	250
Dibromochloromethane	124-48-1	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 water µg/L	Low soil µg/kg	Med. Soil µg/kg
1,2-Dibromoethane	106-93-4	5	1	5	250
1,2,3-Trichloropropane	96-18-4	5	1	5	250
1,1,2-Trichloroethane	79-00-5	5	1	5	250
Benzene	71-43-2	5	1	5	250
Ethylmethacrylate	97-63-2	5	1	5	250
trans-1,3-Dichloropropene	10061-02-6	5	1	5	250
Bromoform	75-25-2	5	1	5	250
4-Methyl-2-pentanone	108-10-1	20	5	20	1000
2-Hexanone	591-78-6	20	5	20	1000
Tetrachloroethene	127-18-4	5	1	5	250
Toluene	108-88-3	5	1	5	250
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	250
2-Chloroethyl vinyl ether	110-75-8	N/A[2]	N/A	50	1000
Vinyl acetate	108-05-4	10	2	10	500
Chlorobenzene	108-90-7	5	1	5	250
Ethylbenzene	100-41-4	5	1	5	250
Styrene	100-42-5	5	1	5	250
t-1,4-Dichloro-2-butene	110-57-6	5	1	5	250
m and p Xylenes		5.0	0.5	2.5	125
o-xylene	95-47-6	5.0	0.5	2.5	125
Total xylenes	1330-20-7	5	1	5	250
1,3-Dichlorobenzene	541-73-1	5	1	5	250
1,4-Dichlorobenzene	106-46-7	5	1	5	250
1,2-Dichlorobenzene	95-50-1	5	1	5	250

[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

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
Ethanol	500	2000	5000	10000	20000
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

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
Dibromochloromethane	5	20	50	100	200
1,2-Dibromoethane	5	20	50	100	200
1,2,3-Trichloropropane	5	20	50	100	200
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

[1] **Levels for 25 mL purge are 5 times lower in all cases**

Table 3

STL Appendix IX Standard and Reporting Limits, 5 mL purge

Compound	CAS Number	Reporting Limits			
		5 mL Water µg/L	25 mL 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

[1] Cyclohexanone decomposes to 1,1-dimethoxycyclohexane in methanolic solution. Reporting limits cannot be accurately determined.

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
Isopropylbenzene Mike wants this moved to primary standard	5	20	50	100	200

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-	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
	5					
Trichloroethene	79-01-6	X	X	X	X	X
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	25	96	96
Chlorobenzene-d5	25	117	119
1,4-Dichlorobenzene-d4	25	152	152

Notes:

1) 10 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	25
Dibromofluoromethane	25
Toluene-d_8	25
4-Bromofluorobenzene	25

Notes:

1) 10 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 Compounds

Compound	Standard Concentration µg /mL
1,1-Dichloroethene	25
Trichloroethene	25
Toluene	25
Benzene	25
Chlorobenzene	25

Notes:

1) 10 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 from historical 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	8240B Min. RF	8260B Min. RF
Chloromethane	0.300	0.100
1,1-Dichloroethane	0.300	0.100
Bromoform	>0.100	>0.100
1,1,2,2-Tetrachloroethane	0.300	0.300
Chlorobenzene	0.300	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

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
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58
Iodomethane	142	127	141

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
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
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
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.

18. SUMMARY

18.1. This appendix lists modifications to the main body of the SOP that are necessary for analysis of drinking water by method 524.2.

18.2. A target analyte list based on the list in method 524.2 is frequently requested for analysis by method 8260B. STL's standard analyte list for this test, and the internal and surrogate standards used, are listed in Tables A-1 to A-4 below. In all other respects the method is as described in the main body of this SOP. Note that this without the modifications listed in Section 19, the method is ***not*** appropriate for drinking water analysis by method 524.2.

19. MODIFICATIONS REQUIRED FOR DRINKING WATER ANALYSIS BY METHOD 524.2

19.1. This method can be applied to surface water, ground water and drinking water.

19.2. Purge sample volume is normally 25 mL, but lesser volumes may be used if adequate sensitivity is obtained.

19.3. Sample concentrations are calculated using initial calibration curve.

19.4. Only one internal standard -- Fluorobenzene -- is used for this method, and therefore all target analytes are assigned to it.

19.5. A maximum of 25 ng of BFB is used for tuning for method 524.2

19.6. BFB tuning criteria for mass 75 are 30-80% of mass 95.

19.7. The recovery limits for the initial demonstration of capability are 80-120% with %RSD less than 20%.

19.8. Initial calibration curve requirements:

19.8.1. The number of calibration standards depends on the calibration range used. For a range of up to a factor of 20 (e.g. 1µg/L - 20µg/L) a minimum of three standards are necessary. For a factor of up to 50 four standards are necessary, and for a factor of up to 100 five standards are necessary.

19.8.2. All target compounds must have RSD $\leq$ 20%.

19.8.3. 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.

19.9. Continuing calibration verification (CCV) requirements:

19.9.1. All target compounds must have %D ≤ 30%.

19.9.2. The internal standards in each CCV must be over 70% of the abundance found in the CCV analysis immediately preceding it *and* over 50% of the calibration point in the initial calibration curve whose concentration matches that of the CCV.

19.9.3. The same analysis run may be used to satisfy the requirements for an LCS (also known as a laboratory fortified blank, LFB) and a continuing calibration verification. The LCS/CCV does not need to be a second source standard.

19.10. Method clarifications, modifications and additions

19.10.1. Section 7.1 requires that the trap packing materials be 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.

19.10.2. Section 7.8.2 of the source method requires that each calibration standard be prepared by diluting the appropriate volume of the working standard with organic-free water adjusted to pH < 2 in a volumetric flask. STL prepares calibration standards by diluting the the appropriate volume of the working standard with organic-free water in the gas-tight syringe that will be used to inject the sample into the purge and trap device.

19.10.3. Sections 9.8 and 9.9 of the source method require that duplicate spiked blanks and a second-source initial calibration verification standard be analyzed at least quarterly. Since some STL do not normally analyze drinking waters samples, these QC samples will be analyzed only during the conduct of projects that require this method.

Table A-1

STL 8260 Drinking Water List Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Dichlorodifluoromethane	75-71-8	10	2	10	500
Chloromethane	74-87-3	10	2	10	500
Bromomethane	74-83-9	10	2	10	500
Vinyl chloride	75-01-4	10	2	10	500
Chloroethane	75-00-3	10	2	10	500
Trichlorofluoromethane	75-69-4	10	2	10	500
Acetone[1]	67-64-1	20	10	20	1000
Methylene chloride	75-09-2	5	2	5	250
1,1-Dichloroethene	75-35-4	5	1	5	250
1,1-Dichloroethane	75-34-3	5	1	5	250
trans-1,2-Dichloroethene	156-60-5	2.5	0.5	2.5	125
Methyl *tert*-butyl ether (MTBE)[1]	1634-04-4	20	5	20	250
2,2-Dichloropropane	590-20-7	5	1	5	250
cis-1,2-Dichloroethene	156-59-2	2.5	0.5	2.5	125
1,2-Dichloroethene (Total)	540-59-0	5	1	5	250
Chloroform	67-66-3	5	1	5	250
Bromochloromethane	74-97-5	5	1	5	250
1,2-Dichloroethane	107-06-2	5	1	5	250
Dibromomethane	74-95-3	5	1	5	250
2-Butanone[1]	78-93-3	20	5	20	1000
1,1,1-Trichloroethane	71-55-6	5	1	5	250
Carbon tetrachloride	56-23-5	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
cis-1,3-Dichloropropene	10061-01-5	5	1	5	250
Trichloroethene	79-01-6	5	1	5	250
Dibromochloromethane	124-48-1	5	1	5	250
1,2-Dibromoethane	106-93-4	5	1	5	250
1,2,3-Trichloropropane	96-18-4	5	1	5	250
1,1,2-Trichloroethane	79-00-5	5	1	5	250
Benzene	71-43-2	5	1	5	250
trans-1,3-Dichloropropene	10061-02-6	5	1	5	250
Bromoform	75-25-2	5	1	5	250

Table A-1

STL 8260 Drinking Water List Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
4-Methyl-2-pentanone[1]	108-10-1	20	5	20	1000
2-Hexanone[1]	591-78-6	20	5	20	1000
Tetrachloroethene	127-18-4	5	1	5	250
Toluene	108-88-3	5	1	5	250
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	250
Chlorobenzene	108-90-7	5	1	5	250
1,1,1,2-Terrachloroethane	630-20-6	5	1	5	250
Ethylbenzene	100-41-4	5	1	5	250
Styrene	100-42-5	5	1	5	250
m and p Xylenes		2.5	0.5	2.5	125
o-xylene	95-47-6	2.5	0.5	2.5	125
Total xylenes	1330-20-7	5	1	5	250
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
1,3-Dichlorobenzene	541-73-1	5	1	5	250
1,4-Dichlorobenzene	106-46-7	5	1	5	250
1,2-Dichlorobenzene	95-50-1	5	1	5	250
4-Isopropyltoluene	99-87-6	5	1	5	250
n-Butylbenzene	104-51-8	5	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	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

[1] Not included on the method 524.2 analyte list, but includes in the calibration standard as an add on frequently requested by method 8260B.

Table A-2

Internal Standards, Method 8260A Drinking water list

	Standard Concentration µg/mL	Quantitation ion
Fluorobenzene	25	96
Chlorobenzene-d5	25	119
1,4-Dichlorobenzene-d4	25	152

Notes:

1) Fluorobenzene only is used for method 524.2

2) 10 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.

3) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table A-3

Surrogate Standards, Drinking water list

Surrogate Compounds	Standard Concentration µg/mL
1,2-Dichloroethane-d_4[1]	25
Dibromofluoromethane[1]	25
Toluene-d_8[1]	25
1,2-Dichlorobenzene-d4[2]	25
4-Bromofluorobenzene[1,2]	25

[1] 8260B surrogate

[2] 524.2 surrogate

Notes:

1) 10 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.

Table A-4

STL Drinking water list Standard: Calibration Levels

Compound	Level 1		Level 2		Level 3		Level 4		Level 5	
	5 mL	25 mL	5 mL	25 mL	5 mL	25 mL	5 mL	25 mL	5 mL	25 mL
Dichlorodifluoromethane	10	2	40	10	100	20	200	60	400	120
Chloromethane	10	2	40	10	100	20	200	60	400	120
Bromomethane	10	2	40	10	100	20	200	60	400	120
Vinyl chloride	10	2	40	10	100	20	200	60	400	120
Chloroethane	10	2	40	10	100	20	200	60	400	120
Trichlorofluoromethane	10	2	40	10	100	20	200	60	400	120
Acetone[1]	10		40	10	100	20	200	60	400	120
Methylene chloride	5	1	20	5	50	10	100	30	200	60
1,1-Dichloroethene	5	1	20	5	50	10	100	30	200	60
1,1-Dichloroethane	5	1	20	5	50	10	100	30	200	60
trans-1,2-Dichloroethene	5	1	20	5	50	10	100	30	200	60
Methyl *tert*-butyl ether (MTBE)	10		40	10	100	20	200	60	400	120
2,2-Dichloropropane	5	1	20	5	50	10	100	30	200	60
cis-1,2-Dichloroethene	5	1	20	5	50	10	100	30	200	60
Chloroform	5	1	20	5	50	10	100	30	200	60
Bromochloromethane	5	1	20	5	50	10	100	30	200	60
1,2-Dichloroethane	5	1	20	5	50	10	100	30	200	60
Dibromomethane	5	1	20	5	50	10	100	30	200	60
2-Butanone[1]	10		40	10	100	20	200	60	400	120
1,1,1-Trichloroethane	5	1	20	5	50	10	100	30	200	60
Carbon tetrachloride	5	1	20	5	50	10	100	30	200	60
Bromodichloromethane	5	1	20	5	50	10	100	30	200	60
1,2-Dichloropropane	5	1	20	5	50	10	100	30	200	60
cis-1,3-Dichloropropene	5	1	20	5	50	10	100	30	200	60
Trichloroethene	5	1	20	5	50	10	100	30	200	60

Table A-4

STL Drinking water list Standard: Calibration Levels

Compound	Level 1		Level 2		Level 3		Level 4		Level 5	
Dibromochloromethane	5	1	20	5	50	10	100	30	200	60
1,2-Dibromoethane	5	1	20	5	50	10	100	30	200	60
1,2,3-Trichloropropane	5	1	20	5	50	10	100	30	200	60
1,1,2-Trichloroethane	5	1	20	5	50	10	100	30	200	60
Benzene	5	1	20	5	50	10	100	30	200	60
trans-1,3-Dichloropropene	5	1	20	5	50	10	100	30	200	60
Bromoform	5	1	20	5	50	10	100	30	200	60
4-Methyl-2-pentanone[1]	10		40	10	100	20	200	60	400	120
2-Hexanone[1]	10		40	10	100	20	200	60	400	120
Tetrachloroethene	5	1	20	5	50	10	100	30	200	60
Toluene	5	1	20	5	50	10	100	30	200	60
1,1,2,2-Tetrachloroethane	5	1	20	5	50	10	100	30	200	60
Chlorobenzene	5	1	20	5	50	10	100	30	200	60
Ethylbenzene	5	1	20	5	50	10	100	30	200	60
Styrene	5	1	20	5	50	10	100	30	200	60
m and p Xylenes	5	1	20	5	50	10	100	30	200	60
o-xylene	5	1	20	5	50	10	100	30	200	60
Isopropylbenzene	5	1	20	5	50	10	100	30	200	60
Bromobenzene	5	1	20	5	50	10	100	30	200	60
n-Propylbenzene	5	1	20	5	50	10	100	30	200	60
2-Chlorotoluene	5	1	20	5	50	10	100	30	200	60
4-Chlorotoluene	5	1	20	5	50	10	100	30	200	60
1,3,5-Trimethylbenzene	5	1	20	5	50	10	100	30	200	60
tert-Butylbenzene	5	1	20	5	50	10	100	30	200	60
1,2,4-Trimethylbenzene	5	1	20	5	50	10	100	30	200	60
sec-butylbenzene	5	1	20	5	50	10	100	30	200	60
1,3-Dichlorobenzene	5	1	20	5	50	10	100	30	200	60
1,4-Dichlorobenzene	5	1	20	5	50	10	100	30	200	60

Table A-4

STL Drinking water list Standard: Calibration Levels

Compound	Level 1		Level 2		Level 3		Level 4		Level 5	
1,2-Dichlorobenzene	5	1	20	5	50	10	100	30	200	60
4-Isopropyltoluene	5	1	20	5	50	10	100	30	200	60
n-Butylbenzene	5	1	20	5	50	10	100	30	200	60
1,2-Dibromo-3-chloropropane	5	1	20	5	50	10	100	30	200	60
1,2,4-Trichlorobenzene	5	1	20	5	50	10	100	30	200	60
Napthalene	5	1	20	5	50	10	100	30	200	60
Hexachlorobutadiene	5	1	20	5	50	10	100	30	200	60
1,2,3-Trichlorobenzene	5	1	20	5	50	10	100	30	200	60

[1] Not included in the STL Standard test, but included in the standard as a frequently requested add-on.

20. REQUIREMENTS FOR EPA 624

20.1. 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.

20.2. The tune period for this method is defined as 24 hours.

20.3. The initial calibration curve for this method requires at least three points.

20.4. Sample concentrations are calculated using the average RRF from the initial calibration curve.

20.5. Each target analyte is assigned to the closest eluting internal standard.

20.6. Initial demonstration of Proficiency

20.6.1. The spiking level for the four replicate initial demonstration of proficiency is 20 µg/L. The acceptance criteria are listed in Table B-2

20.7. Initial calibration curve requirements:

20.7.1. Target compounds must have RSD ≤ 35%.

20.7.2. 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.

20.8. Continuing calibration verification requirements:

20.8.1. The continuing calibration standard is from a different source than the initial calibration standard. The acceptance criteria are listed in Table B-2.

20.9. Matrix Spike and LCS requirements

20.9.1. 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.

20.10. Method clarifications, modifications and additions

20.10.1. 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.

20.10.2. Section 5.3.2 of the source method describes a packed analytical column. STL routinely employs capillary columns when performing this method.

20.10.3. 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 B-1.

Method 624 Analytes and Reporting Limits

Analytes	µg/L
Benzene	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	5
Carbon tetrachioride	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 B-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

QUANTERRA® 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: CORP-OP-0001, Revision 3.5 (Dated 4/22/99))

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Table A1 Herbicide surrogate spiking solutions
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Table A4 Herbicide matrix spike and LCS components

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 Quality Assurance Management Plan (QAMP).

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 Quanterra 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 Quanterra 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	√				
Methylene Chloride Collection Tank	√	√			
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	√	√	√	√	

EQUIPMENT AND SUPPLIES	Sep fun.	CLLE	Soni	Sox	Conc
Glass Funnel: 75 X 75 mm	√	√	√	√	√
Disposable Pipets	√	√	√	√	√
Aluminum foil	√	√	√	√	√
Paper Towels	√	√	√	√	√

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 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.	√	√			
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					√	

REAGENTS	GPC	Florisil	Sulfur	Acid	A/B	FC
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 $\leq$ 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

10.2. Extraction is initiated within 7 days of the sampling date for aqueous samples, 14 days for solid and waste samples.

20.2. 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. Quanterra QC Program

Further details of QC and corrective action guidelines are presented in the Quanterra 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.

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.

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.

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.

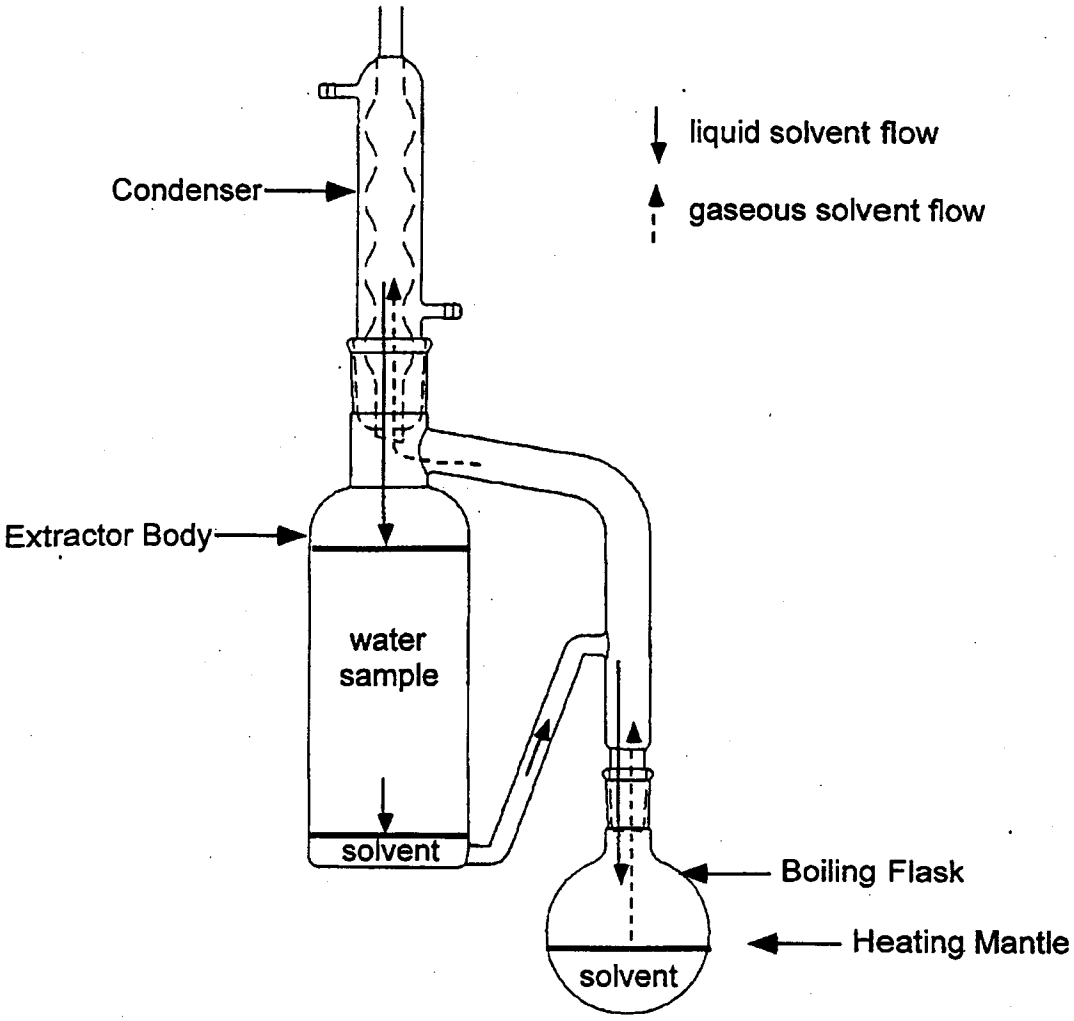
Note: Alternative methods of measurement of sample volume include: a.) transferring the sample to a measuring cylinder and b.) marking a meniscus on the sample bottle and then measuring the volume of water required to fill the bottle to the meniscus after the sample is transferred. The former method 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.

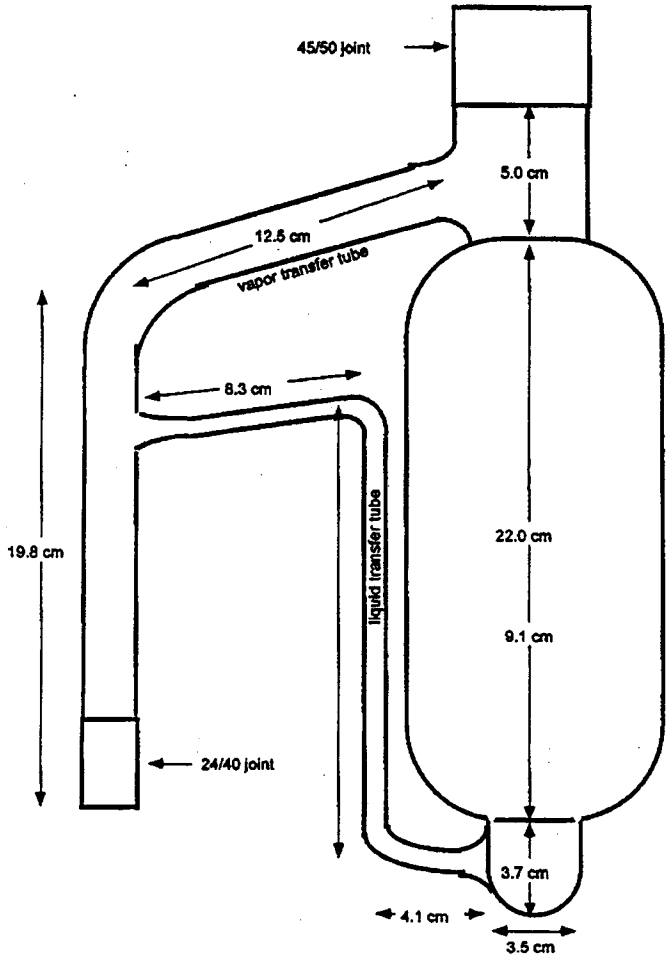
11.3.3.2. Prepare a method blank, LCS and MS/MSD for each batch as specified in section 9 of this SOP. Use 1 L of reagent water for method blanks and LCS. The method blank is spiked with the surrogates, the LCS and matrix spikes with the surrogates and matrix spiking solutions. Note that different spiking solutions are used for methods 625, 8270 and TCLP.

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. 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.

11.5.5.1. Sample weights less than 30 g but over 5 g may be used if the appropriate reporting limits can be met.

11.5.5. 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.

11.5.6. 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.

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) For GC/MS analysis, the surrogate solution should be 10x more concentrated than the normal solution (i.e. 1000/1500 ppm). For pesticides the normal surrogate solution is appropriate.

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.

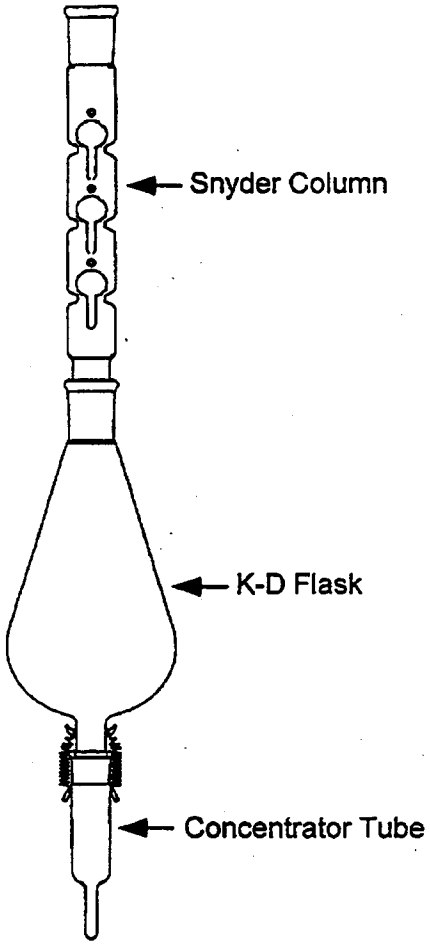
11.7. Concentration

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 (80-90°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 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 10 mL of hexane, 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 (90°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), PAH (8100) 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.

11.8.1.5.4. 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.

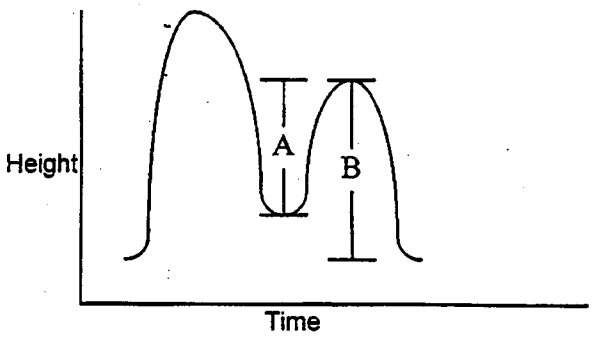
11.8.1.6.11. Solvent exchange pesticide/PCB sample extracts into hexane and concentrate to 5.0 mL. Refer to the concentration section 11.7.

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) assembly, Zymark Benchmate, or equivalent.

Note 2: Follow the lab specific procedure when using the Zymark benchmate.

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 Quanterra 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: 8270[1]	1-2	11-12
625	11-12	1-2
Pest/PCB: 8080 & 608	5-9	None
OPP: 8140	6-8	None
OPP: 8141	as received	None
Hydrocarbons: 8015	as received	None
PAH: 8310, 8100 & 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
Type	Exchange Solvent for Analysis	Final Volume for Analysis in mL
Semivolatiles	N/A	1.0 or 5.0*
PCB	Hexane	10.0 for solids 5.0 for H2O
Pesticides	Hexane	10.0 for solids 5.0 for H2O
PAH by HPLC	Acetonitrile	Refer to facility SOP for HPLC
PAH by GC	N/A	1.0
TPH	N/A	1.0
OPP	Toluene, hexane or isooctaneHexane/Acetone	2.0

*Requires high sensitivity mass spec tune (refer to CORP-MS-0001NC)

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	CH_2Cl_2	10 mL[1]	0.5 mL CH_2Cl_2
Pesticides	CH_2Cl_2	10 mL[1]	5 mL, hexane
OPP	CH_2Cl_2	10 mL	5 mL

[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
Pest	0.2 ppm DCB/TCX	1.0
TPH	40ng C9	1.0
PCB	0.2 ppm DCB/TCX	1.0

Table 4

Matrix Spike and LCS Solutions

Analyte Group	Matrix Spike Solution ID	Volume (mL)
BNA	100/150 ppm BNA	0.5
BNA TCLP	BNA TCLP Spike	1.0
BNA NPDES	BNA NPDES Spike	1.0
Pest	Pest NPDES Spike	1.0
Pest TCLP	Pest TCLP Spike	1.0
Pest NPDES	Pest NPDES Spike	1.0
Pest/PCB	Pest NPDES 1 ug/mL for regular pesticides - solid and waters. Full analyte Sspike.	1.0
PCB	10 ppm PCB Spike	1.0

Table 5
Surrogate Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
BNA	2-Fluorobiphenyl	Methanol	150
	Nitrobenzene-d5		100
	p-Terphenyl-d14		100
	2-Fluorophenol		100
	Phenol-d6		150
	2,4,6-Tribromophenol		150
	1,2-Dichlorobenzene-d4		100
	2-Chlorophenol-d4		150
Pest/PCB	Decachlorobiphenyl	Methanol Acetone	0.2
	Tetrachloro-m-xylene		0.2
TPH	Tetrachloro-m-xyleneC9	Methanol	40.0

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

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	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
BNA NPDES		Methanol	
	Acenaphthene		100
	Acenaphthylene		100
	Anthracene		100
	Benzo(a)anthracene		100

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	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
	Diethyl phthalate		100
	Dimethyl phthalate		100
	2,4-Dinitrotoluene		100
	2,6-Dinitrotoluene		100
	Di-n-octylphthalate		100

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
	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
	2-Nitrophenol		100
	4-Nitrophenol		100
	Pentachlorophenol		100
	Phenol		100
	2,4,6-Trichlorophenol		100

Table 6

Matrix Spike Components

Type	Compounds	Solvent	Conc. (µg/mL)
Pest TCLP	Heptachlor	Methanol Acetone	0.5
	Heptachlor epoxide		0.5
	Lindane		0.5
	Endrin		0.5
	Methoxychlor		1.0
Pest NPDES /Pest	Aldrin	Methanol Acetone	0.2
	alpha-BHC		0.2
	beta-BHC		0.2
	delta-BHC		0.2
	gamma-BHC (Lindane)		0.2
	4,4'-DDD		1.0
	4,4'-DDE		0.2
	4,4'-DDT		1.0
	Dieldrin		0.2
	alpha-Endosulfan		0.2
	beta-Endosulfan		1.0
	Endosulfan Sulfate		1.0
	Endrin		1.0
	Heptachlor		0.2
	Heptachlor Epoxide		0.2

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

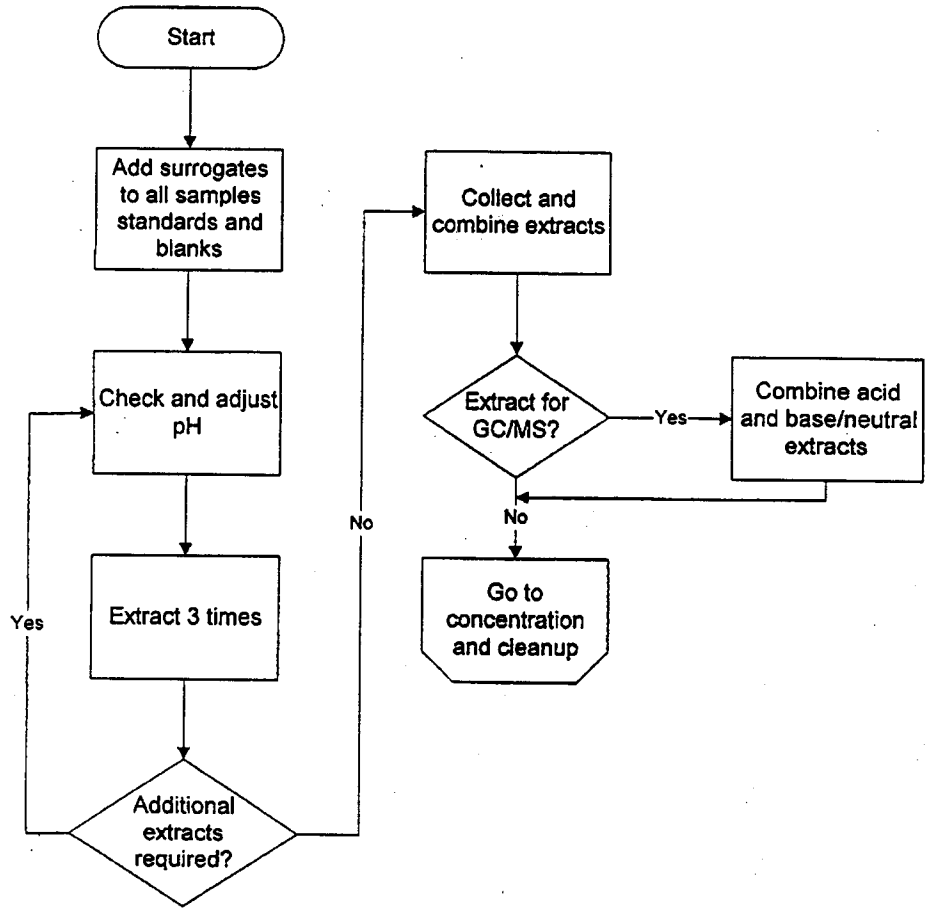
Polynuclear Aromatic Hydrocarbons (8310)	
Compound	**Final Concentration**
Acenaphthylene	10 µg/mL
Carbazole	10 µg/mL
Naphthalene	10 µg/mL
1-methylnaphthalene	10 µg/mL
2-methylnaphthalene	10 µg/mL
Acenaphthene	10 µg/mL
Fluorene	2 µg/mL

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
indeo(1,2,3-cd)pyrene	2 µg/mL
p-terphenyl-d14 (surrogate)	10 µg/mL
benzo(e)pyrene (surrogate)	5 µg/mL

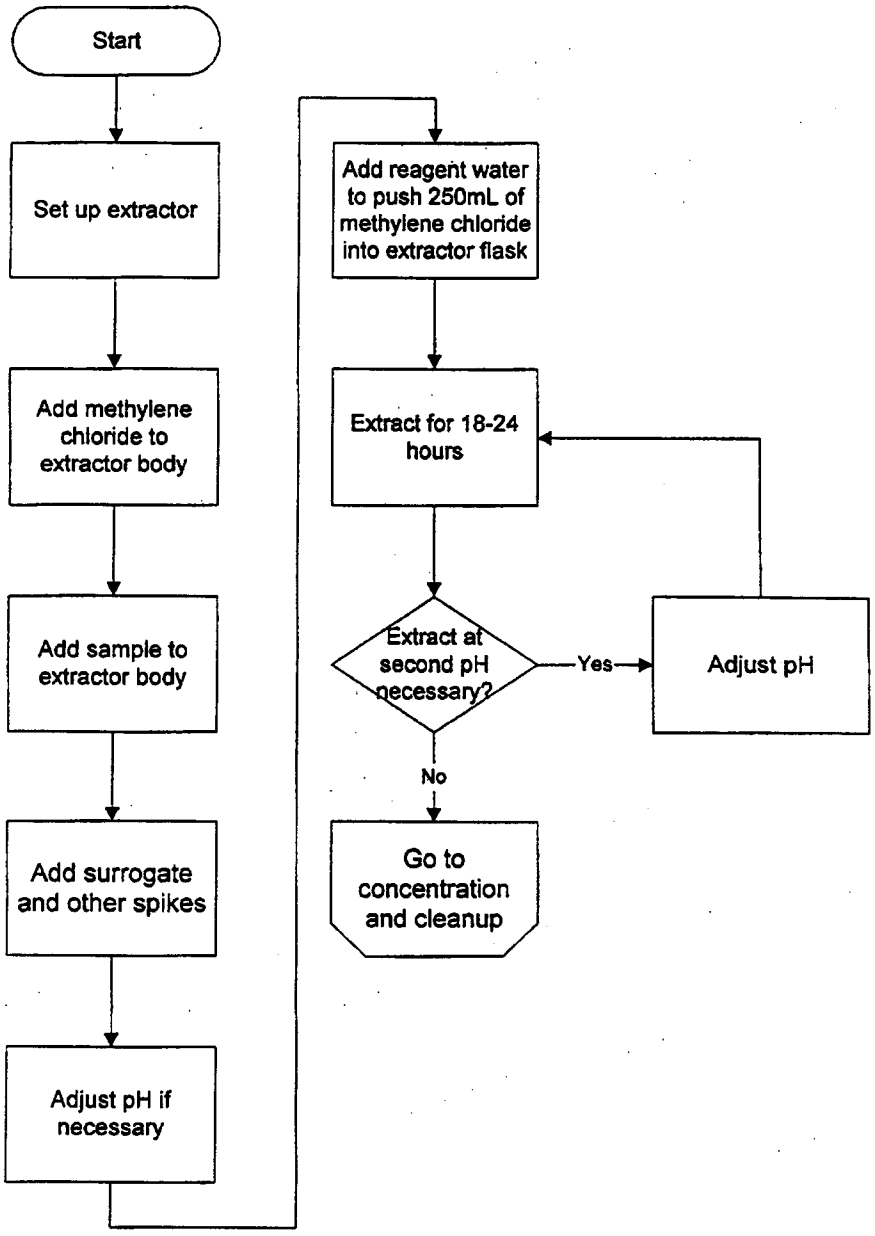
Polynuclear Aromatic Hydrocarbons (8100)	
Compound	**Final Concentration**
acenaphthene	40 µg/mL
acenaphthylene	40 µg/mL
anthrancene	40 µg/mL
benzo(a)anthracene	40 µg/mL
benzo(a)pyrene	40 µg/mL
bezno(b)fluoranthene	40 µg/mL
benzo(g,h,I)perylene	40 µg/mL
benzo(k)fluoranthene	40 µg/mL
chrysene	40 µg/mL
dibenz(a,h)anthracene	40 µg/mL
fluoranthene	40 µg/mL
fluorene	40 µg/mL
indeno(1,2,3-cd)pyrene	40 µg/mL
naphthalene	40 µg/mL
phenanthrene	40 µg/mL
pyrene	40 µg/mL
2-fluorobiphenyl (surrogate)	20 µg/mL
benzo(e)pyrene (surrogate)	20 µg/mL

17.5. Flow diagrams

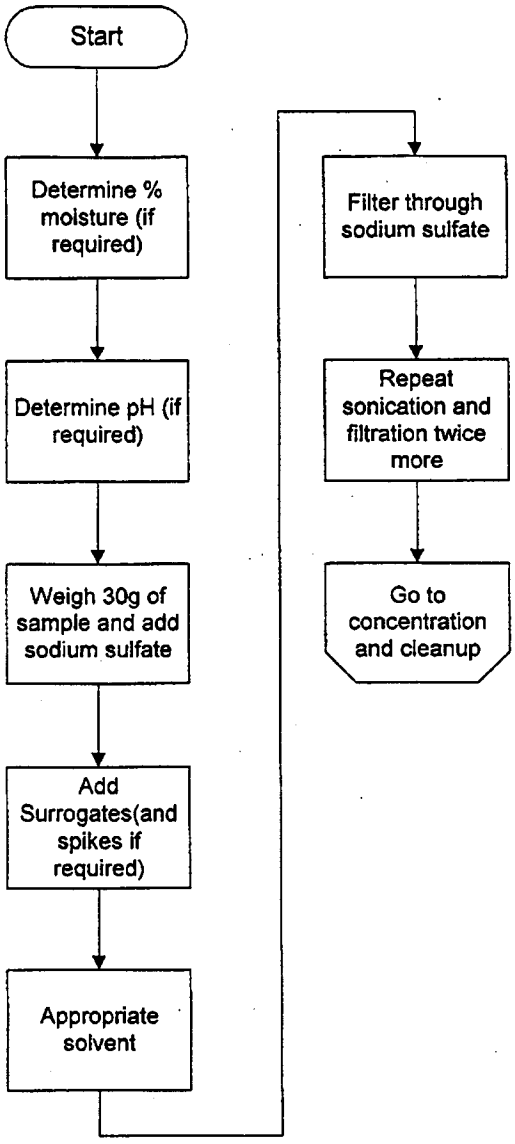
17.5.1. Separatory funnel extraction



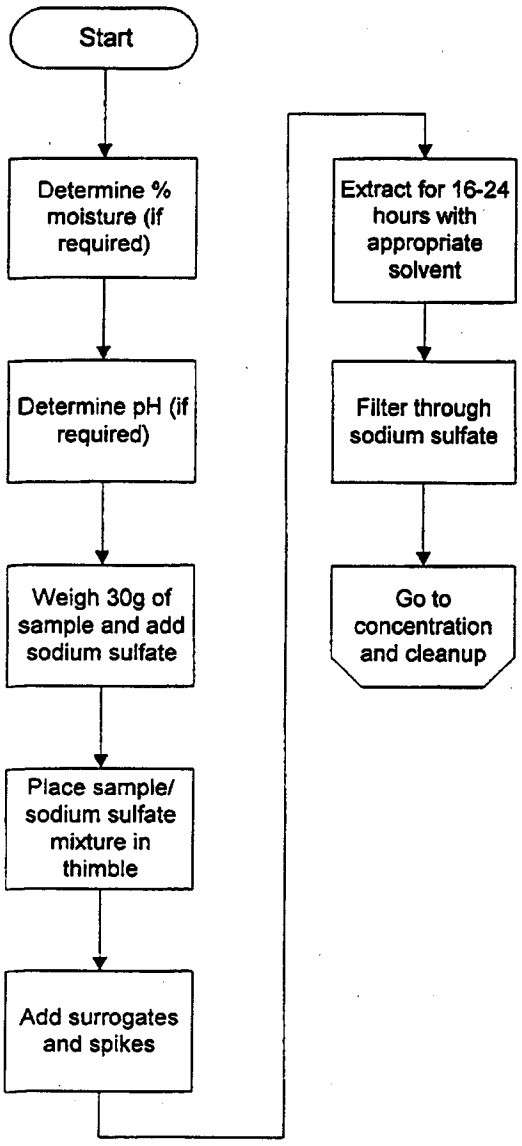
17.5.2. Continuous liquid/liquid extraction



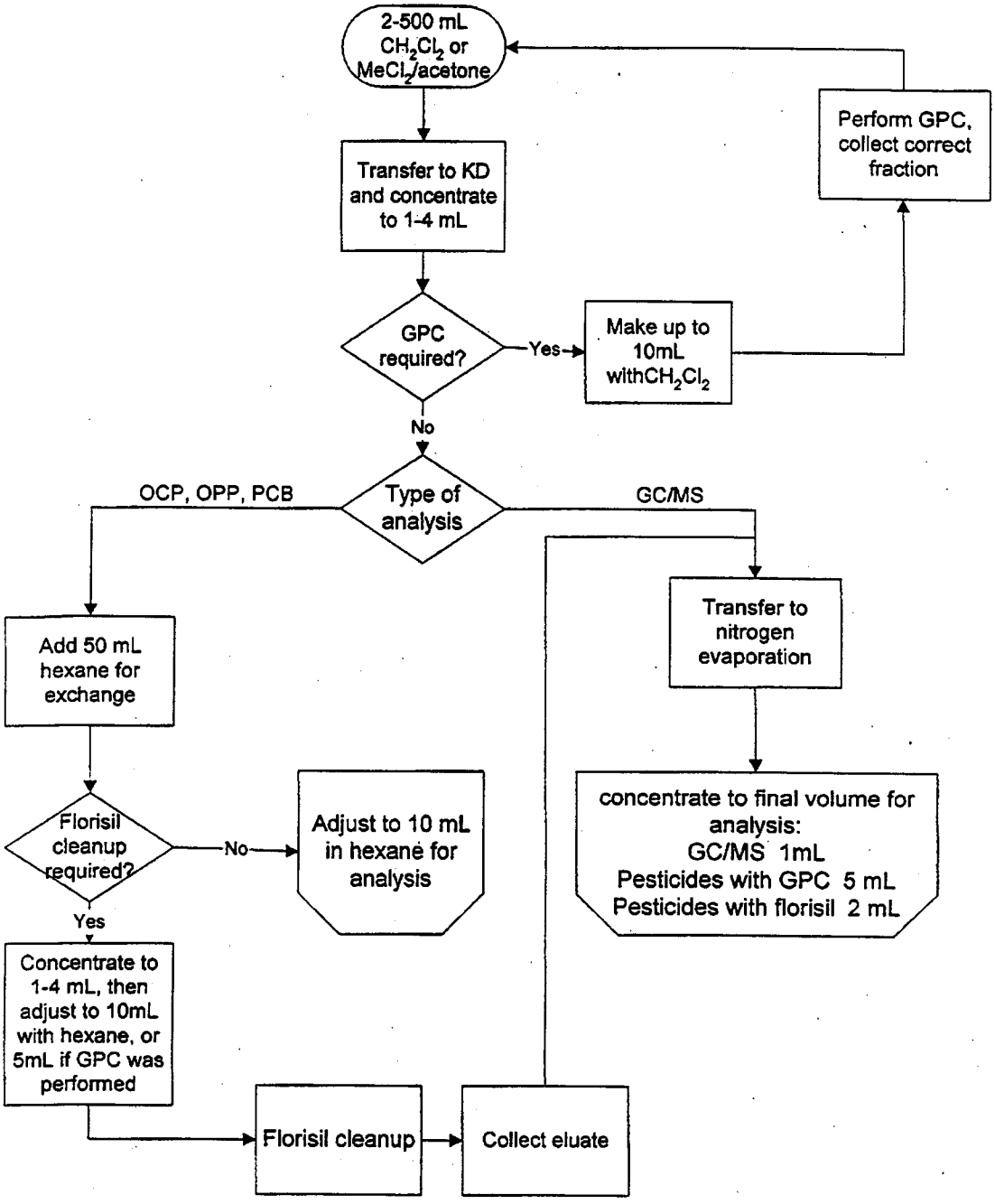
17.5.3. Sonication Extraction



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.

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 ≤ 2, and more acid to adjust the pH to ≤ 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 ≤ 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)

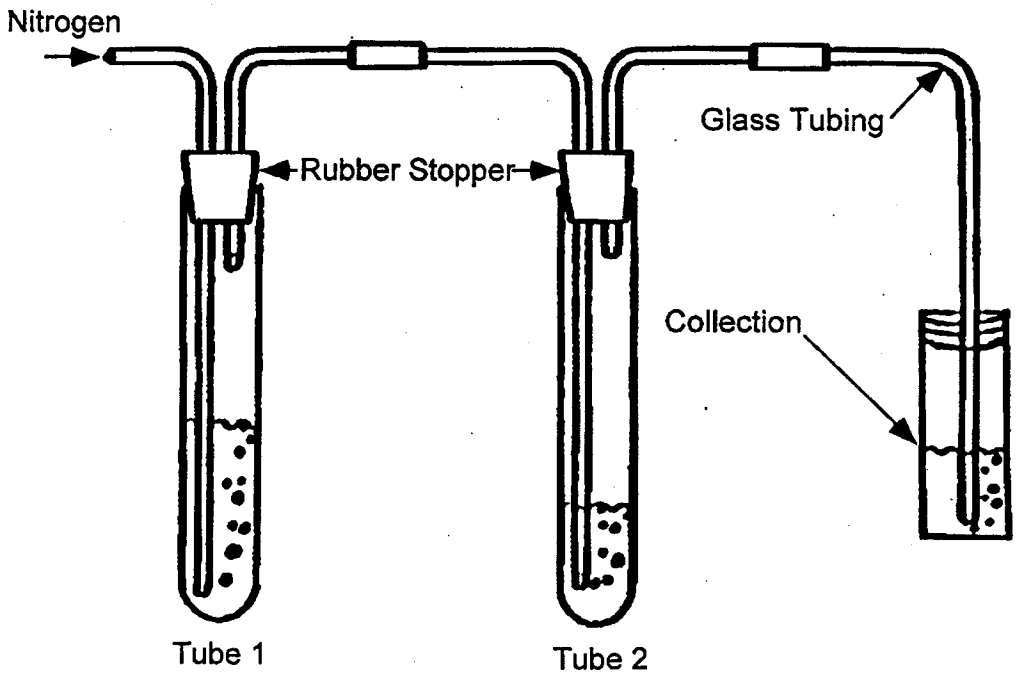
28.7.1. Assemble the diazomethane apparatus (see figure below) in a hood. Add 10 mL of diethyl ether to tube 1. Add 5 mL of 2% methanolic KOH, 3 mL of ether and 0.5-1 g of diazald to tube 2.

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

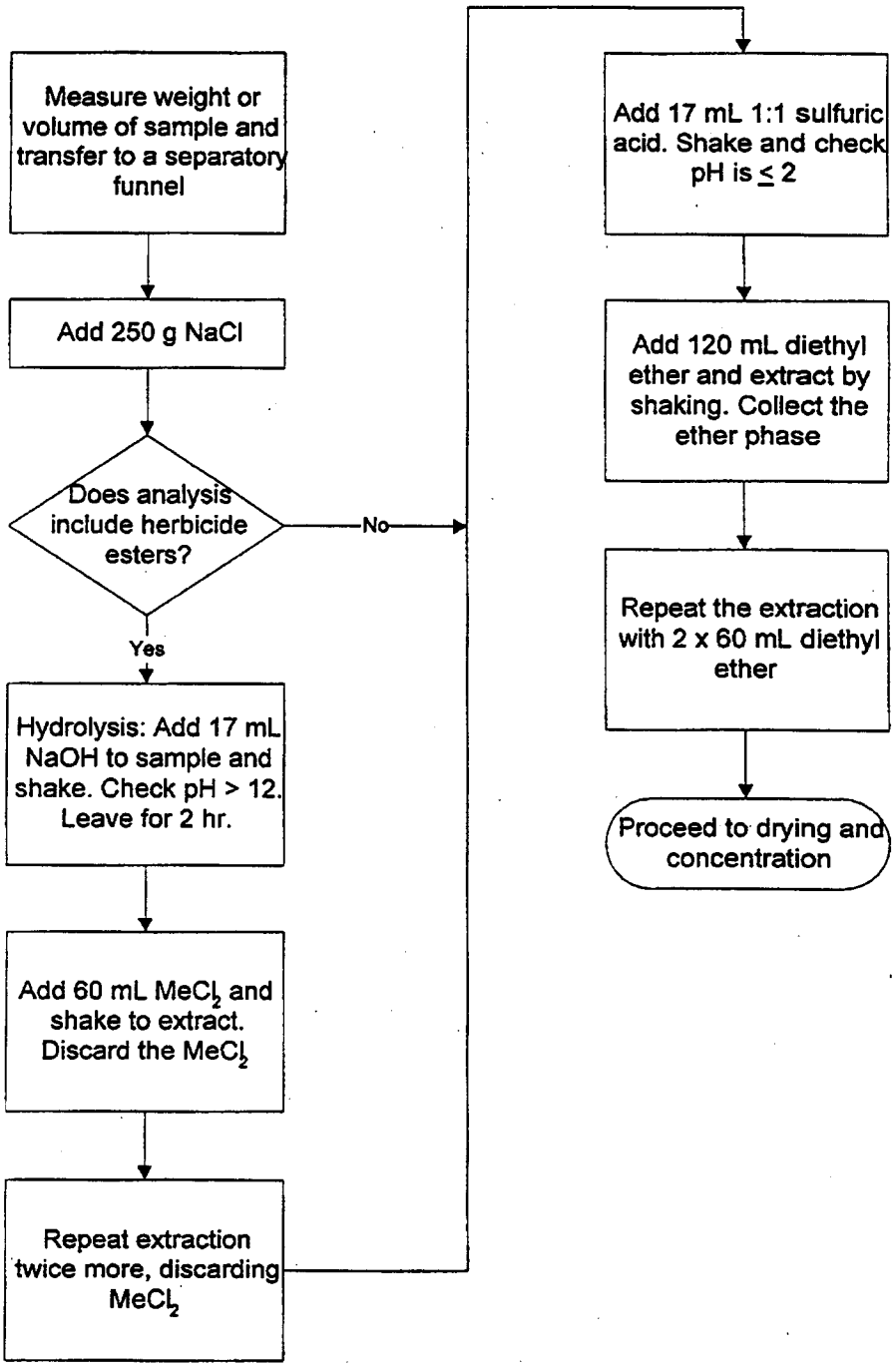
Table A3 Herbicide Surrogate Spike Components			
Type	Compounds[1]	Solvent	Conc. (ug/mL)
Herbicides WS	2,4-DCAA	Acetone	2
Herbicides SS	2,4-DCAA	Acetone Methanol	20

[1]The surrogate is spiked as the free acid

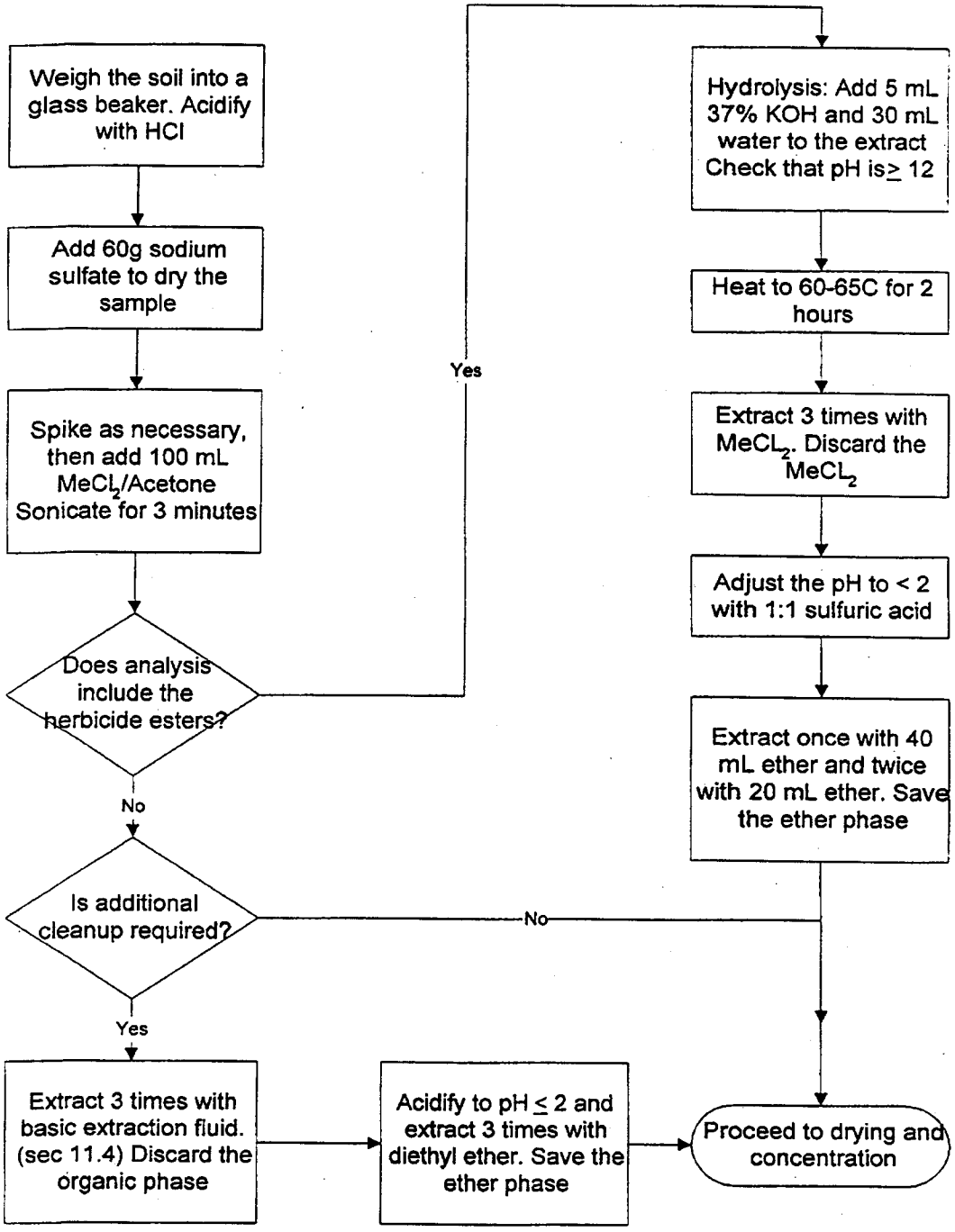
Table A4 Herbicide Matrix Spike Components				
Type	Compounds[1]	Compounds[1] Solvent	Soil Conc. (ug/mL) Solvent	Water Conc. (ug/mL)
Herbicides MS	2,4-D	Acetone	20 Methanol	2
	2,4-DB		20	2
	2,4,5-TP (Silvex)		5	0.5
	Dalapon		10	1
	Dicamba		10	1
	Dichloroprop		10	1
	Dinoseb		3	.3
	2,4,5-T		5	0.5

[1]The herbicide spiking solution contains the herbicides as the free acids.

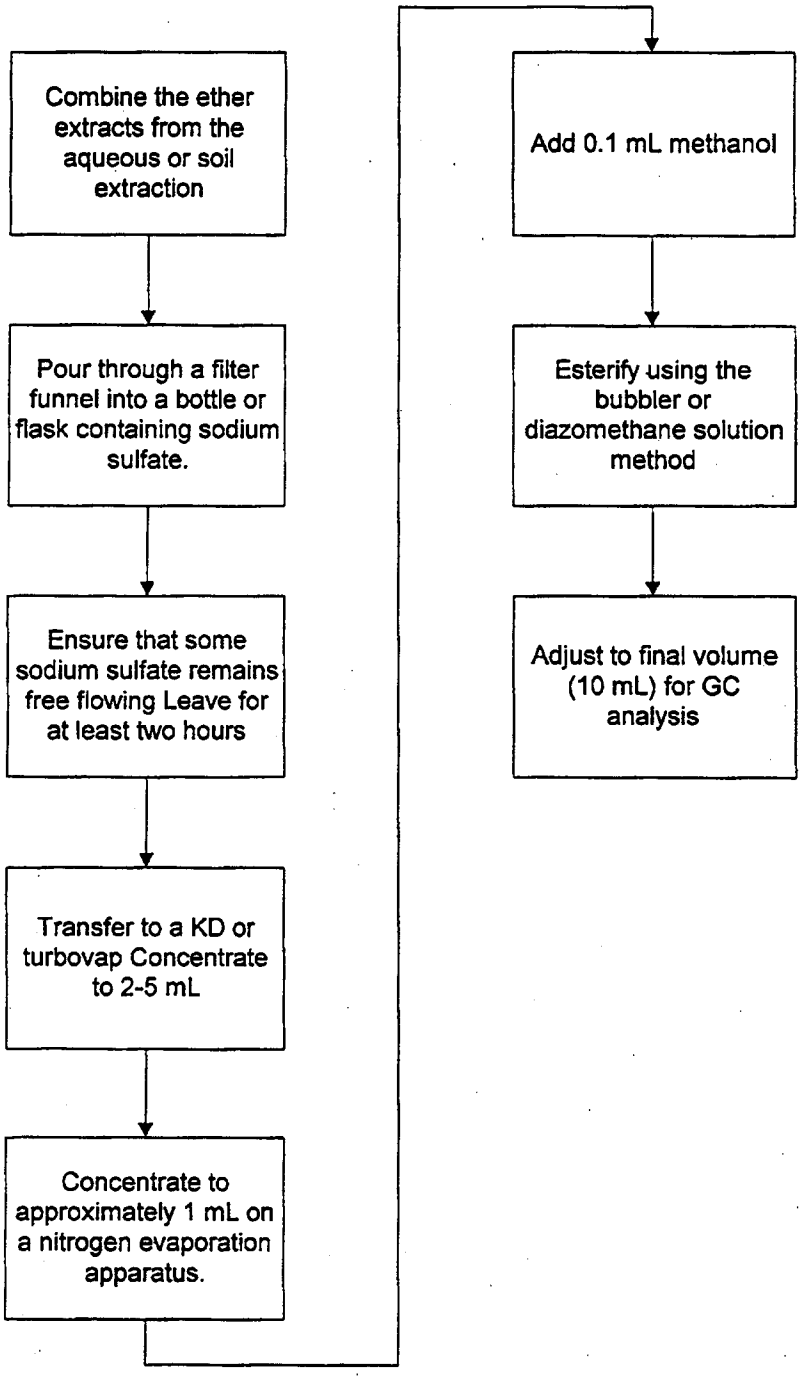
Extraction of Aqueous Samples



Extraction of Soils and Sediments



Drying, Concentration and Esterification



STL STANDARD OPERATING PROCEDURE

TITLE: GC/MS ANALYSIS BASED ON METHODS 8270C AND 625

(SUPERSEDES: Revision 2.1, 01/25/99)

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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.

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 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 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 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 $\leq 6^{\circ}C$ 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 LCSPrecision 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 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.

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 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 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 $\leq$ 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. Use of 1/Concentration^2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. 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%. Otherwise the correlation coefficient (coefficient of determination 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.

10.4.8 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.

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 preceeding 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 QAMP, 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

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

$\overline{RF}$ = 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 $\leq$ 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 8270B.

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 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
2,4-Dinitrotoluene	121-14-2	10	330
2,6-Dinitrotoluene	606-20-2	10	330

Table 1

STL Primary Standard[1] and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
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 primary standard is the standard normally used at STL. 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 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
4-Nitroquinoline-1-oxide	56-57-5	100	3300

Table 2

STL Appendix IX[1] Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous µg/L	Low Soil/Sediment µg/kg
*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 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 3

Reportable Analytes for STL Standard Tests, Primary Standard

Analyte	CAS Number	STL 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
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

Table 3

Reportable Analytes for STL Standard Tests, Primary Standard

Analyte	CAS Number	STL Standard List	TCLP	TCL	Appendix IX
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
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 Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	STL 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
4-Nitroquinoline-1-oxide	56-57-5				X
*Parathion[2]	56-38-2				X

Table 4

Reportable analytes for STL Standard Tests, Appendix IX Standard

Semivolatiles	CAS Number	STL Standard List	TCLP	TCL	Appendix IX
*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

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
Dimethylphthalate	163	194	164
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

Table 7

Analytes in Approximate Retention Time Order and Characteristic Ions, Primary Standard

Analyte	Primary	Secondary	Tertiary
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
Parathion	109	97	291

Table 8

Analytes in Approximate Retention Time Order and Characteristic Ions, Appendix IX Standard

Analyte	Primary	Secondary	Tertiary
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
4,6-Dinitro-2-methylphenol	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
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
*Isodrin	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
*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
4,4'-DDE[1]	60	32.0	19.2-119.7
4,4'-DDT[1]	60	61.6	D-170.6

Table 14
Initial demonstration recovery and precision limits

Compound	Spiking concentration µg/L	Limit for Relative Standard Deviation	Limit for average recovery, %
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
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, 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 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
Butyl benzyl phthalate	85-68-7	10

Analytes	CAS Number	Aqueous µg/L
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
Benzo(a)anthracene	100

LCS Compounds	Spiking Level, ng/µL in extract[1]
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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QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: GAS CHROMATOGRAPHIC ANALYSIS BASED ON METHOD 8000B, 8021B, 8081A, 8082, 8151A, 8310, and 8141A SW-846

(SUPERSEDES: Revision 5.2, Dated 07/19/99)

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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.

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-0001) Solid samples are prepared using sonication, Soxhlet or pressurized fluid extraction (SOP # CORP-OP-0001). 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 Quality Assurance Management Plan (QAMP).

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 Quanterra® associates. The following requirements must be met:

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. Refer to the Quanterra® 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 at ≤6°C. 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 ≤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 ≤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 Quanterra® 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 Quanterra® 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-0001 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 Quanterra® 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. 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; however, if the matrix spike and matrix spike duplicate are within limits; the batch may be acceptable.

9.7.1. Refer to the Quanterra® 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.

9.7.4. 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.

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. Quanterra® QC Program

Further details of QC and corrective action guidelines are presented in the Quanterra® 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 ≤ 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 ≤ 20%.

The equation for average response factor is:

$$\text{Average response factor} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

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 ± 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 ≤ 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.

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 each continuing calibration must be within the retention time windows established by the 12-hour calibration. 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. (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, but not for any other calibration verification standards.

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-0001 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 will only be reported at client request.

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

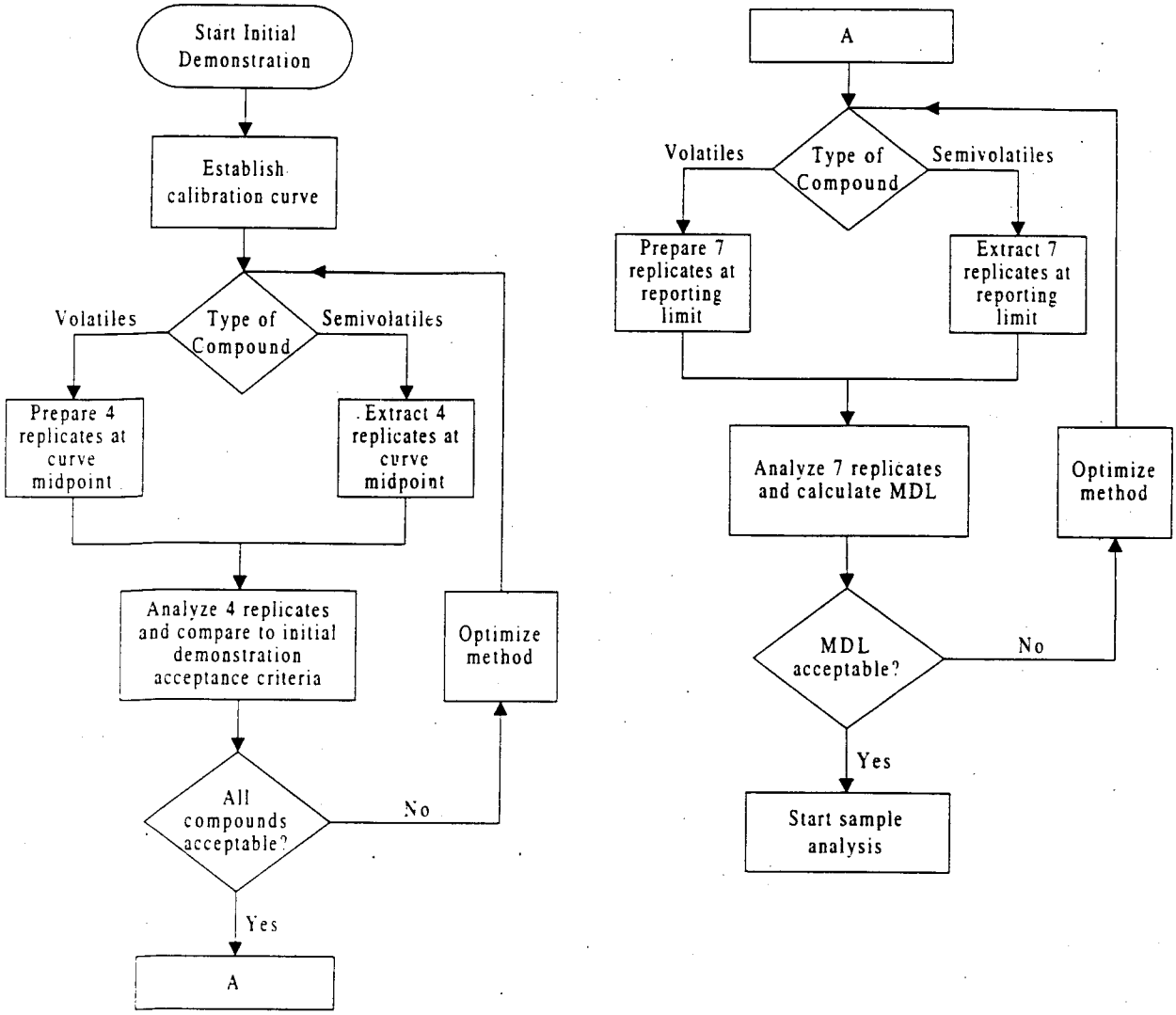
The calibration criteria in section 10.11 have been rewritten to improve consistency with SW-846 and to improve clarity.

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.

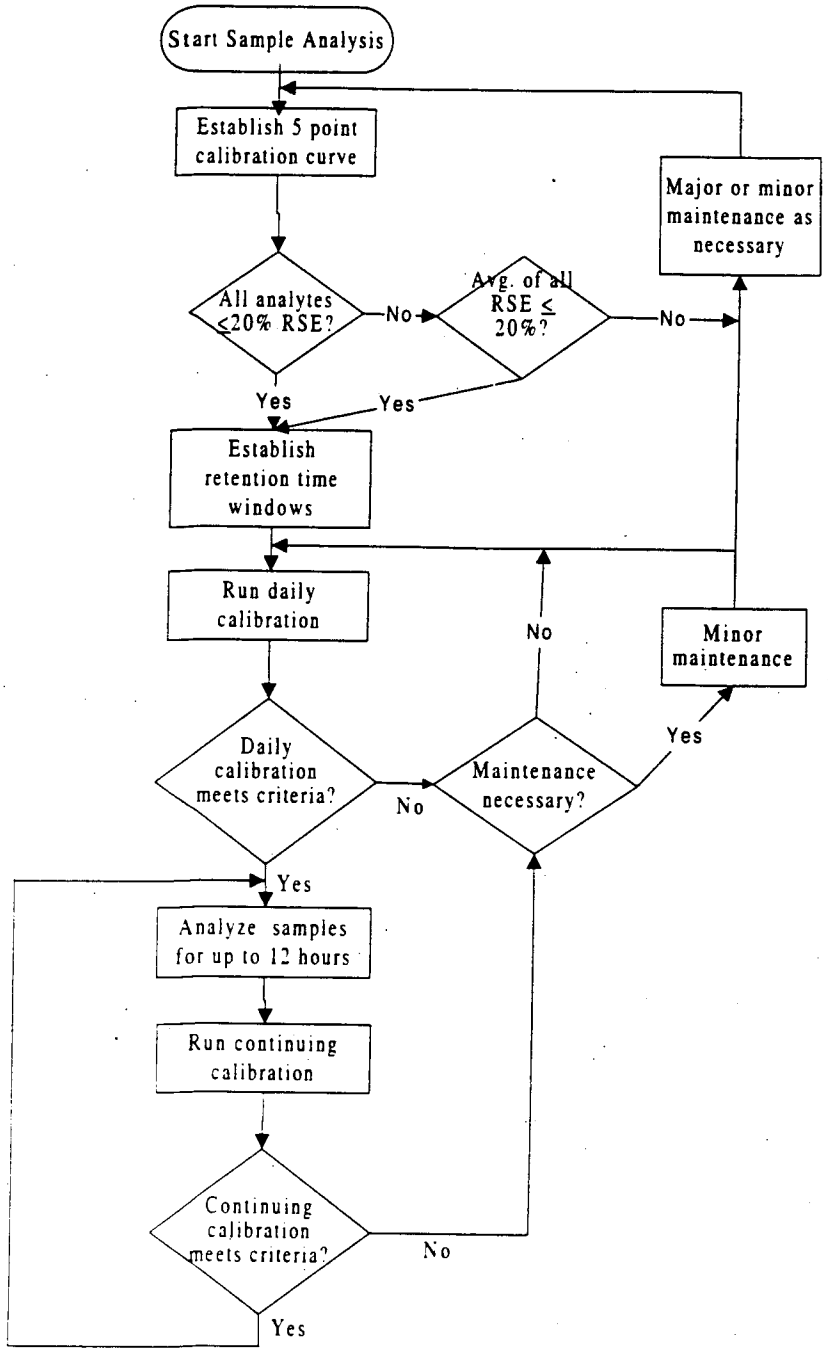
17.4. Flow Diagrams

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.

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 QAMP 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. 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 $\geq$ -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.

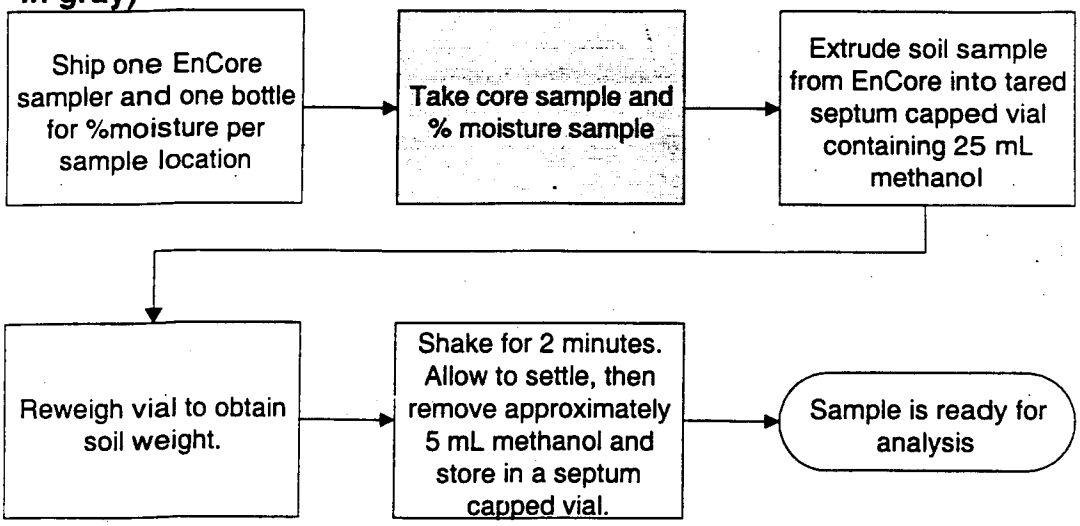
8.8. Aqueous samples are stored in glass containers with Teflon lined septa at 4°C +/- 2°C, with minimum headspace.

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.

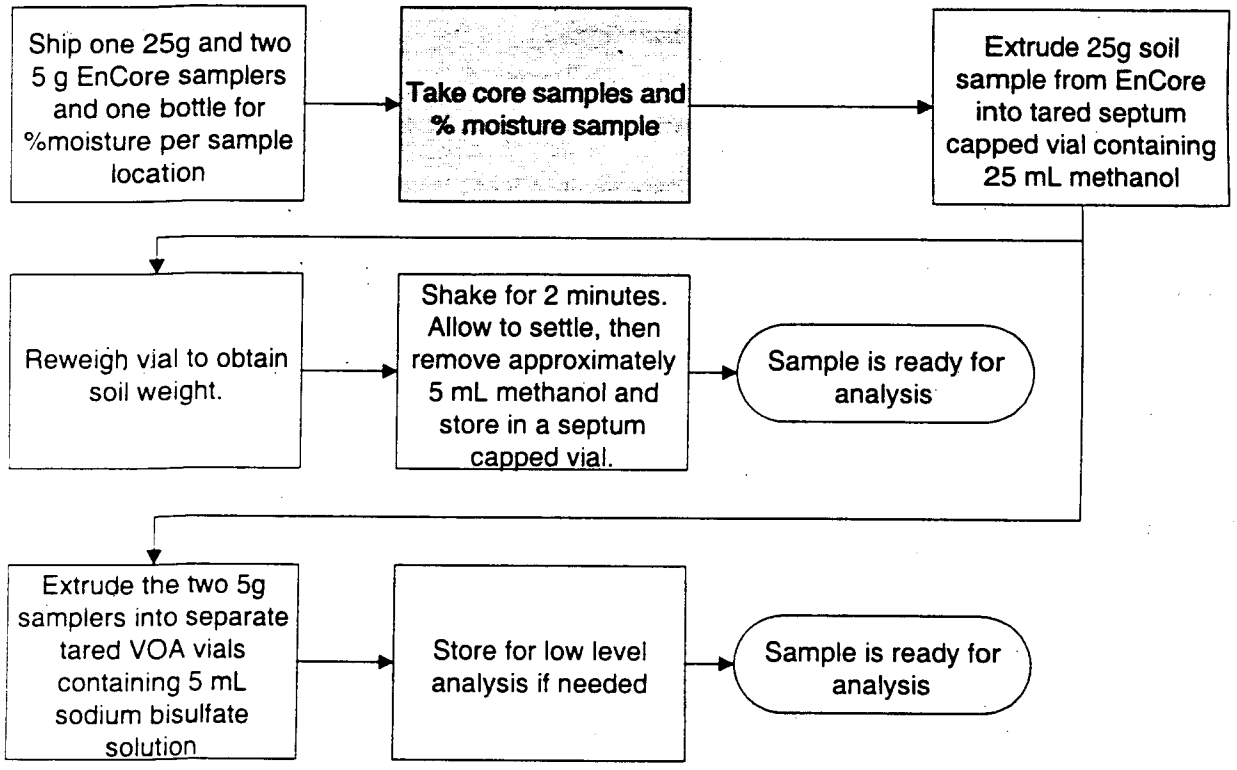
8.10. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14 day holding time. However they should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative). Maximum holding time 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.

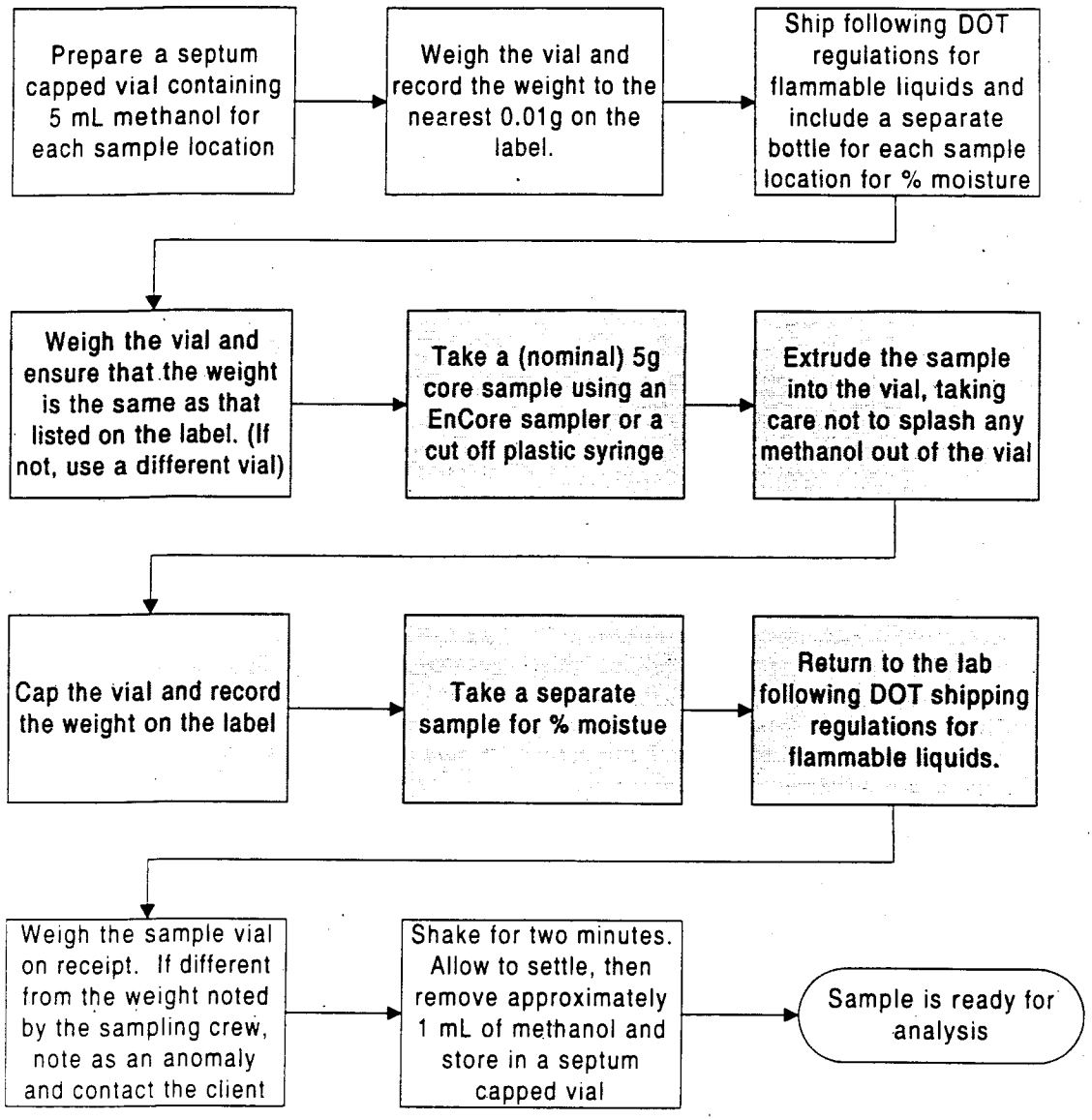
EnCore procedure when low level is not required (field steps in gray)



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 <u>after</u> 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
	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 volatiles by 8021B	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
	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
	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

Table A-1
Standard Analyte List

Test	Compound	CAS number	Reporting Limit, µg/L or µg/kg		
			Aqueous	Low Soil	Medium Soil
	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 Quanterra® sample extraction SOPs. (CORP-OP-0001)

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.

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 QAMP 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-0001.

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-0001. Use or 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. 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 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.

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-8 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.

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-0001.

11.3. Cleanup

Cleanup procedures are described in SOP No. CORP-OP-0001.

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-8.

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) unless it is determined that sample interference has adversely affected the quantitation of that surrogate. Tetrachloro-m-xylene (TCMX) recovery is reported in lieu of DCB in samples having the interference, and also may be used in samples in which DCB recovery is low. Corrective action is only necessary if DCB and TCMX are both outside of acceptance limits.

12.6. Calculation of Column Degradation/% Breakdown (%B)

$$DDT\ \%B = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100$$

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.

$$Endrin\ \%B = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_{E}} \times 100$$

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. 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

SW846, Update III, December 1996, Method 8081A

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
Chlorobenzillate	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	6.67	200
Aldrin	0.20	6.67	200
Heptachlor	0.20	6.67	200
Dieldrin	0.50	16.7	500
Endrin	0.50	16.7	500
4,4'DDT	0.50	16.7	500
Tetrachloro-m-xylene (Surrogate)	0.20	6.67	200
Decachlorobiphenyl (Surrogate)	0.20	6.67	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]
Solvent blank	
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).

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

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 Quanterra® sample extraction SOPs. (CORP-OP-0001). The PCBs are determined and quantitated as Arochlor mixes.

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.

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 QAMP 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-0001.

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-0001.

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. All standards for this method must be replaced

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.

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 should be included in the daily calibration check.

10.3.3. The retention time windows for any analytes included in the daily calibration 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.

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-0001.

11.3. Cleanup

Cleanup procedures are described in SOP No. CORP-OP-0001.

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. However, if the pattern is not clear, or if no historical data for the site is available, then second column confirmation must be performed.

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, or if the pattern is not clear or there is no historical data leading to a suspicion that Arochlors may be present. 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) unless it is determined that sample interference has adversely affected the quantitation of that surrogate. Tetrachloro-m-xylene (TCMX) recovery is reported in lieu of DCB in samples having the interference, and also may be used in samples in which DCB recovery is low. 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

SW846, Update III, December 1996, Method 8082

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
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	Solvent blank	
12-31	Sample 1-20 (or as many samples as can be analyzed in 12 hours	
	Solvent blank (optional)	
32	Aroclor 1016/1260	Level 3

etc

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-0001. 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.

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 QAMP 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-0001.

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.

7.2. Refer to Table D-3 and D-4 for details of calibration and other 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-5 for minimum performance criteria for the initial demonstration of capability.

9.3. Refer to Table D-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 the free acids, and then esterified using the same process as for samples (See SOP Corp-OP-0001)

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-0001.

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 Quanterra® sample extraction SOPs. (CORP-OP-0001).

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.

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 QAMP 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.

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.

10.2. Calibration standards are made up 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-0001.

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.

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

SW846, Update III, December 1996, Method 8310.

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	5
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.	15	85	2.0 mL/m
	25 min.	5	95	2.0 mL/m
	30 min.	45	55	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	Compound
Acenaphthene	Dibenzo(a,h)anthracene
Acenaphthylene	**Fluoranthene**
Anthracene	Fluorene
Benzo(a)anthracene	**Indeno(1,2,3-cd)pyrene**
Benzo(a)pyrene	**Naphthalene**
Benzo(b)fluoranthene	**Phenanthrene**
Benzo(g,h,i)perylene	Pyrene
Benzo(k)fluoranthene	Terphenyl-d14*
Chrysene	Benzo(e)pyrene*

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
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	
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.

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.

3. DEFINITIONS

Refer to the QAMP 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.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to Section 6 of the 8000B section of this SOP.

6.2. Refer to Table F1 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

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 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-0001.

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	

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: ACID DIGESTION OF AQUEOUS SAMPLES BY SW846 AND MCAWW 200 SERIES METHODS

(SUPERSEDES: NONE)

Prepared by: _____
Technology Standardization Committee

Approved by: _____
Director, Quality Assurance

Approved by: _____
Director, Environmental Health and Safety

Approved by: _____
Management

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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) and Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) 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, solids, sludges, wastes, sediments, air sampling media, biological tissue 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.

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

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 tc volume.

2.2. Method 3010A / Method 200.7 Section 9.3 - Preparation for Total Metals Analysis by FLAA or ICP Spectroscopy

A representative aliquot of sample is refluxed with nitric acid. This step is repeated until the digestate is light in color or until its color has stabilized. 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

A representative aliquot of sample is refluxed with nitric acid. This step is repeated until the digestate is light in color or until its color has stabilized. 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

A representative aliquot of sample is heated with nitric acid and peroxide until the digestion is complete or until the volume is reduced by one-half. 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)

A representative aliquot of sample is heated with nitric acid until the volume is reduced to 15 - 20 mL. 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 Quanterra 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. 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 Quanterra 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 or other adjustable heating source capable of maintaining a temperature of 90 - 95°C.

6.2. 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. 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 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 pipet 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 Quanterra 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: The ICP LCS/MS working spike solution is provided directly by the vendor, no further standard preparation is necessary.

7.4. Working GFAA LCS/MS spike solution: Prepare the GFAA working LCS spike solution by diluting the custom stock solution (7.2) 100x. 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 Quanterra 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 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.

7.8. 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 <u>always</u> 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 Quanterra 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 Quanterra 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 0.5 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 0.5 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 which 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 preparation 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 and record 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 (See Section 17.1.1.2).

11.10.4. Measure two extra aliquots of sample selected for the MS/MSD analysis. Spike each aliquot with 0.5 mL of spiking solution (7.3 or 7.4).

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 0.5 mL of spiking solution (7.3 or 7.4)

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 90 - 95°C 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, though Whatman 41 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. Add 1.5 mL concentrated HNO_3 and 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 1.5 mL of concentrated HNO_3.

11.13.2. Cover with ribbed watch glass.

11.13.3. Place beaker on hotplate (90-95 °C) and evaporate to low volume of 5 - 10 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. Cool the beaker in a fume hood.

11.13.5. Add another 1.5 mL portion of concentrated HNO_3 and re-cover the beaker.

11.13.6. Continue refluxing until the digestion is complete.

Note: Digestion is complete when the digestate is light in color or does not change in appearance. For most samples the addition of two nitric acid aliquots is sufficient, additional aliquots of nitric acid may be added if necessary.

11.13.7. Evaporate to low volume of 5 - 10 mL while ensuring that no portion of the bottom of the beaker is allowed to go dry.

11.13.8. Cool the beaker in a fume hood.

11.13.9. Add 5 mL of 1:1 HCl.

11.13.10. Cover and reflux for an additional 15 minutes to dissolve precipitate or residue.

11.13.11. Wash down beaker walls and watch glass with reagent water.

11.13.12. Filter sample, if insoluble materials are present, through Whatman 41 filter paper that has been pre-rinsed with dilute nitric acid (1%).

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.

11.13.13. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

11.13.14. 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 (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 (90-95 °C) and evaporate to low volume of 5 - 10 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. Cool the beaker in the fume hood.

11.14.5. Add another 1.5 mL portion of concentrated HNO_3.

11.14.6. Recover with watch glass.

11.14.7. Continue refluxing and adding acid as necessary until digestion is complete.

Note: Digestion is complete when the digestate is light in color or does not change in appearance. For most samples the addition of two nitric acid aliquots is sufficient, additional aliquots of nitric acid may be added if necessary.

11.14.8. After the digestion is complete evaporate to low volume of 5 - 10 mL while ensuring that no portion of the bottom of the beaker is allowed to go dry.

11.14.9. Cool the beaker in the fume hood.

11.14.10. Add 10 mL of reagent water and mix sample.

11.14.11. Heat sample for 10 to 15 minutes more to dissolve any residue.

11.14.12. Cool the beaker in the fume hood.

11.14.13. Wash down beaker walls and watch glass with reagent water.

11.14.14. Filter sample, if insoluble materials are present, though Whatman 41 filter paper that has been pre-rinsed with dilute nitric acid (1%).

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.

11.14.15. Rinse beaker and filter paper with reagent water to ensure complete sample transfer.

11.14.16. 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 0.5 mL of concentrated HNO_3.

11.15.2. Heat, until the digestion is complete, at 90 - 95°C or until the volume has been reduced to slightly less than 25 mL.

11.15.3. Cool beaker.

11.15.4. Filter sample, if insoluble materials are present, though Whatman 41 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 0.5 mL of concentrated HNO_3.

11.16.2. Heat, until the digestion is complete, at 90 - 95°C 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 41 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.

11.16.5. 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, Quanterra 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. 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.

17.1.1.3. 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 3005A

17.1.2.1. In order to matrix match the digestate to the ICP calibration standards, Section 11.12.8 requires the addition of 1.5 mL of concentrated nitric acid to the digestate prior to dilution to final volume. This step ensures that bias due to differences in acid matrix will not be a factor in the analytical determination. Since this step is performed post-digestion it does not impact the digestion recoveries. This approach to matrix matching was discussed with Olliver Fordham of OSW who indicated that it was an acceptable practice.

17.1.3. Modifications Specific to Method 3010A

17.1.3.1. Section 11.13.7 of this SOP requires the sample be reduced to a volume of 5 - 10 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.

17.1.3.2. 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.

17.1.4. Modifications Specific to Method 3020A

17.1.4.1. Section 11.14.8 of this SOP requires the sample be reduced to a volume of 5 - 10 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.

17.1.5. Modifications Specific to Method 7060A/7740

17.1.5.1. 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.

17.1.6. 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

None.

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 2. Method 3010A / Method 200.7 Section 9.3 (Section 11.13) .

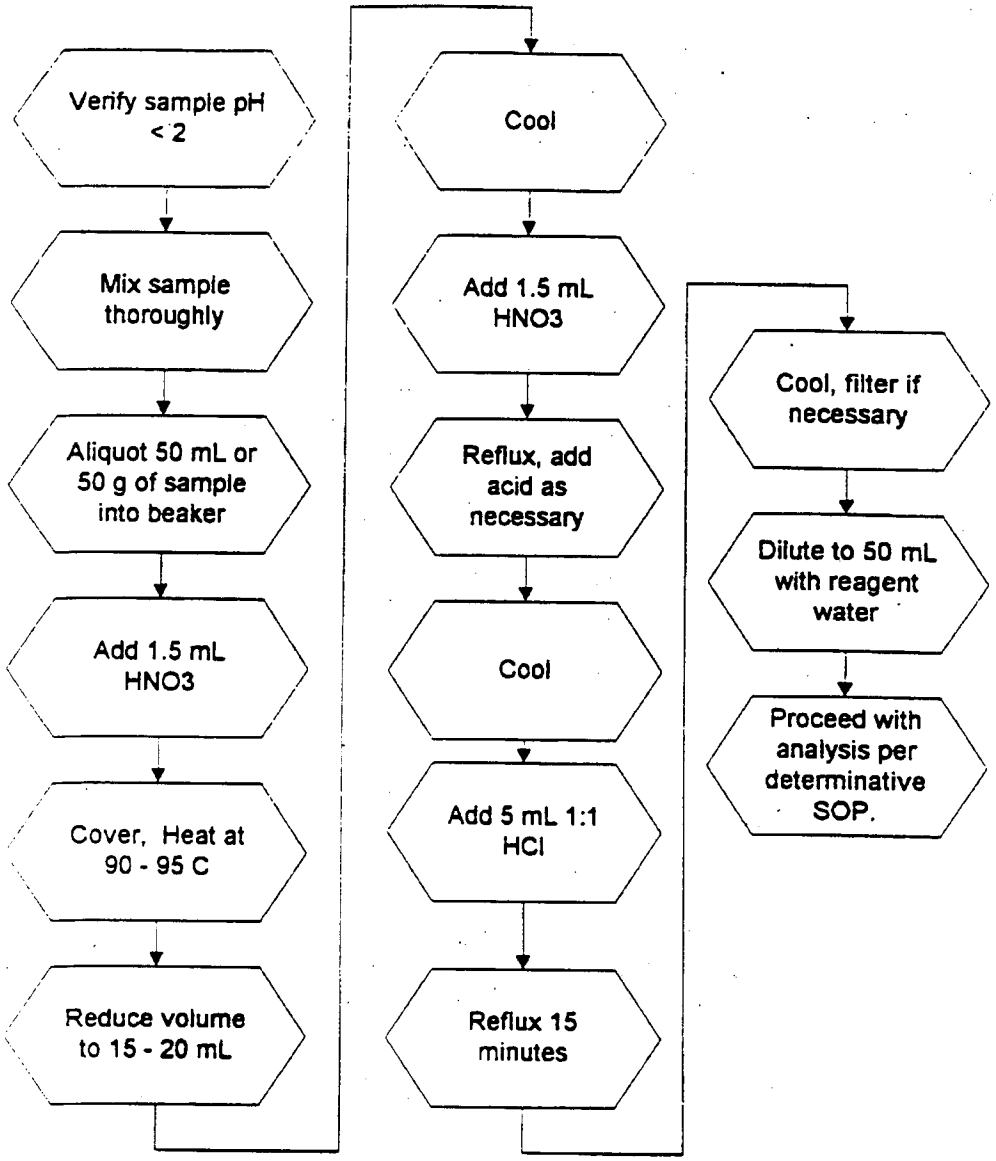


Figure 1. Method 3005A / Method 200.7 Section 9.4 (Section 11.12)

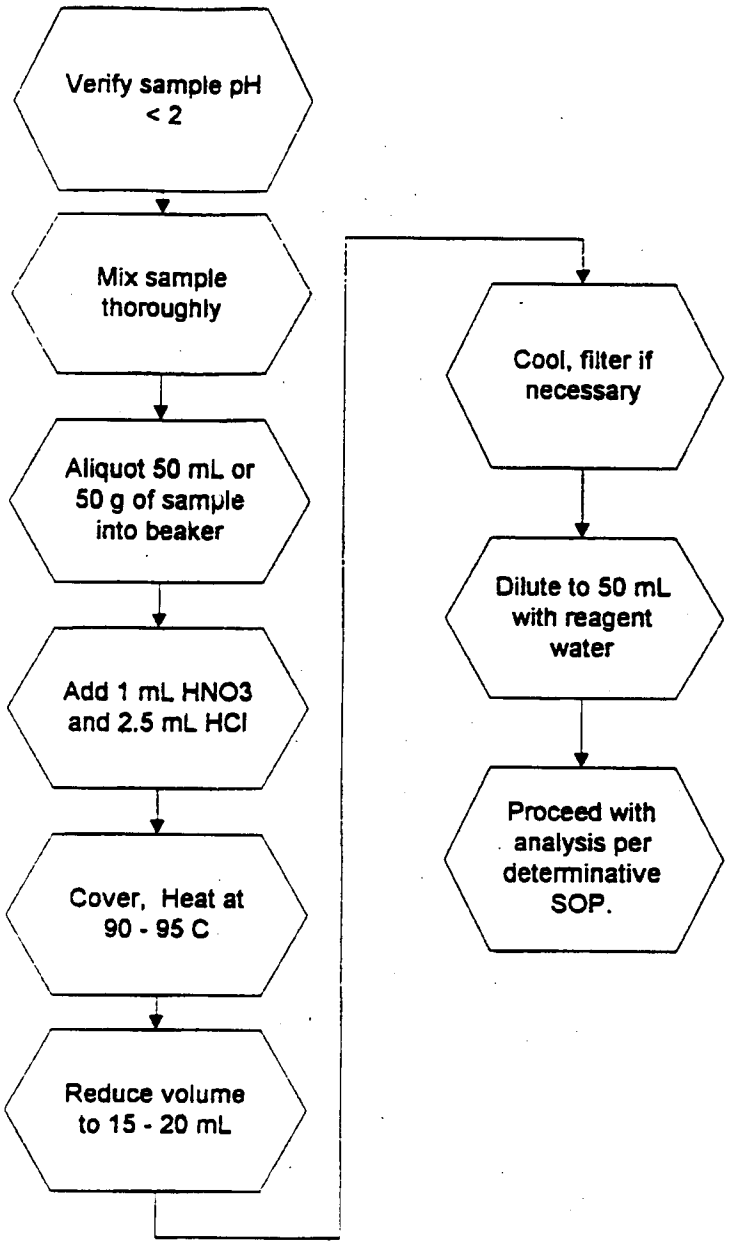


Figure 3. Method 3020A / Method 200.0 Section 4.1.3 (Section 11.14)

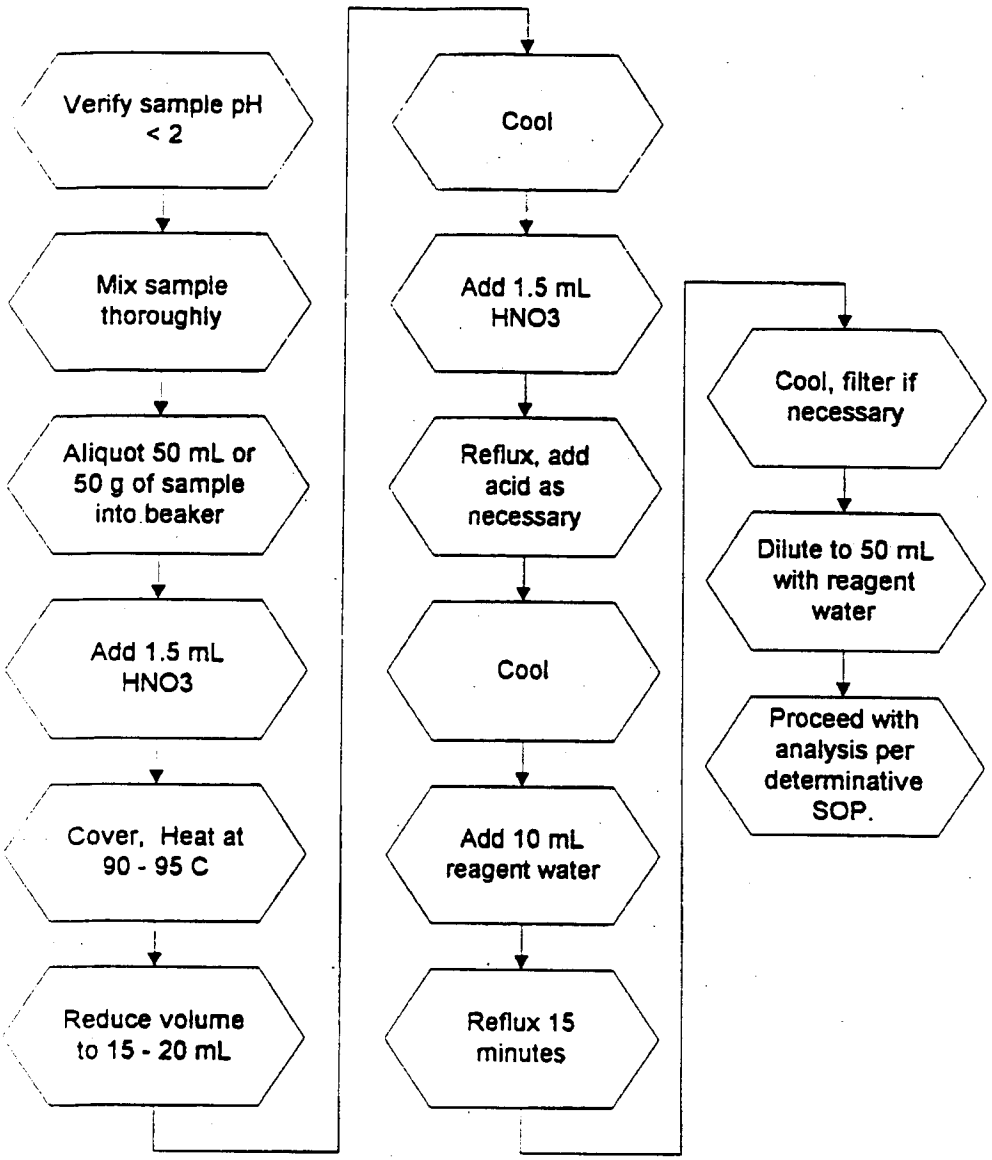


Figure 4. Method 7060A/7740A and Method 206.2/270.2 (Section 11.15)

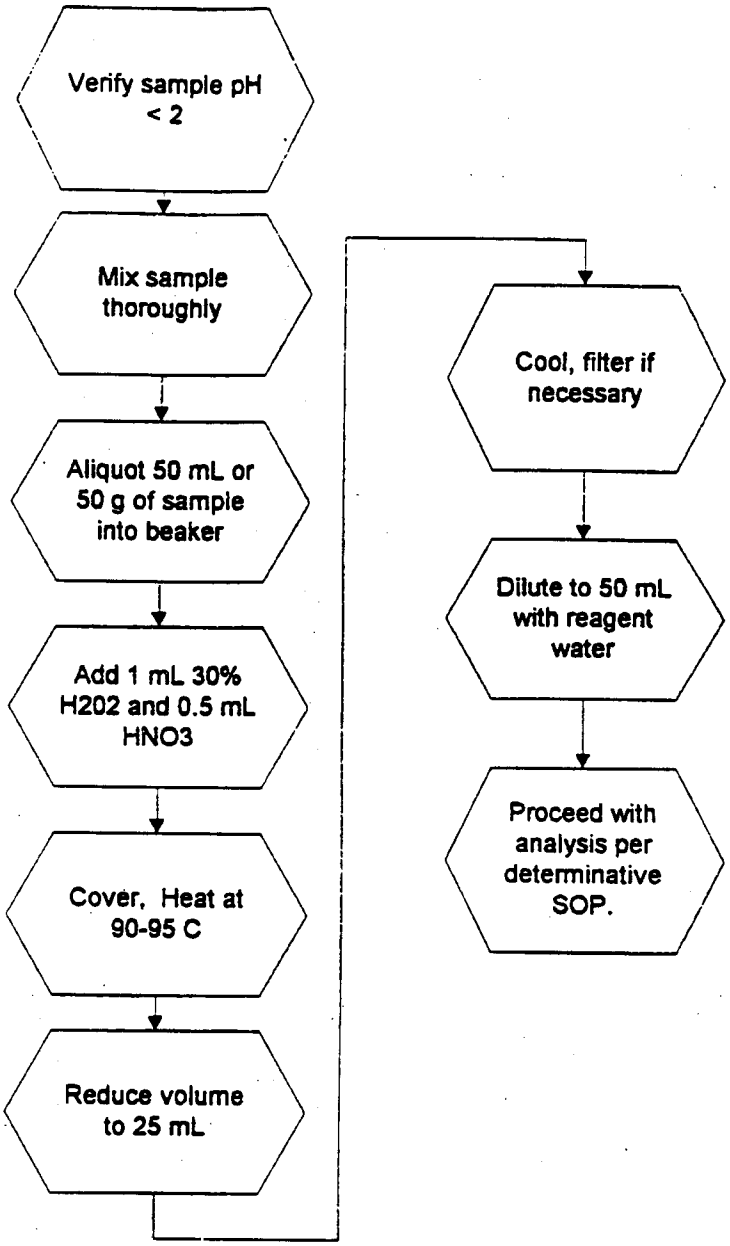


Figure 5. Method 200.0 Section 4.1.4 (Section 11.16)

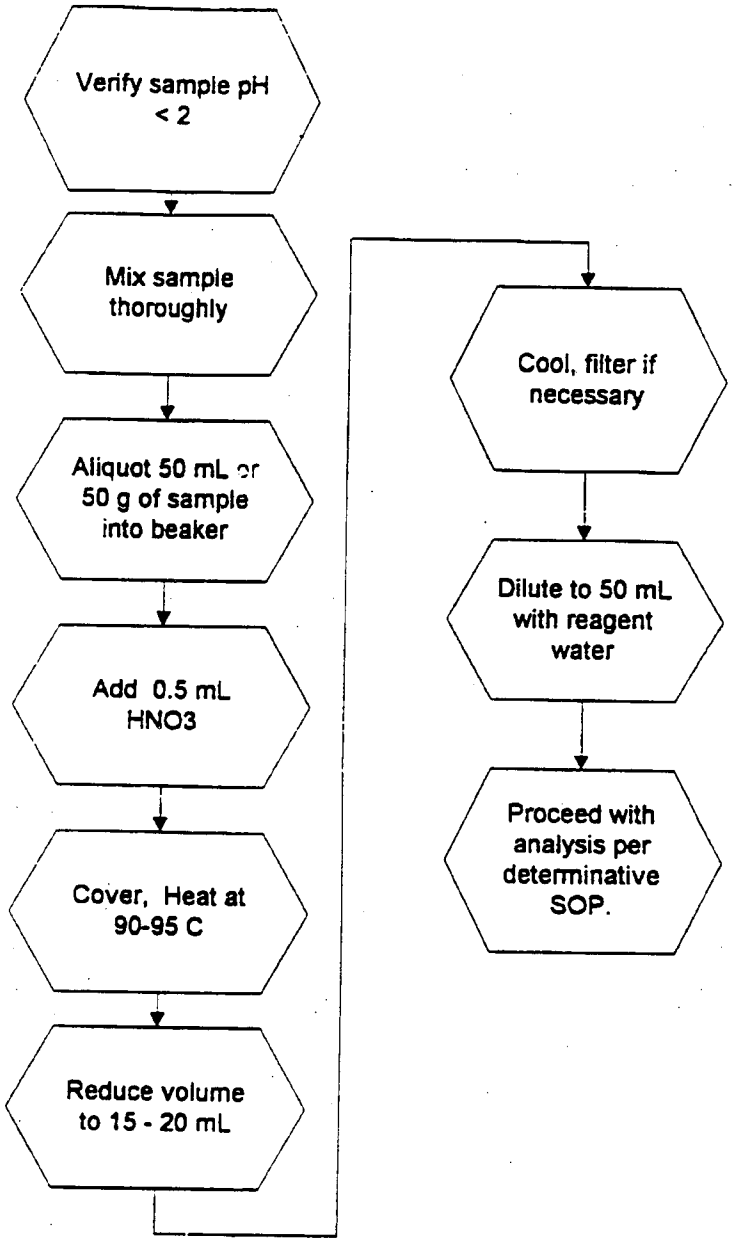
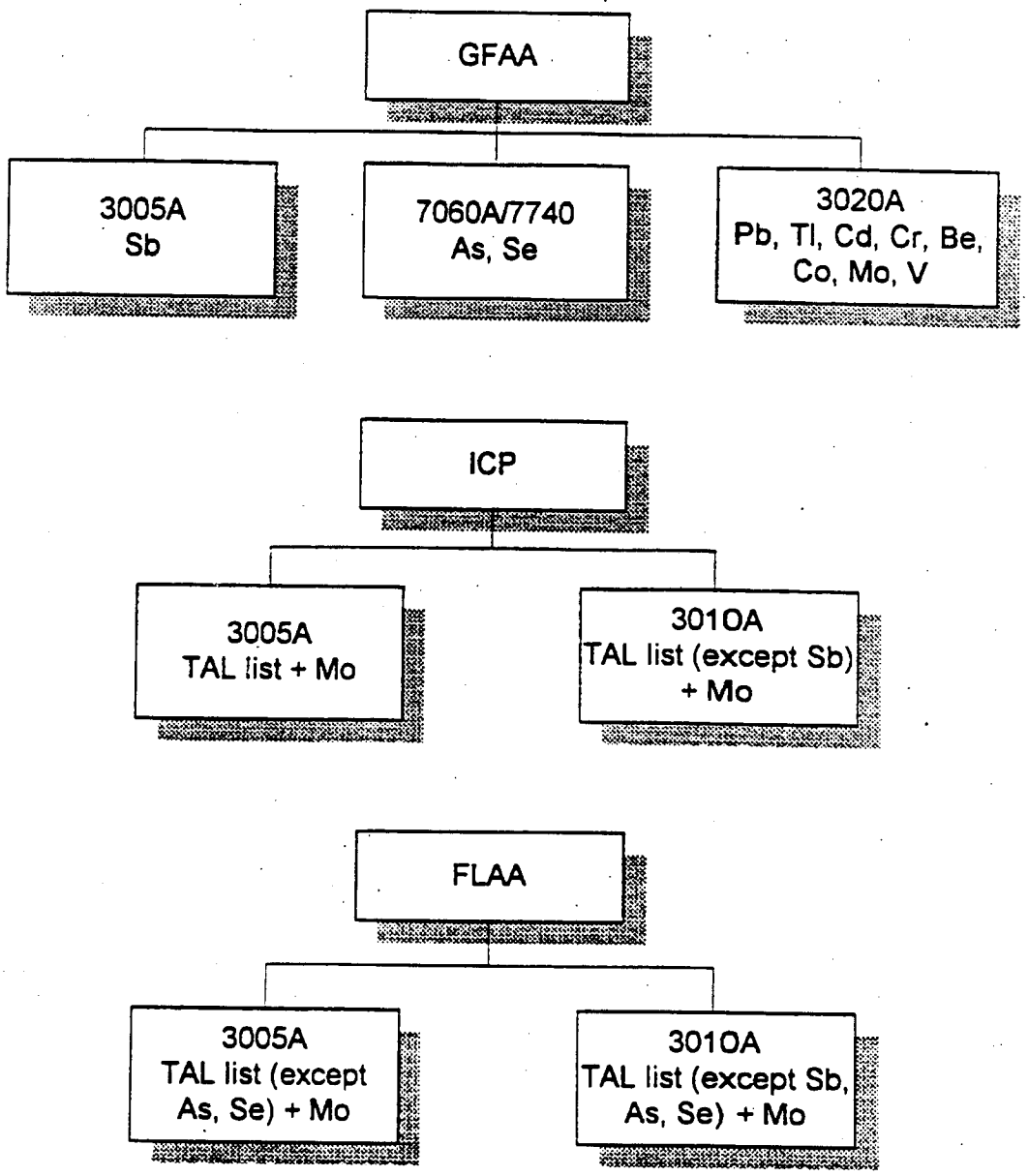
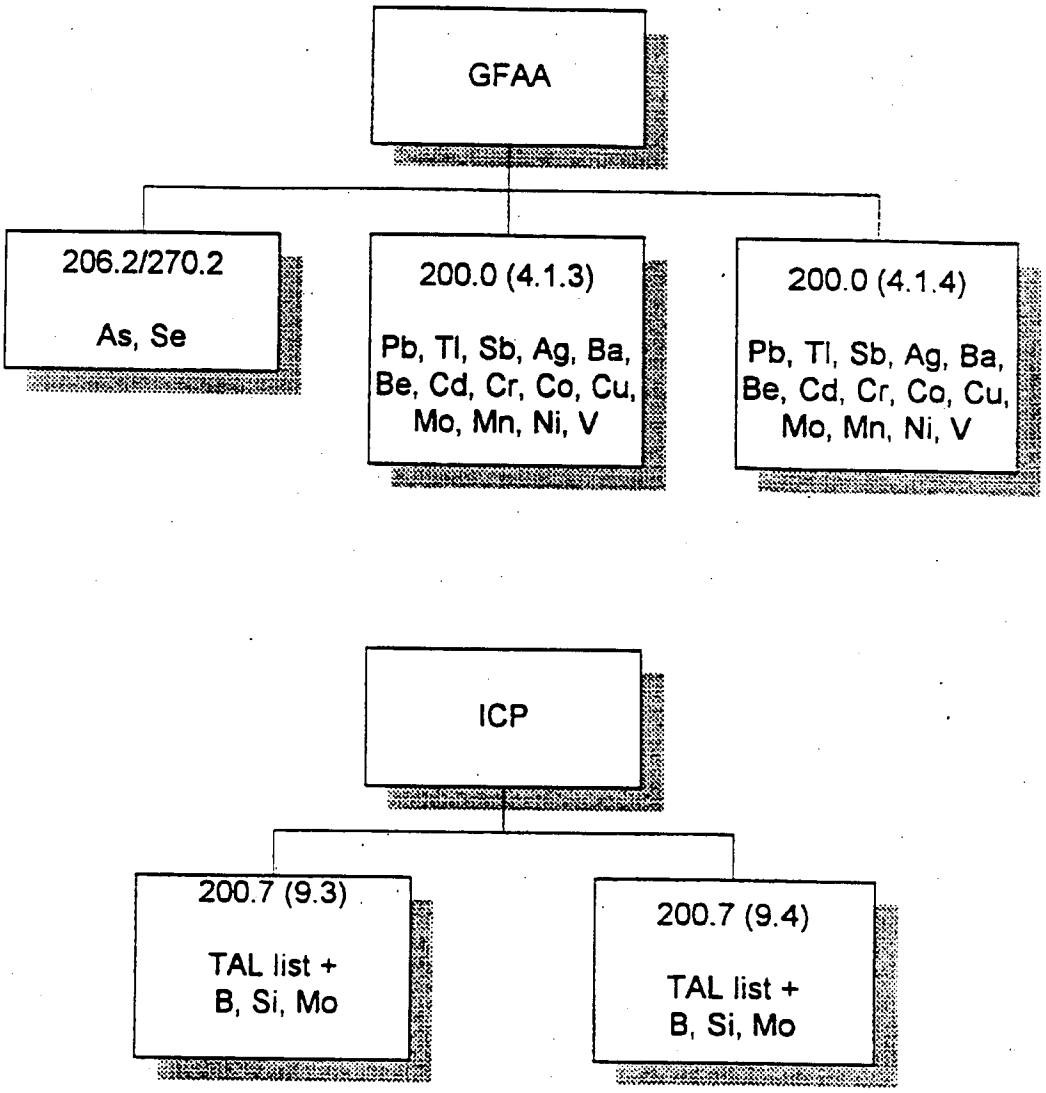


Figure 6. Overview of SW846 Aqueous Preparation Methods by Determinative Method



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	X	
Molybdenum	Mo	7439-98-7	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	200	2000
Antimony	50	500
Arsenic	200	2000
Barium	200	2000
Beryllium	5	50
Cadmium	5	50
Calcium	5000	50000
Chromium	20	200
Cobalt	50	500
Copper	25	250
Iron	100	1000
Lead	50	500
Lithium	100	1000
Magnesium	5000	50000
Manganese	50	500
Molybdenum	100	1000
Nickel	50	500
Phosphorous	1000	10000
Potassium	5000	50000
Selenium	200	2000
Silver	5	50
Sodium	5000	50000
Strontium	100	1000
Thallium	200	2000
Vanadium	50	500
Zinc	50	500
Boron	100	1000
Silica	1000	10000
Tin	200	2000
Titanium	100	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 (7.3) 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	4000	40
Selenium	400	4000	40
Lead	400	4000	40
Thallium	400	4000	40
Antimony	400	4000	40
Cadmium	40	400	4
Chromium	100	1000	10
Silver	50	500	5

* 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 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

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

QUANTERRA METALS PREP BENCHSHEET

Quanterra Metals Preparation Log

Prep Date : _____ Matrix : _____ QC Batch: _____ SOP (circle) : CORP-IP 0001 Rev 0
Analyst : _____ Prep Type (circle): ICP GFAA Method #: _____ CORP-IP-0002 Rev 0
CORP-IP-0003 Rev 0
Other _____

SPIKE	ID	LOT #	Prep Date	Vol. Added	Custom Spike/Comments
LCS					
MS/MSD					

#	Sample ID	Initial Wt (g)/Vol(mL)	Final Wt (g)/Vol(mL)	Comments
BLK				
LCS				
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
MS				
MSD				

Analyst Signature: _____ Date: _____ Reviewed by: _____ Date: _____

CHAIN-OF-CUSTODY INFORMATION FOR DIGESTATES:
Relinquished By: Date: Received By: Date:

APPENDIX C

CONTAMINATION CONTROL GUIDELINES

APPENDIX C. 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.

Controlled Copy
Copy No. _____
Implementation Date: _____

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SOP No. CORP-IP-0002DEN
Revision No. 2
Revision Date: 01/12/00

QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: ACID DIGESTION OF SOILS, SW846 METHOD 3050B

(SUPERSEDES: CORP-IP-0002 REV 1)

Prepared by: Kurt Hill and Richard Clinkscales

Reviewed by: 1/12/2000
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Approved by: 1-12-00
Larry Penfold, Quality Assurance

Approved by: 1-13-00
Scott Kelly, Health and Safety

Approved by: 1-13-00
Timothy O'Shields, Laboratory Manager

Approved by: 1-13-00
Kurt Hill, Corporate Technology

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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), Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP), and Inductively Coupled Plasma-Mass Spectrometry (ICP/MS) as specified in SW846 Method 3050B. Additional elements (B, Si, Sn, and Ti) may also be included in the digestion scheme, but see caveat in 1.5 below.

1.2. Samples prepared by the protocols detailed in this SOP may be analyzed by ICP, ICP/MS 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. 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.

1.4. 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.

1.5. Silicon or silica are occasionally requested as part of the Method 3050B digestion. However, this digestion will only include acid soluble silicon, and will not dissolve crystalline silica. The analysis is for silicon, with the final result is sometimes expressed as silica rather than silicon.

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 or antimony by GFAA analysis. The digestates are then filtered and diluted to 100 mL/100 g.

3. DEFINITIONS

Additional definitions of terms used in this SOP may be found in the glossary of the QAMP.

3.1. 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. Refer to Appendix D for additional contamination control guidelines.

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 Quanterra® 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 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 Quanterra® 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 90-95°C.

6.2. 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. Repipetors or suitable reagent dispensers.

6.11. Calibrated automatic pipettes with corresponding pipet 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 Quanterra® 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: The ICP LCS/MS working spike solution is provided directly by the vendor, no further standard preparation is necessary.

7.4. Working GFAA LCS/MS spike solution: Prepare the GFAA working LCS/MS spike solution by diluting the custom stock solution (7.2) 100x. 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 Quanterra® 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. MDL's must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in Quanterra® 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 Quanterra™ 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.

9.4.1. Soil method blanks are prepared by taking 1 mL or 1 g of reagent water through the procedure described in Section 11.10.

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 1 mL or 1 g aliquot of reagent water with 1 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 1 mL 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 the metals preparation log. 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. All preparation procedures must be carried out in a properly functioning hood.

11.4. Custody of samples is transferred from the Sample Receiving group to the Metals group, which is documented using an internal Chain-Of-Custody form provided by Sample Receiving. One exception is AFCEE program samples, which are documenting in the internal custody records maintained in the books in the Sample Receiving area.

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. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards.

11.9. Preparation of Soils, Sediments and Sludges for Analysis by GFAA, ICP, and ICP/MS.

11.9.1. Mix sample thoroughly by stirring with a clean plastic or wooden spoon or spatula.

11.9.2. 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.

11.9.3. Measure additional aliquots of the designated samples for the MS and MSD analyses.

11.9.4. Add 10 mL of 1:1 HNO_3 and mix the sample. Add 10 mL of 1:1 HNO3 to the tubes used for the method blank, and LCS.

11.9.5. Spike each of the MS and MSD aliquots with 1 mL of the working spiking solutions (7.3 or 7.4).

11.9.6. Spike the LCS aliquot with 1 mL of the working spiking solution (7.3 or 7.4).

11.9.7. Heat sample to 95°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.

11.9.8. Allow sample to cool.

11.9.9. Add 5 mL of concentrated HNO_3 and replace vapor recovery device.

11.9.10. Reflux at 95°C for 30 minutes.(Add reagent water as needed to ensure that the volume of solution is not reduced to less than 5 mL.)

11.9.11. If brown fumes are observed, repeat step 11.10.10 until no more fumes are evolved.

11.9.12. Using a vapor recovery device, allow the sample to evaporate to 5 - 10 mL while ensuring that no portion of the bottom of the beaker is allowed to go dry. Alternatively heat at 95°C for 2 hours.

11.9.13. Allow the samples to cool.

11.9.14. Add 2 mL of reagent water and 3 mL of 30 % H_2O_2. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

11.9.15. Replace the vapor recovery device and heat sample until effervescence subsides.

11.9.16. Allow the sample to cool.

11.9.17. 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 6 mL of 30 % H_2O_2.

11.9.18. Continue heating at 95°C until the volume is reduced to approximately 5 mL. Alternatively the sample may be heated for 2 hours.

11.9.19. Allow the sample to cool. This reduces the possibility fo a violent reacion when HCl is added.

11.9.20. If the sample is being prepared for ICP analyses add 10 mL of concentrated HCl and reflux for an additional 15 minutes without boiling. This step is omitted for analysis by ICP-MS and GFAA

11.9.21. Allow the sample to cool.

11.9.22. Wash down beaker walls and vapor recovery device with reagent water.

11.9.23. Filter sample through Whatman 41 filter paper or equivalent into a graduated cylinder or 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

11.9.24. 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. QA-003, Quanterra® 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. 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.

17.2. Modifications from previous SOP

17.2.1. ICP/MS has been added as an appropriate determinative technique.

17.2.2. The table listing appropriate elements has been removed. Any elements meeting the requirements in section 13 may be determined.

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 11.9.20)

17.2.5. Definition of the method as determining total environmentally available metals has been added.

17.3. Facility Specific SOPs

17.3.1. Flame atomic absorption is not performed at this lab. References to FLAA have been omitted.

17.3.2. An explanation of the limitations of analysis for silicon by this method was added to the Scope and Application section.

17.3.3. Details were added to Section 11.4 describing the Denver lab's documentation for internal transfer of sample custody.

17.3.4. Nitric acid is added to samples prior to the addition of matrix spikes. This is done in the interest of safety. This avoids spattering of the acidic spike solution with calcareous or highly alkaline solids.

17.3.5. A cooling step was added in Section 11.9.19 in the interest of safety.

17.3.6. Osmium is deleted from the analyte list in Table 1.

17.3.7. Spike levels are changed to those in use at the Denver lab. Most are lowered by a factor of two.

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. Soil Sample Preparation (Section 11.10)

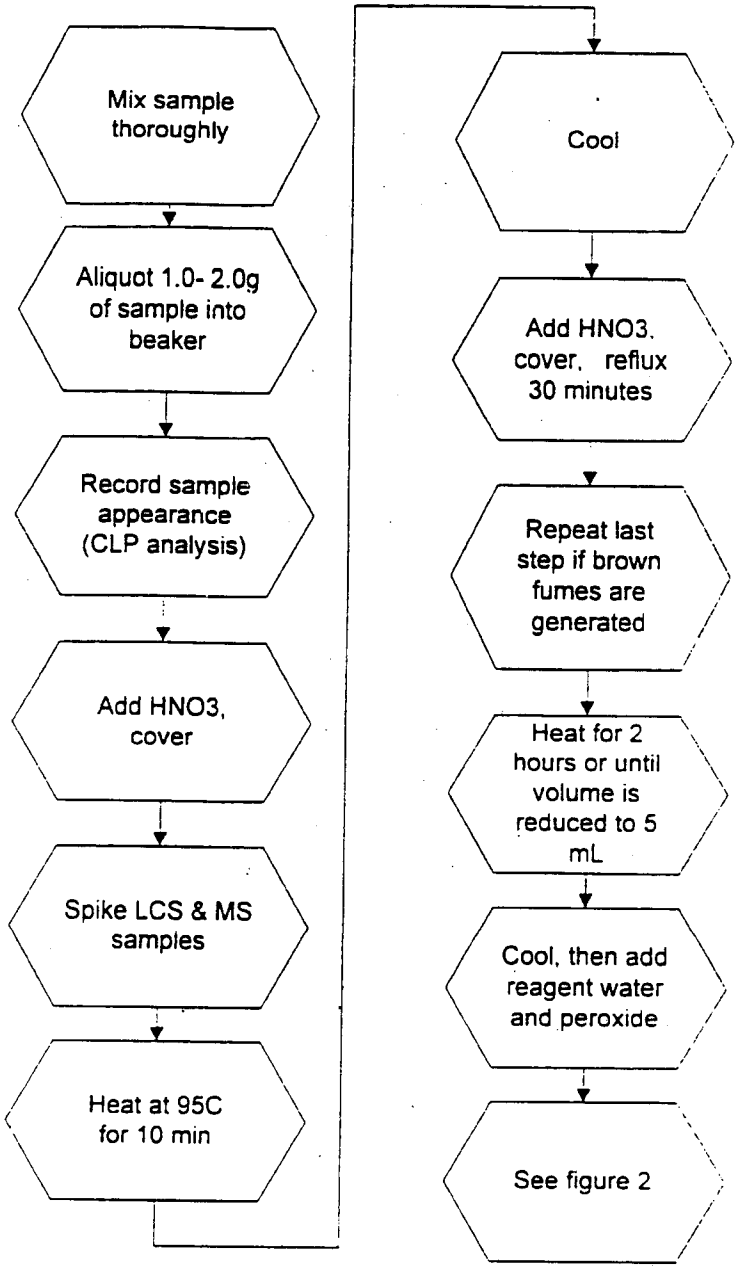
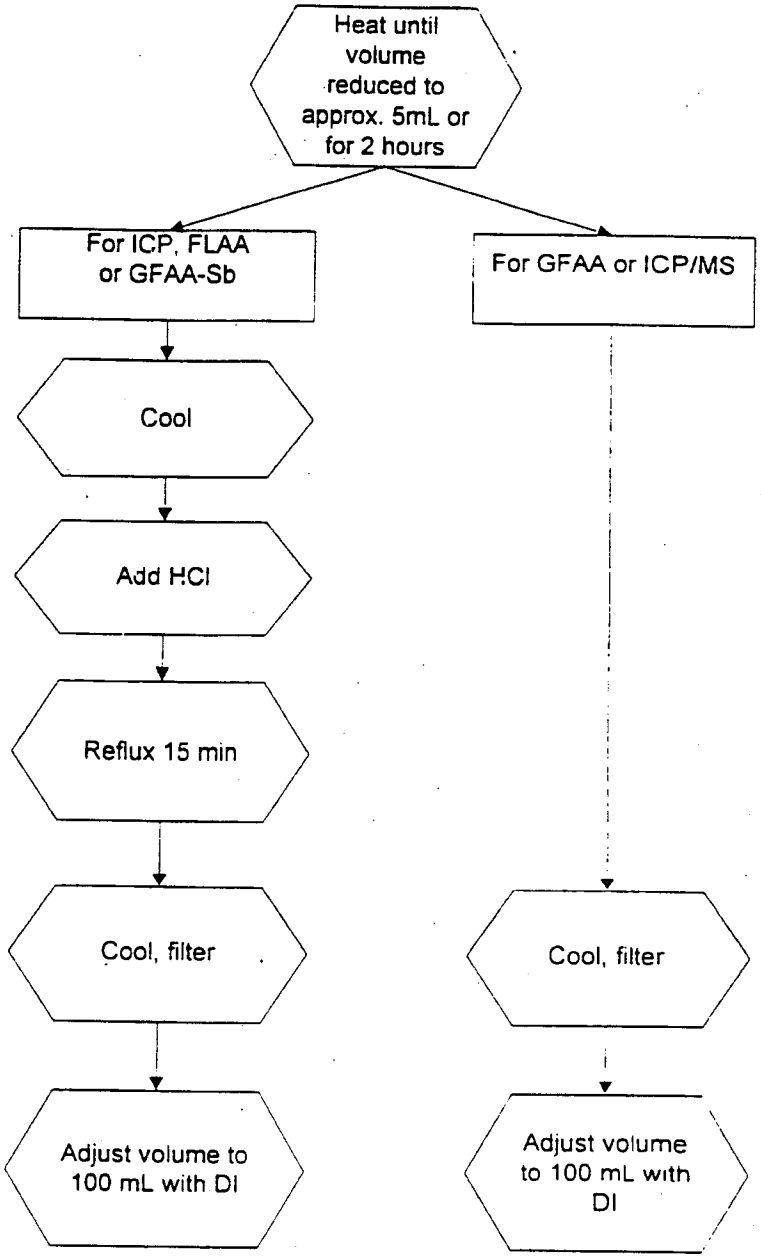


Figure 2. Soil Sample Preparation (continued)



APPENDIX A

TABLES

TABLE I. Method 3050B Approved Analyte List for ICP/GFAA/ICP-MS

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 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	200	2000	200
Antimony	50	500	50
Arsenic	200	2000	200
Barium	200	2000	200
Beryllium	5	50	5
Boron	100	1000	100
Cadmium	5	50	5
Calcium	5000	50000	5000
Chromium	20	200	20
Cobalt	50	500	50
Copper	25	250	25
Iron	100	1000	100
Lead	50	500	50
Lithium	100	1000	100
Magnesium	5000	50000	5000
Manganese	50	500	50
Molybdenum	100	1000	100
Nickel	50	500	50
Phosphorous	1000	10000	1000
Potassium	5000	50000	5000
Selenium	200	2000	200
Silica ***	1000	10000	1000
Silver	5	50	5
Sodium	5000	50000	5000
Strontium	100	1000	100
Thallium	200	2000	200
Tin	200	2000	200
Titanium	100	1000	100
Vanadium	50	500	50
Zinc	50	500	50

* 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 100 mL of sample.

** Final soil spike concentration based on the addition of 1.0 mL working spike (7.3) to 1.0 g of sample/100 mL final volume (assumes 100% solids).

*** As discussed in 1.4, the analysis is only for acid soluble silicon, which is often expressed as silica.

TABLE IV. 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.

TABLE III. 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	200	10,000	20	2
Selenium	200	10,000	20	2
Lead	200	10,000	20	2
Thallium	200	10,000	20	2
Antimony	200	10,000	20	2
Cadmium	25	1,000	2.5	0.25
Chromium	200	10,000	20	2
Silver	25	1,000	2.5	0.25

* 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).

APPENDIX B

QUANTERRA® METALS PREPARATION BENCHSHEET

RQC057 Quanterra Incorporated Run Date: 1/11/00
METALS PREP LOG/BATCH SUMMARY Time: 11:38:34

BATCH NUMBER: 0006307 PREP DATE: 1/07/00
DUE DATE 1/28/00 INITIALS: _____

LOT NUMBER	WORK ORDER	QC	ICP/WEIGHT	HG/WEIGHT	GFA/WEIGHT	FLA/WEIGH
D0A050101	D733W	4V	X _____g	_____g	_____g	_____g
WATER	TO DUE DATE:		1/28/00			
	D733WS		_____g	_____g	_____g	_____g
	D733WD		_____g	_____g	_____g	_____g
D0A050101	D733X	4V	X _____g	_____g	_____g	_____g
WATER	TO DUE DATE:		1/28/00			
D0A050101	D7340	4V	X _____g	_____g	_____g	_____g
WATER	TO DUE DATE:		1/28/00			
D0A050122	D73A0	4V	X _____g	_____g	_____g	_____g
WATER	TO DUE DATE:		1/28/00			
D0A050122	D73A4	4V	X _____g	_____g	_____g	_____g
WATER	TO DUE DATE:		1/28/00			
D0A050180	D73VW	4V	X _____g	_____g	_____g	_____g
WATER	TO DUE DATE:		1/28/00			
D0A050180	D73VX	4V	X _____g	_____g	_____g	_____g
WATER	TO DUE DATE:		1/28/00			
D0A050180	D7402	4V	X _____g	_____g	_____g	_____g
WATER	TO DUE DATE:		1/28/00			
D0A060000	D75MKB	4V	X _____g	_____g	_____g	_____g
WATER	DUE DATE:		0/00/00			
	D75MKC		_____g	_____g	_____g	_____g

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LEVEL 2
BLANK AND CHECK STANDARD ON BATCH _____
MS/MSD AND PDS ON BATCH _____
CURVE PREPPED FOR HG _____
CORRECT SPIKES ADDED _____
SPIKING SOLUTIONS DOCUMENTED ON BATCH LOG _____

Hot Block temperature
& final volume goes here.

APPENDIX C

CONTAMINATION CONTROL GUIDELINES

APPENDIX C. 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.

QUANTERRA 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 1)

Prepared by: Sevda Aleckson and Bill Nash

Reviewed by: _____
Director, Technology

Approved by: _____
Director, Quality Assurance,

Approved by: _____
Director, Environmental Health and Safety,

Approved by: _____
Management,

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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

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 Quanterra 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. The preparation of standards and reagents will be conducted in a fume hood or well ventilated area.

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.

5.7. 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 Quanterra 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 11.10.

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.

9.1.1. Instrument Detection Limit (IDL) - The IDL for each analyte must be determined for each analyte wavelength used on 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 Quanterra reporting limit. The CLP IDL procedure can be used for this method.

9.1.2. Method Detection Limit (MDL) - An MDL must be determined for each analyte prior to the analysis of any client 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 as detailed in Quanterra QA Policy QA-005. The spike level must be between the calculated MDL and 10X the MDL to be considered valid. The result of the MDL determination must be below the

Quanterra reporting limit (RL). MDL studies for the determination of metals in soil need not be performed; an appropriate soil MDL may be computed from the experimentally determined MDL for metals in aqueous solution.

9.1.3. Linear Range Verification (LR) - The linear range must be determined on 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 define the linear range limit must be analyzed during a routine analytical run. 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.

9.1.4. 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.

9.1.5. 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 ± 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 observed 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.

9.1.6. Rinse Time Determination - Rinse times must be determined annually. 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. Until in-house control limits are established, for method 6010B, a control limit of 80 - 120% (85-115% for 200.7) 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% (115% for 200.7) 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 (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.

- 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 11.11 or 11.12 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 11.11 or 11.12 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 Quanterra 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 11.11 or 11.12 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 Quanterra guidelines of ± 2x RL from zero. ICSA results for the non-interfering elements with RLs> 10 µg/L must fall within the Quanterra 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. 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.

9.10. 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 <u>two exposures</u> 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. For unattended overnight auto-runs it is strongly recommended that the frequency of ICSA/ICSAB analysis be increased to every 4 hours.

11.9. 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.

11.10. 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	5	10
SW846 3005	2	5
SW846 3010	3	5

The following procedural guidelines must be followed when using an internal standard:

11.10.1. 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.)

11.10.2. 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.

11.10.3. 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.

11.10.4. The internal standard raw intensity counts must be printed on the raw data.

11.10.5. 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).

11.10.5.1. If the internal standard counts fall within ±30% of the counts observed in the ICB then the data is acceptable.

11.10.5.2. 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.

11.11. The following analytical sequence must be used for Methods 6010B and 200.7:

Instrument Calibration
ICV
ICB
ICSA

ICSAB
8 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.

11.12. 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-002 for details.

11.13. Full method required QC must be available for each wavelength used in determining reported analyte results.

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 Quanterra 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% (15% for 200.7) 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-0002, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Method 200.7 & CLP-M, SOW ILMO3.0.

16.5. QA-003, Quanterra 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. Quanterra 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. The alternate run sequence presented in Section 11.12 is consistent with method requirements. Additional QC (i.e., ICSA) analyses were added to accommodate the CLP protocol requirements.

17.1.1.3. 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," Quanterra has adopted the procedure in EPA CLP ILMO4.0.

17.1.1.4. 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 Quanterra laboratories receive no prior information from clients regarding when to expect a new or unusual matrix, Quanterra 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 Quanterra labs, matrix interference is determined by evaluating data for the LCS and MS/MSD. Quanterra 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. Quanterra 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." Quanterra 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. Quanterra 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 IDLIf not, repeat the analysis two or more times and average the results. If the average is not within three standard deviation 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. Quanterra 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 1 of this SOP.

17.3. Facility-Specific SOPs

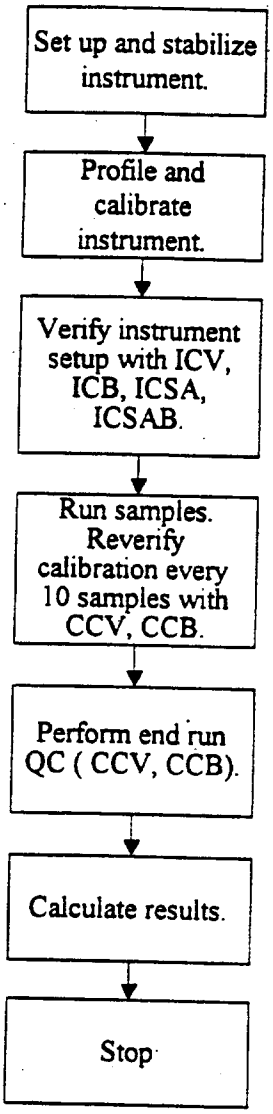
Each facility shall review and revise as appropriate this SOP to reflect any facility-specific requirementsIf no facility-specific amendments are required, the SOP can be adopted as is.

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
Lithium	Li	7439-93-2	X	X	50	5
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
Phosphorus	P	7723-14-0	X	X	300	30
Potassium	K	7440-09-7	X	X	5000	500
Selenium	Se	7782-49-2	X	X	250	25
Silicon	Si	7631-86-9		X	500	N/A
Silver	Ag	7440-22-4	X	X	10	1
Sodium	Na	7440-23-5	X	X	5000	500
Strontium	Sr	7440-24-6	X		50	5
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
Bismuth	Bi	7440-06-99	200	20
Zirconium	Zr	7440-06-77	100	10
Tungsten	W	7440-03-37	500	50
Tellurium	Te	1349-48-09	500	50
Thorium	Th	7440-02-91	500	50
Uranium	U	7440-06-11	500	50
Palladium	Pd	7440-00-53	100	10

NOTE: Analysis of all elements listed may not be available at all Quanterra facilities.

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
Lithium	1000	-1000
Magnesium	50000	50000
Manganese	500	500
Molybdenum	1000	1000
Nickel	500	500
Phosphorous	10000	10000
Potassium	50000	50000
Selenium	2000	2000
Silver	50	50
Sodium	50000	50000
Strontium	1000	1000
Thallium	2000	2000
Vanadium	500	500
Zinc	500	500
Boron	1000	1000
Silicon	10000	10000
Tin	2000	2000
Titanium	1000	1000
Bismuth	1000	1000
Zirconium	1000	1000
Tellurium	1000	1000
Thorium	1000	1000
Uranium	1000	1000
Tungsten	1000	1000
Palladium	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
Lithium	10000	50	1000	5000
Magnesium	100000	5000	25000	50000
Manganese	10000	15	1000	5000
Molybdenum	10000	40	1000	5000
Nickel	10000	40	1000	5000
Phosphorous	10000	300	1000	5000
Potassium	100000	5000	25000	50000
Selenium	10000	250	1000	5000
Silver	2000	10	500	1000
Sodium	100000	5000	25000	50000
Strontium	10000	50	1000	5000
Thallium	20000	2000	5000	10000
Vanadium	10000	50	1000	5000
Zinc	10000	20	1000	5000
Boron	10000	200	1000	5000
Silicon	10000	500	1000	5000
Tin	10000	100	1000	5000
Titanium	10000	50	1000	5000
Bismuth	10000	200	1000	5000
Zirconium	10000	100	1000	5000
Tellurium	10000	500	1000	5000
Thorium	10000	500	1000	5000
Uranium	10000	500	1000	5000
Tungsten	10000	500	1000	5000
Palladium	10000	100	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

* Custom Quanterra solutions contain analytes common to all Quanterra 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	RSD between duplicate exposures ≤5%	Terminate analysis; Correct the problem; Prepare new standards; Recalibrate following system performance.
ICV	Beginning of every analytical run.	Method 200.7: 95 - 105 % recovery. Method 6010B: 90 - 110 % recovery.	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.	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 11.11 and 11.12 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. (85-115% for 200.7) Samples for which the contaminant is < RL and the LCS results are > 120% (115% for 200.7) 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. I 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

QUANTERRA ICP DATA REVIEW CHECKLIST

Quanterra ICP Data Review Checklist

Run/Project Information:

Run Date: _____ **Analyst:** _____ **Instrument:** _____
Prep Batches Run: _____

Circle Methods used: **6010B / 200.7: CORP-MT-0001 Rev 2**
CLP ILMO3.0/4.0 : CORP-MT-0002 Rev 1

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 ? (6010B, CLP = 90 - 110%, 200.7 = 95 -105%[ICV])				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP) ?				
4. CRI analyzed? (for CLP only)				
5. 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 run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits ?				
5. Dilution Test 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 ?				

Analyst: _____ **Date:** _____
Comments: _____

_____

_____

2nd Level Reviewer : _____ **Date:** _____
Comments: _____

_____

_____

APPENDIX C

CROSS REFERENCE OF TERMS USED IN METHODS 6010B, 200.7, AND BY QUANTERRA

CROSS REFERENCE OF TERMS COMMONLY USED IN METHODS EPA 200.7, SW6010B, AND QUANTERRA INC. SOP

EPA 200.7	SW6010B	Quanterra 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

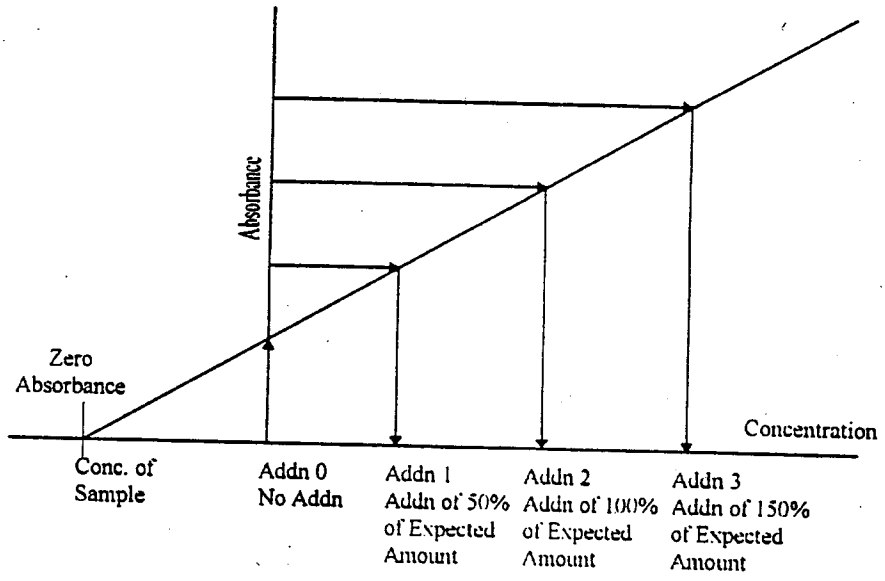
MSA GUIDANCE

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.



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)

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Implementation Date 6-14-99

SOP No. DEN-MT-0002
Revision No. 4.0
Revision Date: 6/7/99

OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY,

EPA METHODS 6020 AND 200.8

(SUPERSEDES: DEN-MT-0002, REVISION 3.0)

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1. SCOPE AND APPLICATION

1.1. This procedure describes multi-elemental analysis by inductively coupled plasma-mass spectrometry (ICP-MS) based on SW-846 protocol as described in EPA Method 6020. The source method lists fifteen elements approved for analysis by ICP/MS (Al Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Ag, Tl, and Zn). This procedure has been developed for thirty-five elements (see Table VIII), and additional elements may be included provided that the method performance criteria presented in Section 9. However, project approval may be required from the controlling agencies for compliance testing beyond the fifteen elements included in the promulgated method.

1.2. This procedure also describes the requirements for performing analysis of ground waters, surface waters and drinking water.

1.3. The procedure is applicable to the analysis of waters, soils, and wastes. No digestion is required prior to analysis for dissolved elements in water samples, but the samples must be filtered and preserved prior to analysis. Preliminary acid digestion is required for groundwater, aqueous samples, sludges, sediments, and other solid wastes for which total (acid-leachable) elements. See SOP # CORP-IP-001 for preparation details.

2. SUMMARY OF METHOD

2.1. Aqueous samples, digestates or leachates are nebulized into a spray chamber where a stream of argon carries the sample aerosol through the quartz torch and injects it into an R.F. plasma. There the sample is decomposed and desolvated. The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrapole mass spectrometer capable of providing a resolution better than or equal to 0.9 AMU peak width at 10% of the peak height. For analysis by methods 200.8 the resolution requirement is 1.0amu at 5% peak height. The ions are sorted according to their mass-to-charge ration and measured with a channel electron multiplier. Interference must be assessed and valid corrections applied, or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents and the constituents of the sample matrix. Recommended elemental equations which correct for many of these interferences are listed in Table I. Use of the internal standard technique is required to compensate for suppressions and enhancements caused by sample matrices.

3. DEFINITIONS

3.1. *Dissolved Metals* - Those elements which pass through a 0.45 μm membrane filter (sample is acidified after filtration).

3.2. *Suspended Metals* - Those elements which are retained by a 0.45 μm membrane filter.

3.3. *Total Metals* - The concentration determined on an unfiltered sample following vigorous acid digestion.

3.4. *Total Recoverable Metals* - The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acids.

3.5. *Instrument Detection Limit (IDL)* - See Section 9.1.1.

3.6. *Sensitivity* - The slope of the analytical curve (i.e. the functional relationship between raw instrument signal and the concentration).

3.7. *Tuning Solution* - This is a multi-element solution containing analytes which are representative of the entire mass range capable of being scanned by the instrument. It is used to optimize the sensitivity of the instrument and to verify the mass resolution meets method criteria.

3.8. *Initial Calibration Verification/Quality Control Standard (ICV/QCS)* - A multi-element standard of known concentrations prepared to verify instrument calibration. This solution must be an independent standard prepared near the mid-point of the calibration curve, and at a concentration other than that used for instrument calibration.

3.9. *Continuing Calibration Verification (CCV).* - A multi-element standard of known concentrations prepared to monitor and verify the instrument daily continuing performance .

3.10. *Interference Check Standard (ICS)* - A solution containing both interfering and analyte elements of know concentration that is used to verify background and interelement correction factors.

3.11. *Laboratory Control Sample / Laboratory Fortified Blank (LCS/LFB)* - A multi-element standard of known concentrations which is carried through the entire sample preparation and analysis procedure. This solution is used to verify the accuracy of the sample preparation.

3.12. *Reagent Blank* - High purity (> 18 megohm-cm)water containing the same acid matrix as the calibration standards carried through the entire digestion process.

3.13. *Calibration Blank* - High purity (> 18 megohm- cm) water acidified with the same acid concentrations present in the standards and samples Also referred to as the Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB).

3.14. *Method Reporting Limit (MDL)*. See section 9.1.3.

4. INTERFERENCES

4.1. Isobaric Interferences - Isobaric interferences in the ICPMS are caused by isotopes of different elements forming ions with the same nominal mass-to-charge ratio (m/z). Most interferences of this type are corrected for by the instrument software.

4.2. Isobaric Molecular and Doubly Charged Ion Interferences - Isobaric molecular interferences are caused by ions consisting of more than one atom or charge. Table III lists isobaric interferences which might possibly affect required analytes. When these interferences cannot be avoided by the use of another isotope with sufficient natural abundance, corrections must be applied and the data flagged to indicate the presence of interferences.

4.3. Physical Interferences - Physical interferences are associated with the transport and nebulization process. Internal standards are used to compensate for these types of interferences.

4.3.1. One or more of the elements shown in Table V is selected as an internal standard. A representative project sample is first screened to determine if the elements proposed for use as internal standards is already present in the sample. The native concentration of the elements should be less than 10% of the internal standard spiking concentration.

4.3.2. Internal standards should be added at a level to give approximately 50,000 - 800,000 counts of raw signal intensity. The mass of the internal standard should ideally be within 50 AMU of the mass of the measured analyte.

4.3.3. Matrix effects will be monitored by comparing the internal standard intensity in the sample to the internal standard intensity of the calibration blank. When performing method 6020 the internal standard intensities must be between 30% and 120% of the intensities in the calibration blank. If they fall outside this window, a five fold dilution (1:4) is performed on the sample to correct for matrix effects and the sample reanalyzed. When performing method 200.8

the internal standards must be between 60% and 125% of the calibration blank. If they are outside this window the calibration blank is reanalyzed to verify internal standard intensities. If the intensities are within limits the sample is diluted by a factor of 2 (1:1) and reanalyzed.

4.3.4. Memory effects are dependent on the relative concentration differences between samples and/or standards which are analyzed sequentially. The rinse period between samples must be long enough to eliminate significant memory interference.

4.3.5. In order to reduce the interference of chloride, digested samples for method 200.8 must be diluted 2.5X prior to analysis.

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 (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**:

Sulfuric acid, hydrochloric acid, nitric acid, and hydrofluoric acid.

Note: Sulfuric and hydrochloric acids are used in cleaning the ICP/MS 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.

5.4. 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.

5.5. 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.

5.6. 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.

5.7. 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. Argon gas: High purity grade (99.99%).

6.2. Inductively Coupled Plasma Mass Spectrometer capable of providing resolution, less than or equal to 0.9 AMU at 10% peak height from 6-253 AMU and 1.0AMU at 5% peak height from 6-253 AMU with a data system that allows corrections for isobaric interferences and the application of the internal standard technique.

6.3. A four channel peristaltic pump.

6.4. Appropriate water cooling device.

6.5. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.6. Autosampler with autosampler tubes.

7. REAGENTS AND STANDARDS

7.1. Calibration standards are purchased as custom Quanterra multielement mixes or as single element solutions. All standards must be stored in FEP fluorocarbon or previously 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. See Table XII.

7.2. Check Calibration Standard (ICV/CCV)

A quality control standard similar to the calibration standards (10 ppm solutions) and prepared in the same acid matrix. This solution must be from an independent standard and made at a concentration near the midpoint of the calibration curve. This standard is composed of analytes from a different source from those used in the calibration of the instrument. See Table XII.

7.3. The tuning solution is purchased as custom Quanterra multielement mixes or as single element solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. The solution 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.

7.4. The use of hydrochloric and sulfuric acids should be minimized due to higher incidence of molecular-ion interferences with the presence of these acids. Excessive amounts of nitric acid can also lead to molecular interferences.

7.5. Reagent water:

ASTM Type I or equivalent for the elements of interest, generated using an ion-exchange water polishing system capable of achieving 18.0 megohm-cm.

7.6. Nitric acid, 1% HNO_3:

Carefully dilute 20 mL of concentrated HNO_3 to 2.0 L with reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Aqueous samples are preserved with nitric acid to a pH of 2, and may be stored in plastic or glass. Preservation must be verified prior to analysis.

8.2. Soil samples do not require preservation, but must be stored at 4° ± 2°C until the time of preparation.

8.3. The analytical holding times for metals are six months from the time of collection.

8.4. Solid and aqueous samples must be digested prior to analysis by the appropriate method.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

Table XI provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action. Prior to analysis of any analyte the following requirements must be met.

9.1.1. Instrument Detection Limit (IDL) - IDLs can be estimated by calculating the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as a separate analytical sample. The IDL should be performed every three months.

9.1.2. Linear Calibration Ranges - Linear calibration ranges are primarily detector limited. The upper limit of the linear calibration range should be established for each analyte by determining the signal responses from a minimum of three different concentration standards, one of which is close to the upper limit of the linear range. Care should be taken to avoid potential damage to the detector during this process. The linear calibration ranges which may be used for the analysis of samples should be judged by the analyst from the resulting data. The upper LDR limit should be an observed signal no more than 10% below the level, extrapolated from lower standards. For samples analyzed by method 200.8, determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware of operating conditions would dictate they be redetermined. Linear calibration ranges should be performed annually.

9.1.3. Method Detection Limit (MDL) - The laboratory shall determine a method detection limit for all analytes of interest initially and annually thereafter. The MDL study is performed and calculated according to Quanterra Policy number CORP-QA-005, which is based on 40CFR Part 136 Appendix B.

9.2. Method Blank/Laboratory Reagent Blank (MB/LRB) - 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, calcium, magnesium, potassium, sodium 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 / Laboratory Fortified Blank (LCS/LFB) - One LCS/LFB must be processed for each preparation batch (not to exceed 20 field samples). The historical limits for the LCS/LFB for each analyte are in the LIMS system. These limits are not to exceed 85-115% for method 200.8. If the LCS/LFB exceeds these limits for any analyte, that analyte is judged to be out of control and must be corrected before the analysis can be reported.

9.4. Matrix Spike / Matrix Spike Duplicate / Laboratory Fortified Sample Matrix / Laboratory Fortified Matrix Duplicate (MS/MSD/LFM/LFMD) - One MS/MSD pair must be processed for each preparation batch (not to exceed 20 field samples). A matrix spike is a field sample to which known concentrations of analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same samples (spiked identically as the MS) prepared and analyzed along with the sample and other

batch QC. The historical spike recovery acceptance limits for each analyte are in the LIMS system. These limits should not exceed 70-130% for method 200.8, and the spike concentration must be the same level as the LFB. If they are not in control and all other quality control criteria have been met then a matrix interference is suspected. A nonconformance memo should be generated and distributed to the program manager and QA.

9.5. ICV/CCV/QCS - Calibration accuracy is verified at the beginning of each analytical run by analyzing a second-source initial calibration verification (ICV) standard. A continuing calibration verification (CCV) standard is analyzed at a 10% frequency throughout the run. ICV must be within 10% of the expected value, or the analysis is terminated. The CCV must be within 10% of the expected value, or the analysis is terminated and all samples since the last successful ICV/CCV reanalyzed. When performing method 200.8, if the failing CCV is within 85-115% recovery the previous samples need not be reanalyzed.

9.6. ICB/CCB/CB - The initial calibration blank must be analyzed immediately following the ICV. The continuing calibration blank must be analyzed at a frequency of 10% throughout the remainder of the analytical run. The ICB/CCB must be less than the reporting limit to be accepted.

9.7. Interference Check Solutions (ICSA/ICSAB) ***method 6020 only***- The interference check solution is prepared with known concentrations of interfering elements so a determination may be made as to the magnitude of the interference on analytes of interest as well as a test of any software corrections. The required elements and their concentrations are listed in Table VI. The interference check solutions must be analyzed at the beginning of every analytical run or once every 12 hours, whichever is more frequent. The results of solution "A" and solution "AB" should be monitored for possible interferences. See Table VI for analyte concentrations.

Note: It may not be possible to obtain absolutely clean ICSA/ICSAB standards. If contamination can be confirmed by another method (ICP/GFAA), acceptance criteria will be applied at that level and the data accepted.

9.8. Internal Standards:

9.8.1. Method 6020

The intensities of all internal standards must be monitored throughout the run. The internal standard in the samples must be between 30% and 120% of the intensity of the calibration blank. If the sample falls outside of this criteria, a

fivefold (1: 4) dilution must be performed and the sample analyzed and the same acceptance criteria applied.

9.8.2. Method 200.8

The intensities of all internal standards must be monitored throughout the run. The internal standards in samples must be between 60 and 125% of the intensity in the calibration blank. If the sample intensities fall outside this range the calibration blank is reanalyzed to confirm the instrument has not drifted out of control. If the criteria is met, the sample is diluted by a factor of 2 (1:1) and reanalyzed.

9.9. Serial Dilution ***method 6020 only*** - One serial five-fold dilution should be analyzed per batch for each matrix. If the analyte concentration is within linear range of the instrument and sufficiently high (generally, a factor of 100 times above the reporting limit), the serial dilution must agree within 10% of the original analysis. If not, an interference effect must be suspected, which must be described in an anomaly report and included in the final report narrative. Samples identified as blanks cannot be used for serial dilution.

9.10. Post-Digestion Spike Addition (PDS) ***method 6020 only*** - If the serial dilution fails to meet the acceptance criteria, a PDS must be performed as follows. An analytical spike added to a portion of a prepared sample, or its dilution, should be recovered within 75 - 125% of the known value. If the PDS fails to meet this criterion, matrix interference should be suspected.

9.11. General Corrective Action Requirements - The general requirements for evaluation of QC results and corrective action for failures is described in Quanterra Policy # QA-003.

10. CALIBRATION AND STANDARDIZATION

10.1. Instrument Start Up

Set up the instrument according to manufacturers operating instructions. Allow the instrument to become thermally stable for at least 30 minutes before tuning.

10.2. Instrument Tuning / Mass Calibration

10.2.1. Refer to the facility specific instrument SOP and ICP/MS instrument manual for detailed set up and operation protocols. Allow the instrument to become thermally stable before beginning instrument tuning (approximately 30

minutes of warm-up is required).

Tune the instrument with a solution containing elements representing all of th mass regions of interest. The relative standard deviations must be less than 5% after running the tuning solution a minimum of 4 times. For method 200.8, the tuning solution must be analyzed 5 times with a relative standard deviation less than 5%.

10.2.2. Mass calibration and resolution checks using the tuning solution must be completed at the beginning of every day.

10.2.2.1. Mass Calibration Check – The mass calibration results must be within 0.1 amu from the true value. If this criterion is not met, the mass calibration must be adjusted before running samples.

10.2.2.2. Mass Resolution Check - The resolution must be verified to be less than 0.9 AMU full width at 10% peak height. Due to a limitation of the instrument software, the resolution requirement for method 200.8 of 1.0AMU full width at 5% peak height cannot be verified automatically. If the mass resolution requirement of 0.9AMU at 10% peak height is met the 200.8 requirement is also satisfied. See Appendix III.

10.3. Calibrate the instrument for the analytes of interest according to manufacturer's instructions. Routine calibration and calibration verification levels are shown in Table XII. Acceptance criteria are given in Section 9.4 above. The calibration should include a blank and a high standard. Report the average of at least three integrations for both calibration and sample analysis. A calibration must be performed daily and each time the instrument is set up.

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. Analysis

11.3.1. Flush the system with the rinse blank for at least 30 seconds between samples and standards during the analytical run.

11.3.2. Masses which would affect the data quality must be monitored during the analytical run to determine the potential effects of matrix on a given element.

11.3.3. Dilute and reanalyze samples that are more concentrated than the linear range for an analyte or specific isotope of interest . No analyte may be reported from an analysis of a diluted sample in which the analyte concentration is less than 5 times the IDL. (The sample should be diluted to the approximate midrange of the analytical curve.)

11.3.4. In order to reduce the interference of chloride, digested samples for method 200.8 must be diluted 2.5X prior to analysis.

11.3.5. The analytical run sequence should be performed as follows to meet all quality control criteria:

Instrument initialization
Warm-up
Tune instrument
Perform mass calibration
Perform resolution check
Validate tuning criteria
Calibration blank
Calibration standard
ICV
ICB
Calibration standard (200.8 only)
Calibration blank (200.8 only)
ICSA (6020 Only)
ICSAB (6020 Only)
10 Samples (which can include all sample types)
CCV
CCB
10 samples
CCV
CCB
Reslope

CCB
CCV

12. DATA ANALYSIS AND CALCULATIONS

Note: Samples prepared for *dissolved analytes* in ground and surface waters will have a preparation dilution factor of 1.02. Samples prepared for *total recoverable analytes* in drinking water samples will have a preparation dilution factor of 1.25.

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100 \times \left(\frac{\text{Found (ICV)}}{\text{True (ICV)}} \right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100 \times \left(\frac{\text{Found (CCV)}}{\text{True (CCV)}} \right)$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100 \times \left(\frac{\text{SSR - SR}}{\text{SA}} \right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

Note: When sample concentration is less than the instrument detection limit, use SR = 0 for purposes of calculating % Recovery.

12.4. The relative percent difference (RPD) of sample duplicates are calculated according to the following equation:

$$\text{RPD} = 100 \times \left[\frac{\text{(DU1 - DU2)}}{\text{(DU1 + DU2) / 2}} \right]$$

Where:
DU1 = Sample result
DU2 = Sample duplicate result

12.5. The final concentration for an aqueous sample is calculated as follows:

$$\text{Result (mg/L)} = \frac{(C \times V1 \times D)}{V2}$$

Where:
C = Concentration from instrument readout, ppm
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.6. The concentration determined in digested solid samples when reported on a wet weight basis is as follows:

$$\text{Result (mg/kg)} = \frac{(C \times V \times D)}{W}$$

Where:
C = Concentration from instrument readout, ppm
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight, in g, of wet sample digested

12.7. Sample results should be reported according to the following significant figure rules:

12.7.1. All uncorrected values less than the detection limit are reported as "less than" the detection limit.

12.7.2. Positive results for target analytes are reported to three significant figures if the method is used without dilution.

12.7.3. Positive results obtained after dilution and results for non-certified analytes are reported to two significant figures.

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 Table I for the list of analytes that may be analyzed using this SOP for methods 6020 and 200.8.

13.3. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. The matrix spike recovery should fall within historical laboratory control limits and the matrix spike duplicates should compare within 20% RPD. Method blanks must meet the criteria specified in Section 9.2. The laboratory control samples should recover within 30% of the true value until in house control limits are established.

13.4. 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

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

Waste generated in this procedure must be segregated and disposed according to the facility hazardous waste procedure. The Environmental Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. Test Methods For Evaluating Solid Waste, EPA SW-846, 3rd Edition, Final Update II, Method 6020: "Inductively Coupled Argon Plasma - Mass Spectrometry", Revision 0, September 1994.

16.2. Environmental Monitoring Systems Laboratory, EPA Method 200.8, "Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma - Mass Spectrometry", Revision 5.4, EMMC Version.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Deviations from Source Methods

17.1.1. Deviations from method 6020

17.1.1.1. Commercially available standards are purchased and verified at the laboratory rather than being prepared from the solid material. These verification records are kept on file with QA.

17.1.1.2. The results of the calibration blank as well as all other blanks must be less than the reporting limit, not 3 times the instrument IDL.

17.1.1.3. Milli-Q or Nanopure water is substituted when reagent water is called for. This water is tested to be free of contaminants by conductivity (18megOhm) and by the analysis of blanks.

17.1.2. Deviations from method 200.8

17.1.2.1. Commercially available standards are purchased and verified at the laboratory rather than being prepared from the solid material. These verification records are kept on file with QA.

17.1.2.2. The results of the calibration blank as well as all other blanks must be less than the reporting limit, not 3 times the instrument IDL.

17.1.2.3. Milli-Q or Nanopure water is substituted when reagent water is called for. This water is tested to be free of contaminants by conductivity (18 megOhm) and by the analysis of blanks.

17.1.2.4. Resolution criteria of the mass calibration is met if the resolution criteria for method 6020 is satisfied. See Appendix III.

17.1.2.5. The concentration of all analytes in the LFB is 200 µg/L. This is made from a commercially available stock solution and has all analytes at the same level. Verification records for this solution are on file with QA.

17.1.2.6. The diluent for chloride reduction in method 200.8 is 1% HNO_3 in place of reagent water.

17.2. Modifications from the previous revision of the SOP

17.2.1. The tuning resolution check was added.

17.2.2. The concentration of the tuning standard was lowered from 100 ug/L to 10 ug/L due to the improved sensitivity of the newer instrument.

17.2.3. The ICS frequency in Table XI was corrected to match the text.

17.2.4. The ICS concentrations in Table VI were updated.

17.2.5 The SOP was corrected to state that PDS analysis is required when serial dilutions fail, rather than when MS/MSDs fail.

17.3. Table I: Recommended Elemental Equations for Use In Method 6020

17.4. Table II: Contributions of Contaminant Elements to Nearby Analytes when Resolution and Measurement Schemes Vary

17.5. Table III: Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

17.6. Table IV: Changes in Molecular-Ion Interferences with Changing Plasma Conditions

17.7. Table V: Recommended Internal Standards

17.8. Table VI: Interference Check Sample Components and Concentrations

17.9. Table VII: Sample Preservation and Holding Times

17.10. Table VIII: Suggested Mass Choices for Elements Which May Be Monitored Either During the Analytical Run or in a Separate Scan

17.11. Table IX: Tuning Solution

17.12. Table X: Suggested Tuning and Response Factor Criteria

17.13. Table XI: Summary of Quality Control Requirements

17.14. Table XII: ICP/MS Calibration and Calibration Verification Standards

17.15. Appendix I: Data Review Checklist

17.16. Appendix II: Flow Chart

TABLE I: Recommended Elemental Equations

Element	Isobaric Correction	Mathematical Equation
Al	none	(1.0000)(27M)
Sb	none	(1.0000)(121M)
As	ArCl, Se	(1.0000)(75M) - (3.1278)(77M) + (1.0177)(78M)
Ba	none	(1.0000)(135M)
Be	none	(1.0000)(9M)
Cd	MoO, Sn	(1.0000)(114M) - (0.0268)(118M) - (1.0000)(135M)
Ca	none	(1.0000)(44M)
Cr	none	(1.0000)(52M)
Co	none	(1.0000)(59M)
Cu	none	(1.0000)(65M)
Fe	none	(1.0000)(57M)
Pb	none	(1.0000)(208M) + (1.0000)(207M) + (1.0000)(206M)
Mg	none	(1.0000)(25M)
Mn	none	(1.0000)(55M)
Ni	none	(1.0000)(60M)
K	none	(1.0000)(39M)
Se	Ar2	(1.0000)(78M) - (1.1869)(76M)
Ag	none	(1.0000)(107M)
Na	none	(1.0000)(23M)
Tl	none	(1.0000)(205M)
V	ClO, Cr	(1.0000)(51M) - (3.1081)(53M) + (0.3524)(52M)
Zn	none	(1.0000)(66M)
6Li	Li (natural)	(1.0000)(6M) - (0.0813)(7M)
Sc	none	(1.0000)(45M)
Y	none	(1.0000)(89M)
Rh	none	(1.0000)(103M)
In	Sn	(1.0000)(115M) - (0.0149)(118M)
Tb	none	(1.0000)(159M)
Ho	none	(1.0000)(165M)
Bi	none	(1.0000)(209M)

M = Total ion count rate at the specified mass.

TABLE II: Contributions of Contaminant Elements to Nearby Analytes when Resolution and Measurement Schemes Vary

Concentrations listed are the approximate level (µg/L) measured when the interferent is present at 100 mg/L.

Analyte	Interferent Element	Peak Width at 10% of the Peak Height			
		1.0 AMU Integration Width		0.8 AMU Integration Width	
		0.9 AMU	0.3 AMU	0.9 AMU	0.3 AMU
^{121}Sb	^{120}Sn	820	5	10	1
^{121}Sb	^{122}Te	77	none	1	none
^{75}As	^{74}Se, ^{76}Se	910	4	3	none
^{9}Be	^{10}B	1,200	12	9	1
^{112}Cd	^{113}In	1,700	8	10	none
^{114}Cd	^{115}In	5,000	150	180	18
^{116}Cd	^{115}In	30	none	5	none
^{52}Cr	^{51}V	1.4	1.5	none	none
^{53}Cr	^{54}Fe	650	7	1	none
^{59}Co	^{58}Ni, ^{60}Ni	1,500	6	2	none
^{63}Cu	^{62}Ni, ^{64}Ni	190	1	none	none
^{63}Cu	^{64}Zn	4,000	14	9	none
^{65}Cu	^{64}Ni	1	1	none	none
^{65}Cu	^{64}Zn, ^{66}Zn	4,400	22	15	none
^{208}Pb	^{209}Bi	140	14	57	none
^{55}Mn	^{54}Fe, ^{56}Fe	900	8	4	none
^{58}Ni	^{59}Co	3,000	96	75	7
^{60}Ni	^{59}Co	9	4	10	5
^{62}Ni	^{63}Cu	8,500	690	4,500	16
^{107}Ag	^{106}Pd, ^{108}Pd	2,400	22	80	4
^{107}Ag	^{106}Cd, ^{108}Cd	130	3	5	2
^{109}Ag	^{108}Pd, ^{110}Pd	1,800	12	36	3
^{109}Ag	^{108}Cd, ^{110}Cd	1,600	10	37	3
^{51}V	^{52}Cr	2,100	45	410	1
^{64}Zn	^{65}Cu, ^{63}Cu	7,800	57	410	2
^{66}Zn	^{65}Cu	2	none	3	2

TABLE III: Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Analyte	Interferences						
	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{121}Sb	PdO		AgN			AgC	
^{123}Sb	AgO		AgN	SrCl	ZrS	CdC	
^{75}As	CoO	NiOH	NiN	ArCl	CaS	CuC	
^{138}Ba	SnO	SbOH					
^{137}Ba	SbO	SnOH		MoCl			
^{136}Ba	SnO	SnOH				SnC	
^{135}Ba	SnO	SnOH		MoCl			
^{134}Ba	SnO	SnOH	SnN	MoCl		SnC	
^{132}Ba	SnO, CdO	InOH	SnN	MoCl	MoS	SnC	
^{130}Ba	CdO	CdOH	SnN, CdN	MoCl	MoS	SnC	
^{9}Be							
^{114}Cd	MoO	MoOH	MoN	SeCl	SeS		
^{112}Cd	MoO, ZrO	MoOH	MoN	AsCl, SeCl	SeS	MoC	
^{111}Cd	MoO	MoOH	MoN	GeCl			
^{110}Cd	MoO, ZrO		MoN, ZrN	GeCl, AsCl	SeS	MoC	
^{113}Cd	MoO	MoOH		SeCl, AsCl			
^{116}Cd	MoO						
^{106}Cd	ZrO		MoN, ZrN		GeS	MoC, ZrC	
^{108}Cd	MoO, ZrO	ZrOH	MoN, ZrN	GeCl	SeS, GeS	MoC, ZrC	
^{52}Cr	ArO	ClOH				ArC	
^{53}Cr	ClO	ArOH	KN	NCl, OCl		KC	
^{50}Cr	SO		ArN		SO	ArC	Mo^{++}
^{54}Cr		ClOH	ArN, CaN			CaC	
^{59}Cr	CaO	CaOH	ScN	MgCl	AlS	TiC	Sn^{++}
^{63}Cu	TiO, PO_2	TiOH	TiN	SiCl, MgCl	PS	VC	ArNa
^{65}Cu	TiO	TiOH	VN	SiCl	SS,SO,H	CrC	
^{208}Pb							
^{206}Pb							
^{207}Pb							
^{204}Pb							
^{55}Mn	KO	ArOH	KN		NaS	CaC	Cd^{++}

TABLE III: (cont.) Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Analyte	Interferences						
	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{202}Hg	WO						
^{200}Hg	WO	WOH	WN				
^{199}Hg	WO	WOH					
^{201}Hg		WOH					
^{198}Hg	WO	TaOH	WN			WC	
^{204}Hg							
^{196}Hg			WN				
^{58}Ni	CaO	KOH	CaN	NaCl	MgS	TiC	Cd^{++}, Sn^{++}
^{60}Ni	CaO	CaOH	TiN	MgCl, NaCl	SiS	TiC	Sn^{++}
^{62}Ni	TiO	ScOH	TiN	AlCl, MgCl	SiS	TiC, CrC	Sn^{++}
^{61}Ni	SeO	CaOH	TiN	MgCl	SiS	TiC	
^{64}Ni	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	
^{80}Se	ZnO	CuOH	ZnN	ScCl, CaCl	TiS	ZnC	
^{78}Se	NiO	NiOH	ZnN	CaCl, KCl	TiS	ZnC	
^{82}Se	ZnO	CuOH	ZnN	TiCl, ScCl	TiS, CrS		
^{76}Se	NiO	CoOH	NiN	KCl	CaS	ZnC	
^{77}Se	NiO	CuN	CuN	CaCl, ArCl	ScS	CuC	
^{74}Se	NiO	NiN	NiN	ClCl, KCl	CaS	NiC	
^{107}Ag	ZrO	ZrOH		GeCl	AsS	MoC	
^{109}Ag		MoOH	MoN	GeCl	SeS	MoC	
^{205}Tl							
^{203}Tl		WOH					
^{51}V	ClO	SOH	ClN	ClO, ClN	FS	KC	
^{50}V	SO		ArN			ArC	Mo
^{64}Zn	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	
^{66}Zn	TiO	TiOH	CrN	PCl, SiCl	SS	FeC	
^{68}Zn	CrO	VOH	FeN	PCl	ArS	FeC	Ba^{++}
^{67}Zn	VO	TiOH, Cr	CrN	SCl	ClS	MnC	Ba^{++}
^{70}Zn	FeO	CrOH	GeN	ClCl	ArS	NiC	

Note: The information provided in this table does not indicate that all of the described interferences need to be tested. However, the table can be consulted for informational purposes if unusual samples are encountered.

Table IV: Changes in Isobaric Molecular-Ion Interferences with Changing Plasma Conditions**

	Molecular Interference	Nebulizer Flow Rate		
		High	Average	Low
Oxides:	ScO/Sc	0.00326	0.00055	0.00116
	YO/Y	0.00568	0.00395	0.00353
	TbO/Tb	0.0156	0.00648	0.00614
	ClO, Cl	0.00725	0.00227	0.00233
Hydroxides:	ScOH/Sc	0.00040	0.00011	0.00000
	YOH/Y	0.00078	0.00044	0.00048
	TbOH/Tb	0.00034	0.00008	0.00011
	ClOH/Cl	0.00048	0.00031	0.00029
Chlorine:	ClO/Cl	0.00725	0.00227	0.00233
	ClOH/Cl	0.00048	0.00031	0.00029
	ArCl/Cl	0.00605	0.00091	0.00477

** Information for this table is being determined by the EPA.

Table V: Recommended Internal Standards

Method 6020	Method 200.8
Li	Sc
Sc	Y
Y	In
Rh	Tb
In	Bi
Tb	
Ho	
Bi	
Ge	

Table VI: Interference Check Sample Components and Concentrations

(ICSAB minors are suggested spike levels)

Interference Component	Solution A Concentration (mg/L)	Solution AB Concentration (mg/L)
Al	100.0	100.0
Ca	100.0	100.0
Fe	100.0	100.0
Mg	100.0	100.0
Na	100.0	100.0
P	100.0	100.0
K	100.0	100.0
S	100.0	100.0
C	200.0	200.0
Cl	1000.0	1000.0
Mo	2.0	2.0
Ti	2.0	2.0
As	0.0	0.1
Cd	0.0	0.1
Cr	0.0	0.1
Co	0.0	0.1
Cu	0.0	0.1
Mn	0.0	0.1
Ni	0.0	0.1
Se	0.0	0.1
Ag	0.0	0.1
V	0.0	0.1
Zn	0.0	0.1

Table VII: Sample Preservation and Holding Times

Measurement Parameter	Container (1)	Preservative (2)	Maximum Holding Time (3)
Waters:			
Metals (4)	P,G	HNO_3 to pH < 2	6 months
Soils/Sediments/Wastes:			
The preservation required for soil/sediment/waste samples is maintenance at 4°C (± 2°C) until digestion.			

Footnotes:

(1) Polyethylene (P) or glass (G).

(2) Sample preservation is performed by the sampler immediately upon sample collection.

(3) Samples must be analyzed as soon as possible after collection. The times listed are the maximum times that sample may be held before analysis and still considered valid. Holding times are calculated from the date when the sample was collected.

(4) Samples are filtered immediately on-site by the sampler before adding preservative for dissolved elements.

Table VIII: Suggested Mass Choices for Elements Which may be Monitored Either During The Analytical Run or in a Separate Scan

Boldface masses indicate the masses which must have the most impact on data quality and the elemental equations used to collect the data. Suggested masses for method 200.8 are in "quotes".

Mass	Element of Interest
"**27**"	Aluminum
121, "**123**"	Antimony
"**75**"	Arsenic
138, "137", 136, **135**, 134, 132, 130	Barium
"**9**"	Beryllium
114, 112, "111", 110, 113, 116, 106	Cadmium
42, 43, **44**, 46, 48	Calcium
"**52**", **53**, **50**, 54	Chromium
"**59**"	Cobalt
"**63**", **65**	Copper
56, **54**, **57**, 58	Iron
"**208**", "**207**", "**206**", 204	Lead
24, **25**, **26**	Magnesium
"**55**"	Manganese
58, "**60**", 62, **61**, 64	Nickel
39	Potassium
80, **78**, "**82**", **76**, **77**, 74	Selenium
"**107**", **109**	Silver
23	Sodium
"**205**", 203	Thallium
"**51**", **50**	Vanadium
64, "**66**", **68**, **67**, 70	Zinc
72	Germanium
139	Lanthanum
118	Tin
35, 37	Chlorine
"98", 96, 92, **97**, 94	Molybdenum

Note: It is strongly recommended that elements other than those of interest be monitored to indicate other potential molecular interferences which could affect the data quality.

Table IX: Tuning Solution

A tuning solution containing elements representing all of the mass regions of interest must be analyzed. Below are two groups of suggested solutions which cover a typical mass calibration range.

Method 6020

Element	Concentration (µg/L)
Solution A	
Mg	10.
Rh	10.
Pb	10.
Solution B	
Li	10.
Co	10.
In	10.
Tl	10.

Method 200.8

Element	Concentration (µg/L)
Be	10.
Mg	10.
Co	10.
In	10.
Pb	10.

Table X: Suggested Tuning and Response Factor Criteria

Minimum Response from Tuning Solution:

Be	>1,000
Mg	>2,000
Rh	>20,000
Pb	>10,000
Li	>2,000
Co	>20,000
In	>1,000
Tl	>1,000

Suggested Mass Calibration:

Be	9.0122
Mg	23.98
Rh	102.91
Pb	207.98
Li	7.016
Co	58.9332
In	114.904
Tl	204.9744

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QUANTERRA ENVIRONMENTAL SERVICES

Interim SOP Change - This is a controlled document

SOP NUMBER: **DEN-MT-0002, Revision 4**

SOP TITLE: Inductively Coupled Plasma – Mass Spectrometry, EPA Methods 6020 and 200.8

SOP SECTION(S) AFFECTED BY CHANGE: Table XI

REASON FOR CHANGE(S): Correct the frequency of the analysis of the ICSA solution, and the corrective action requirement.

Change Table XI, ICSA frequency from: Beginning and end of every run and every 12 hours.

To: Beginning of every analytical run or once every 12 hours, whichever is more frequent.

Change Table XI, ICSA and ICSAB corrective action from: See Section 9.6.

To: See Section 9.7.

SUBMITTED BY/DATE: **Mark J. Mensik/12-20-99**

APPROVED BY:

Richard Clinkscales, Technical Reviewer — Date 12/20/99

James S. Kelly, Health and Safety Coordinator — Date 12/20/99

Larry Penfold, Quality Assurance Manager (Mark J. Mensik) for — Date 12/20/99

Timothy M. O'Shields, Laboratory Manager — Date 12/20/99

Table XI: Summary of Quality Control Requirements

QC Parameter	Frequency*	Acceptance Criteria	Corrective Action
ICV/QCS	Beginning of every analytical run.	90 - 110% recovery.	Terminate analysis; correct the problem; recalibrate.
ICB/CB	Immediately after each ICV	The result must be < RL.	Terminate analysis; correct the problem; recalibrate.
CCV	Beginning and end of run and every 10 samples OR every 2 hours, whichever is more frequent. Beginning and end of each lot.	90 - 110% recovery.	Reanalyze once. If acceptable, continue. If unacceptable, terminate analysis; correct the problem recalibrate the instrument, reverify calibration and rerun all samples since the last acceptable CCV.
CCB	Immediately following each CCV.	The result must be < RL.	Reanalyze once. If acceptable, continue. If unacceptable, terminate analysis; correct the problem recalibrate the instrument, reverify calibration and rerun all samples since the last acceptable CCB.
ICSA (6020 Only)	Beginning and end of every run and every 12 hours.	Monitor for possible interferences.	See Section 9.6.7
ICSAB (6020 Only)	Immediately following each ICSA.	Monitor for possible interferences.	See Section 9.6.7

12/20/99

Revised 12/20/99

Table XI (cont.) Summary of Quality Control Requirements

QC Parameter	Frequency*	Acceptance Criteria	Corrective Action
Method Blank/Laboratory Reagent Blank	One per lot of 20 field samples or fewer.	The result must be < RL. Sample results greater than 20x the blank concentration or samples for which the contaminant is < RL, do not require redigestion or reanalysis.	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.2 for additional requirements.
Serial Dilution (6020 Only)	One per lot of 20 field samples or fewer.	90 - 110% recovery	See section 9.8 for additional requirements.
Post-Digestion Spike (6020 Only)	See section 9.10.	75-125% recovery	See section 9.8.
Matrix Spike/Laboratory Fortified Matrix	One per lot of 20 field samples or fewer.	Must be within laboratory control limits	See section 9.3 for additional requirements.

Table XII: ICP/MS Calibration and Calibration Verification Checklist

Suggested Levels in µg/L

Element	Calibration	ICV	CCV
Aluminum	100	200	100
Antimony	100	200	100
Arsenic	100	200	100
Barium	100	200	100
Beryllium	100	200	100
Cadmium	100	200	100
Chromium	100	200	100
Cobalt	100	200	100
Copper	100	200	100
Lead	100	200	100
Manganese	100	200	100
Nickel	100	200	100
Silver	100	200	100
Thallium	100	200	100
Zinc	100	200	100

This procedure has been developed for thirty-five elements (See Table VIII). Additional elements may be included in the calibration solution at the above levels. Levels may be adjusted to meet specific regulatory or client programs.

Appendix I: ICP/MS Data Review Checklist

Quanterra Environmental Services - Denver

ICP/MS Technical Data Review Checklist

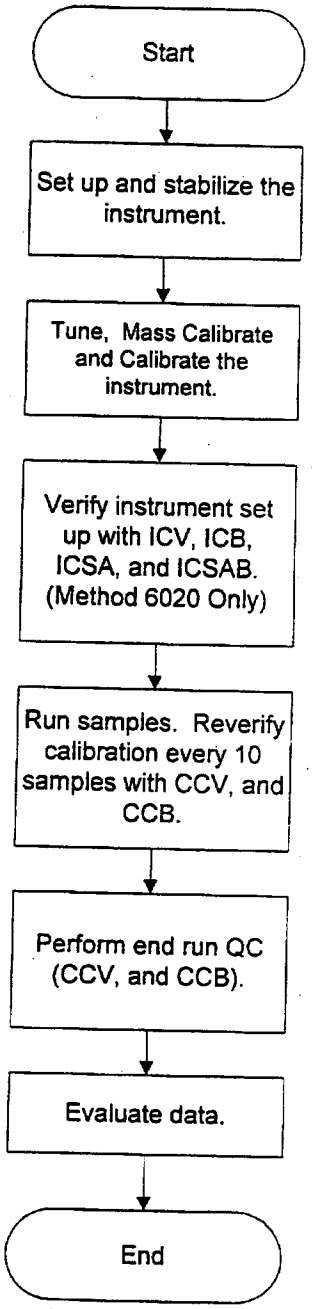
Lab Project ID Number(s): see attached cover sheet SOP # DEN-MT-0002 Check Method Used: ¬ 6020 ¬ 200.8

Review Items	--- Level 1 --- Yes	No	N/A	Level 2	Comments & Samples Affected
Tune					
1. Tune solution analyzed min. of 4 times for 6020 or 5 times for 200.8?					
2. Tune RSD <5%?					
3. Resolution ≤ 0.9 AMU full width at 10% peak height, & within ± 0.1 amu of true mass?					
Initial Calibration					
1. Done with a minimum of 3 integrations of a high standard and blank?					
Initial and Continuing Calibration Verification (ICV/CCV/QCS)					
1. Run at beginning of run, 10% frequency, and end of run?					
2. Measured result within 10% of expected value?					
Calibration Blank (ICB/CCB)					
1. Run at beginning of run, 10% frequency, and end of run?					
2. Measured result ≤ RL?					
Client Samples & QC Sample Results					
1. 10% of samples screened for presence of IS interferences?					
2. Were all samples within linear range, ≤ 90% of LDR for 200.8?					
3. Dilutions due to target elements?____ Dilutions for other reasons?___					
4. All reported results bracketed by in control QC?					
5. All 6020 internal standards for samples 30-120% of intensities in blank or all 200.8 internal standards 60-125% ?					
6. Was a 5X serial dilution analyzed for 6020 and, if so, are results ± 10% of original result, if original ≥ 100x RL?					
7. LCS included in batch and within QC limits?					
8. Method blank included and <RL?					
9. MS and MSD included in batch and within limits?					
10. If serial dilution, PDS analyzed and recovery 75-125%?					
11. Manual calculations documented properly and checked?					
12. Are nonconformances documented on an NCM?					
13. Is the appropriate raw data included?					

1st Level Reviewer: _____ Date: _____

2nd Level Reviewer: _____ Date: _____

Appendix II: Flow Chart



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SOP No. CORP-MT-0005DEN
Revision No. 1.2
Revision Date: 5/20/99
Implementation Date: 5/25/99

DENVER-SPECIFIC

QUANTERRA 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 1.1)

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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. 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.

1.3. Method 7470A is applicable to the preparation and analysis of mercury in ground water, aqueous samples, wastes, wipes, TCLP, EP and other leachates/extracts. Certain solid and sludge type wastes may also be analyzed, however Method 7471A (see *CORP-MT-0007DEN*) is usually the method of choice. All matrices require sample preparation prior to analysis.

1.4. Method 245.1 is applicable to the determination of mercury in drinking, surface and saline waters, domestic and industrial wastes. All matrices require sample preparation prior to analysis.

1.5. The Quanterra reporting limit for mercury in aqueous matrices is 0.0002 mg/L except for TCLP, SPLP or EPTOX leachates for which the reporting limit is 0.002 mg/L.

2. SUMMARY OF METHOD

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 μm membrane. (Sample is acidified after filtration).

3.2. *Suspended Metals*: Those elements, which are retained by a 0.45 μm 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. Seawaters, 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 head space before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

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 Quanterra 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. 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.

5.7. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.

5.8. 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.

5.9. The CVAA apparatus must be properly vented to remove potentially harmful fumes generated during sample analysis.

6. EQUIPMENT AND SUPPLIES

6.1. Temperature controlled, mechanically recirculating water bath capable of maintaining a temperature of 90-95°C.

6.2. Mercury Autoanalyzers

6.2.1. Leeman Labs PS200 with Autosampler

6.2.2. CETAC Mercury Analyzer with Autosampler and Auto-Diluter

6.3. BOD bottles or equivalent.

6.4. *Mercury-free air*, 99.999% Nitrogen supply, compressed cylinder.

6.5. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.6. Class A volumetric flasks.

6.7. Calibrated 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. 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.

7.3. Sulfuric acid (H_2SO_4), concentrated, trace metal grade or better.

7.3.1. Sulfuric acid, 0.5 N: Dilute 14.0 mL of concentrated H_2SO_4 to 1 liter with reagent water.

7.4. Stannous Chloride Solution, 10% (w/v) per manufacturer's instructions

7.4.1. Place approximately 50mL of deionized water into a 1L volumetric flask

7.4.2. Slowly add 100mL of concentrated HCl to the flask and swirl to mix.

7.4.3. Add 100 grams of $SnCl_2$ to the flask.

7.4.4. Place a large stir bar in the flask and put the flask on a stir plate.

7.4.5. Stir the contents of the flask until the reagent is completely dissolved.

7.4.6. Remove the stir bar and make to volume with deionized water.

7.5. 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.

7.6. Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate (reagent grade, "suitable for mercury determination") for every 100 mL of reagent water.

7.7. Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate for every 100 mL of reagent water.

7.8. Purchased Mercury Stock Solutions

7.8.1. 1000 mg/L Hg, primary calibration standard

7.8.2. 10 mg/L Hg, second-source (different vendor than primary calibration standard

7.9. Preparation of the 10 mg/L Calibration Working Standard Solution.

7.9.1. Add approximately 90 mL of 1% HNO_3 to a 100 mL Class A volumetric flask.

7.9.2. Pipet 1.00 mL of the 1000 mg/L stock solution into the flask.

7.9.3. Dilute to the mark on the flask with 1% HNO_3.

7.9.4. Stopper the flask and shake to mix.

7.9.5. Transfer the solution to a 125 mL Nalgene bottle.

7.9.6. Label the bottle in accordance with Quanterra's current policies.

7.9.7. Document the preparation of the solution in the Hg Standards Preparation Log.

7.9.8. Prepare this solution fresh monthly or more often if necessary.

7.10. Preparation of the 100 µg/L Daily Calibration Working Solution

7.10.1. Place a 125 mL Nalgene bottle on the analytical balance and tare.

7.10.2. Add approximately 90 mL (g) of 1% HNO_3 to the bottle.

7.10.3. Pipet 1.00 mL of the 10 mg/L Calibration Working Standard to the bottle.

7.10.4. Bring the solution to a final weight of 100.0 grams.

7.10.5. Cap the bottle and swirl the contents to mix.

7.10.6. Using a permanent marker, label the bottle with the date and "100 µg/L Hg".

7.10.7. Document the preparation of the solution in the Hg Standards Preparation Log.

7.10.8. Prepare this solution each day prior to calibration.

7.11. Preparation of the calibration standards.

7.11.1. 0.2 ug/l Hg standard

7.11.1.1. Place a 125 mL Nalgene bottle on the analytical balance and tare.

7.11.1.2. Add approximately 90 mL (g) of 1% HNO3 to the bottle.

7.11.1.3. Pipet 0.20 mL of the 100 µg/L Calibration Working Solution into the bottle.

7.11.1.4. Bring the solution to a final weight of 100.0 grams.

7.11.1.5. Cap the bottle and swirl the contents to mix.

7.11.1.6. Using a permanent marker, label the bottle with the date and "0.2 µg/L Hg".

7.11.2. 0.5, 1.0, 2.0, 5.0, 10.0 µg/L Hg Calibration Standards

7.11.2.1. Place a 125 mL Nalgene bottle on the analytical balance and tare.

7.11.2.2. Add approximately 80 mL (g) of 1% HNO_3 to the bottle.

7.11.2.3. Pipet either 0.50, 1.0, 2.0, 5.0 or 10.0 mL of the 100 µg/L Calibration Working Solution into the bottle depending on the standard being prepared.

7.11.2.4. Bring the solution to a final weight of 100.0 grams.

7.11.2.5. Cap the bottle(s) and swirl the contents to mix.

7.11.2.6. Using a permanent marker, label the bottle with the date and concentration of the standard.

7.11.2.7. Document the preparation of the solutions in the Hg Standards Preparation Log.

7.11.2.8. Prepare the calibration solutions each day prior to calibration.

7.12. Preparation of LCS and CCV Standards.

7.12.1. The LCS and CCV's are prepared exactly as the 5.0 µg/L Calibration standard.

7.12.2. Prepare sufficient volume of the standard for analysis of an LCS/LCSD and a CCV after every 10 samples.

7.13. Preparation of the 100 µg/L ICV Working Standard.

7.13.1. Place a 125 mL Nalgene bottle on the analytical balance and tare.

7.13.2. Add approximately 90 mL (g) of 1% HNO_3 to the bottle.

7.13.3. Pipet 1.00 mL of the 10 mg/L Stock Solution (Env. Exp.) into the bottle.

7.13.4. Bring the solution to a final weight of 100.0 grams.

7.13.5. Cap the bottle and swirl the contents to mix.

7.13.6. Using a permanent marker, label the bottle "100 µg/L ICV Stock" and the date prepared.

7.13.7. Document the preparation of the solution in the Hg Standards Preparation Log.

7.13.8. Prepare this solution daily prior to analysis.

7.14. Preparation of the Daily 2.0 µg/L ICV Standard

7.14.1. Place a 125 mL Nalgene bottle on the analytical balance and tare.

7.14.2. Add approximately 90 mL (g) of 1% HNO_3 to the bottle.

7.14.3. Pipet 2.00 mL of the "100 µg/L ICV Stock" into the bottle.

7.14.4. Cap the bottle and swirl the contents to mix.

7.14.5. Using a permanent marker, label the bottle "ICV" and the date prepared.

7.14.6. Document the preparation of the solution in the Hg Standards Preparation Log.

7.14.7. Prepare this solution daily prior to analysis.

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. For details concerning frequency and performance of MDL studies, see Quanterra Policy QA-005.

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. 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 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.**

9.4. 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.

9.5. 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.

9.6. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). *The ICV result must be within 10% of the true value.* An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The absolute result for the ICB must be less than the reporting limit (RL). 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 associated samples.

Revised MM 11/19/99

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 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 analysis by 7470A. When performing Hg analysis by 245.1, the CCV results must fall within +/- 5% of the true value. 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. If a mid-run CCV or CCB fails, the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV or CCB failure was not directly instrument related the corrective action will include repreparation of the associated samples.

Revised MM 11/19/99

9.8. 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.

QUANTERRA ENVIRONMENTAL SERVICES

Interim SOP Change - This is a controlled document

SOP NUMBER: **CORP-MT-0005DEN, Revision 1.2**

SOP TITLE: PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS SAMPLES BY COLD VAPOR ATOMIC ABSORPTION, SW846 7470A AND MCAWW 245.1

SOP SECTION(S) AFFECTED BY CHANGE: 9.6

REASON FOR CHANGE(S): To revise the ICV limits for Method 245.1 to comply with Revision 3.0. The limit should be +/- 5% of the true value, not +/- 10% as currently specified in the SOP.

Change section 9.6 from: Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). *The ICV result must be within 10% of the true value.* An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The absolute result for the ICB must be less than the reporting limit (RL). 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 associated samples.

To: 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 analysis by 7470A. When performing Hg analysis by 245.1, the ICV results must fall within +/- 5% of the true value.* An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The absolute result for the ICB must be less than the reporting limit (RL). 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 associated samples.

SUBMITTED BY/DATE: **Mark J. Mensik, 11/19/99**

APPROVED BY:

Richard Clinkscales, Technical Reviewer	**Date** 11/19/99
James S. Kelly, Health and Safety Coordinator	**Date** 11/19/99
Larry Penfold, Quality Assurance Manager	**Date** 11/19/99
Timothy M. O'Shields, Laboratory Manager	**Date** 11/19/99
Corporate Technology	**Date** 11/19/99

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Change section 9.7 from: 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 analysis by 7470A. When performing Hg analysis by 245.1, the CCV results must fall within +/- 5% of the true value. A CCB is analyzed immediately following each CCV.....

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SUBMITTED BY/DATE: **Mark J. Mensik, 11/19/99**

APPROVED BY:

Richard Clinkscales, Technical Reviewer	Date 11/19/99
James S. Kelly, Health and Safety Coordinator for	Date 11/19/99
Larry Penfold, Quality Assurance Manager	Date 11/19/99
Timothy M. O'Sheids, Laboratory Manager	Date 11/19/99
Corporate Technology	Date 11/19/99

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 Quanterra 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 $\geq$ 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.

11.1.2. Transfer 10.0 mL of well mixed sample or standard to a clean sample digestion bottle.

11.1.3. Add 0.5 mL of concentrated H_2SO_4 and 0.25 mL of concentrated HNO_3 mixing after each addition.

Note: All spiking should be done after the initial addition of acids.

11.1.4. Add 1.5 mL of 5% 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: It is important that equal volumes of the potassium permanganate solution are added to all solutions in the batch. Unequal volumes used with the automated method will result in dilution errors. Unequal volumes used with the manual method will result in differing purging efficiencies.

11.1.5. Add 0.8 mL of potassium persulfate solution and heat for two hours in a water bath at 90 - 95°C.

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 0.6 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. Automated determination: Follow instructions provided by instrument manufacturer.

11.2.5. 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.

11.2.6. 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.

11.2.7. The samples must be allowed to cool to room temperature prior to analysis or a decrease in the response signal can occur.

11.2.8. 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.

11.2.9. 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, and field samples.

11.2.10. The following run sequence is consistent with 7470A, CLP and 245.1 and may be used as an alternate to the sequence in 11.2.9. 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.

11.2.11. 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 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. The final concentration for an aqueous sample is calculated as follows:

$$ug/L = C \times D$$

Where:
C = Concentration (ug/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 Quanterra significant figure policy.

12.9. 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).

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. Refer to Quanterra SOP # CORP-QA-0013 for details concerning analyst proficiency testing and documentation.

14. POLLUTION PREVENTION

This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

15. WASTE MANAGEMENT

The laboratory maintains a hazardous waste stream for mercury. Refer to the Quanterra Chemical Hygiene Plan for waste management 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, 1994, Method 245.1, Revision 3.0.

16.3. U.S.EPA Statement of Work for Inorganics Analysis, ILMO3.0.

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 11.2.10 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 Alone

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.

17.1.3. Modifications from 245.1 Alone

Method 245.1 Section 9.3 states concentrations betweeen 1 and 10 ug/L should be reported to one significant figure, and results above 10 ug/L to the nearest ug/L unit value (e.g. two figures in 10-99 ug/L range, three figures in 100-999 ug/L range, etc.). Quanterra reports all Hg results under this SOP to two significant figures.

17.2. Modifications from previous SOP

Revised MAM 11/19/99

17.2.1. The requirements from Method 245.1 regarding acceptance criteria for ICV and CCV have been incorporated. Specifically that the ICV result must fall within +/- 10% and the CCV results must fall within +/- 5%. These criteria only apply when Method 245.1 is employed.

17.2.2. Details concerning the preparation of standards have been added (Sections 7.8 through 7.14).

17.2.3. The option to perform digestions using the autoclave was removed.

17.2.4. The detail about adding equal amounts of additional permanganate solution to all solutions in the batch was corrected (Section 11.1.4).

17.2.5. Cross references to Quanterra MDL policy and Quanterra training SOP added

QUANTERRA ENVIRONMENTAL SERVICES

Interim SOP Change - This is a controlled document

SOP NUMBER: **CORP-MT-0005DEN, Revision 1.2**

SOP TITLE: PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS SAMPLES BY COLD VAPOR ATOMIC ABSORPTION, SW846 7470A AND MCAWW 245.1

SOP SECTION(S) AFFECTED BY CHANGE: 17.2.1

REASON FOR CHANGE(S): To correctly state the ICV and CCV limits for Method 245.1, Revision 3.0, as +/- 5% and +/- 10% of the true value, respectively.

Change section 17.2.1 from: The requirements from Method 245.1 regarding acceptance criteria for ICV and CCV have been incorporated. Specifically that the ICV result must fall within +/- 10% and the CCV results must fall within +/- 5%. These criteria only apply when Method 245.1 is employed.

To: The requirements from Method 245.1 regarding acceptance criteria for ICV and CCV have been incorporated. Specifically that the ICV result must fall within +/- 5 and the CCV results must fall within +/- 10 These criteria only apply when Method 245.1 is employed.

SUBMITTED BY/DATE: **Mark J. Mensik, 11/19/99**

APPROVED BY:

Richard Clinkscales, Technical Reviewer — Date 11/19/99

James S. Kelly, Health and Safety Coordinator — Date 11/19/99

Larry Penfold, Quality Assurance Manager — Date 11/19/99

Timothy M. O'Shields, Laboratory Manager — Date 11/19/99

Corporate Technology — Date 11/19/99

Figure 1. Aqueous Sample Preparation - Mercury

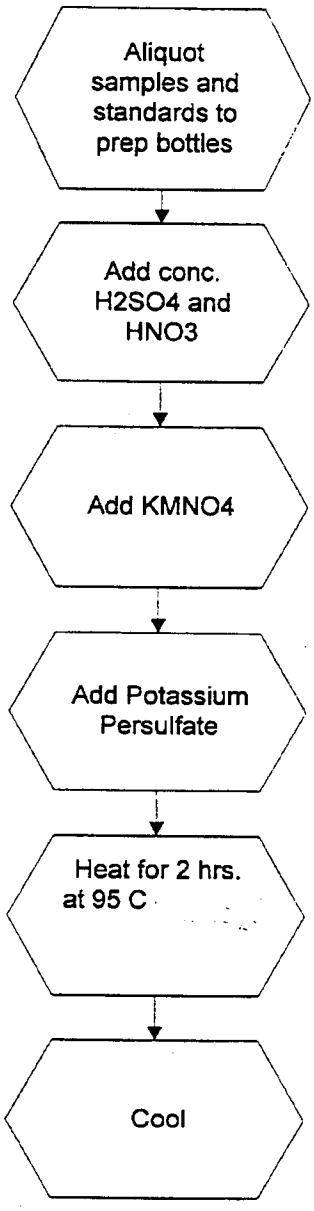
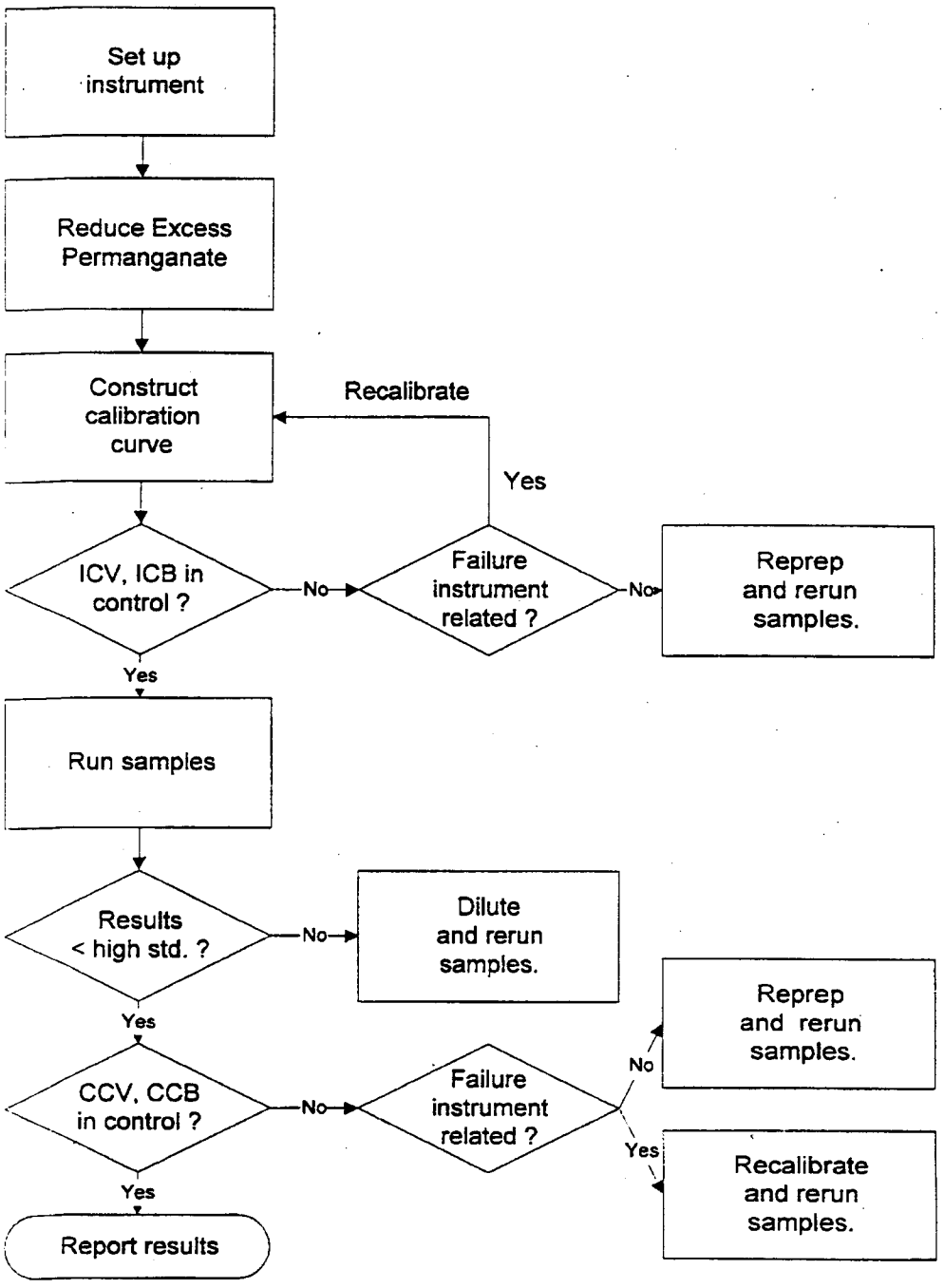


Figure 2. CVAA Mercury Analysis



APPENDIX A

TABLES

TABLE I . MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC STANDARD AND SPIKING LEVELS (µg/L)

	Leeman Labs PS200	*CETAC*
Standard Aqueous RL	*0.2*	*0.2*
TCLP RL	*0.2*	*0.2*
Std 0	*0*	*0*
Std 1	*0.2*	*0.2*
Std 2	*0.5*	*0.5*
Std 3	*1.0*	*1.0*
Std 4	*5.0*	*2.0*
Std 5	*10.0*	*5.0*
*Std 6***	---	*10.0*
ICV	*2.0*	*2.0*
LCS/CCV	*5.0*	*5.0*
Aqueous MS	*5.0*	*5.0*
TCLP MS	*5.0*	*5.0*

* **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.**

Revised MAM 11/19/99

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 batch (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 batch (see Section 9.7).
CCV	Every 10 samples and at the end of the run.	80 - 120 % recovery. 95 - 105% recovery for Method 245.1	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep batch (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 batch (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 11.2.9 and 11.2.11 for exact run sequence to be followed.

Revised MM 11/19/99

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 11.2.11
Matrix Spike Duplicate	See Matrix Spike	75 - 125 % recovery or in-house control limits; RPD $\leq$ 20%. (See MS)	See Corrective Action for Matrix Spike.

QUANTERRA ENVIRONMENTAL SERVICES

Interim SOP Change - This is a controlled document

SOP NUMBER: **CORP-MT-0005DEN, Revision 1.2**

SOP TITLE: PREPARATION AND ANALYSIS OF MERCURY IN AQUEOUS SAMPLES BY COLD VAPOR ATOMIC ABSORPTION, SW846 7470A AND MCAWW 245.1

SOP SECTION(S) AFFECTED BY CHANGE: TABLE II

REASON FOR CHANGE(S): To correctly state the ICV and CCV acceptance criteria for Method 245.1, Revision 3.0, and correctly state the reference sections for ICV, ICB, CCV, and CCB in the Corrective Action column.

Change TABLE II: Refer to revised table on second page of this interim change form.

SUBMITTED BY/DATE: **Mark J. Mensik, 11/19/99**

APPROVED BY:

Richard Clinkscales, Technical Reviewer — Date 11/19/99

James S. Kelly, Health and Safety Coordinator for — Date 11/19/99

Larry Penfold, Quality Assurance Manager — Date 11-19-99

Timothy M. O'Shields, Laboratory Manager — Date 11/19/99

Corporate Technology — Date 11/19/99

TABLE II. Summary Of Quality Control Requirements (Revision 11/19/99)

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICV	Beginning of every analytical run.	90 – 110 % recovery. *95 – 105% recovery for Method 245.1*	Terminate analysis; Correct the problem; Recalibrate or reprep batch (see Section 9.6).
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 batch (see Section 9.6).
CCV	Every 10 samples and at the end of the run.	80 – 120 % recovery. *90 – 110% recovery for Method 245.1*	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV or reprep batch (see Section 9.7).
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 batch (see Section 9.7).
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 11.2.9 and 11.2.11 for exact run sequence to be followed.

TABLE II. Summary of Quality Control Requirements (Continued) (Revision 11/19/99)

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 9.4).
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 9.5) For TCLP see Section 11.2.11
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
QUANTERRA DENVER Hg CHECKLIST

QUANTERRA ENVIRONMENTAL SERVICES, INC.

Industrial Mercury Analysis Raw Data Checklist

Revision 0 -March 25, 1997

Analyst's Checklist

		yes	no	n/a
1.	Were the special instructions for prep and/or analysis followed?	☐	☐	☐
2.	Is the correlation coefficient ≥ 0.995?	☐	☐	☐
3.	Is the blank less than 2 times the reporting limit or properly anomalized?	☐	☐	☐
4.	Are the DCSs within limits or properly anomalized?	☐	☐	☐
5.	Is the ICV and all CCVs within limits?	☐	☐	☐
6.	Are all CCBs within ± one reporting limit from zero?	☐	☐	☐
7.	Were the CCVs and CCBs run with only 10 samples between each set?	☐	☐	☐
8.	Are the reporting limits correct and reflect any dilutions?	☐	☐	☐
9.	Are the number of significant figures correctly reported?	☐	☐	☐
10.	Are the benchsheets complete (including calibration and standard verification #'s)?	☐	☐	☐
11.	Are all comments, footnotes, and anomalies properly documented?	☐	☐	☐
12.	Are holding time violation and rerun forms completed and attached?	☐	☐	☐
13.	Has all sample data been entered into LIMS?	☐	☐	☐
14.	Has all DCS and/or LCS Prep Blank data been entered into LIMS?	☐	☐	☐
15.	Has the data entered into LIMS been checked for errors?	☐	☐	☐
16.	For TCLP results, is the sample data within 20% of Regulatory Level (0.2 mg/L)?	☐	☐	☐

Analyst's Name:_____ Date:_____

Data Review's Checklist

		yes	no	n/a
1.	Have the calculations been checked?	☐	☐	☐
2.	Is the correlation coefficient ≥ 0.995?	☐	☐	☐
3.	Is all the QC data within the control limits and/or properly anomalized?	☐	☐	☐
4.	Are all the significant figures and reporting limits correct?	☐	☐	☐
5.	Have any comments, footnotes, and anomalies been properly documented?	☐	☐	☐
6.	Have any data errors been documented and entered into LIMS?	☐	☐	☐
7.	Has the raw data been copied and filed?	☐	☐	☐
8.	Has the data package been copied and filed?	☐	☐	☐
9.	If TCLP result ≤ 20% of Reg. Level and matrix spike < 50%, was MSA performed?	☐	☐	☐

Reviewed by:_____ Date:_____

Comments:_____

_____

_____

Anomalies:_____

_____

_____

APPENDIX C

MSA GUIDANCE

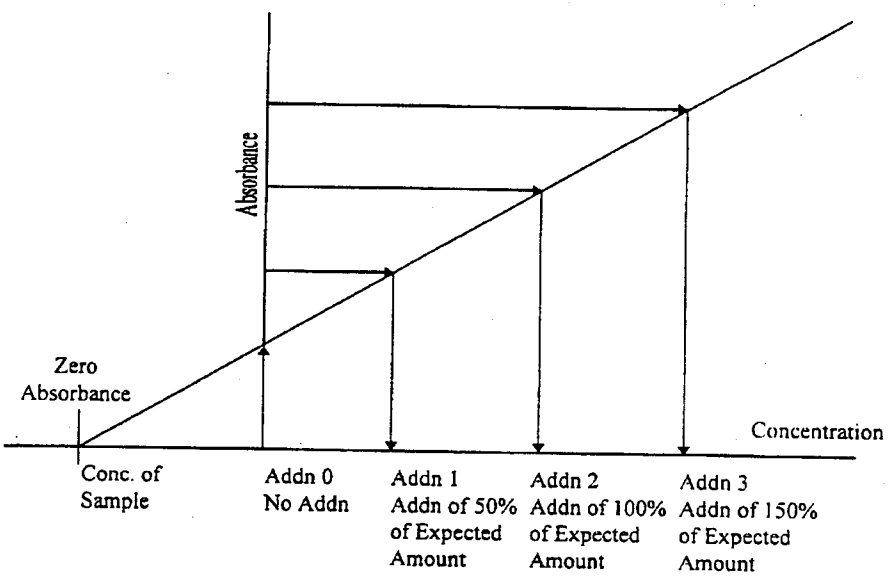
APPENDIX C. MSA GUIDANCE

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 pre-warmed 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 and CETAC Analyzer)

Daily	Monthly	Annually
Change rinse solution.	Check Hg lamp intensity.	Change Hg lamp.
Check aperture.		Check liquid/gas separator.
Check nitrogen flow.		
Check tubing. Replace as needed.		
Check drain.		
Replace magnesium perchlorate		

Quanterra
Environmental Services

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SOP No. CORP-MT-0007DEN
Revision No. 1.1
Revision Date: 3/26/97

DENVER-SPECIFIC

QUANTERRA 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 0)

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Approved by: [signature] 3/26/97
Laboratory Director, Thomas Daniels

Approved by: [signature] 3/28/97
Corporate Technology and/or Corporate Quality Assurance

Proprietary Information Statement:

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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. 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.

1.3. Methods 7471A and 245.5 are applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits and sludge-type materials. All matrices require sample preparation prior to analysis.

1.4. The Quanterra reporting limit for mercury in solid matrices is 0.1 mg/kg based a 0.2 g sample aliquot (wet weight).

2. SUMMARY OF METHOD

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

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 Quanterra 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. 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.

5.7. Do not look directly into the beam of the Hg lamp. The UV light that these lamps radiate is harmful to the eyes.

5.8. 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.

5.9. 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) or autoclave capable of obtaining 15 lbs., 120°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 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. *Mercury-free air.*

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. *Mercury standard (1 ppm): Obtain a NIST-traceable mercury standard from commercial sources. The standard must be replaced at least every six months.*

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7.3. The calibration standards must be prepared fresh daily from the working standard (7.4) by transferring 0, 0.2, 0.5, 1.0, 2.0, 5.0 and 10.0 mL aliquots of the working mercury standard into sample prep 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,

QUANTERRA ENVIRONMENTAL SERVICES
Interim SOP Change - This is a controlled document

SOP NUMBER:	CORP-MT-0007DEN
SOP TITLE:	**Preparation and Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption, SW846 7471A and MCAWW 245.5**
SOP SECTION(S) AFFECTED BY CHANGE:	**7.2 through 7.13**
REASON FOR CHANGES:	Discrepancies discovered during follow-up to Quanterra corporate audit of 9/97.

Change section 7.2 from: "7.2 *Mercury standard (1 ppm): Obtain a NIST-traceable mercury standard from commercial sources. The standard must be replaced at least every six months.*"

To: Delete this section from the SOP

Change section 7.3 from: "7.3 The calibration standards must be prepared fresh daily from the working standard (7.4) by transferring 0, 0.2, 0.5, 1.0, 2.0, 5.0 and 10.0 mL aliquots of standard into 100 mL flasks and diluting to volume with reagent water."

To: "7.2 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 standard into 100 mL flasks and diluting to volume with reagent water."

Sections 7.4 through 7.13 to be renumbered 7.3 through 7.12

SUBMITTED BY/DATE: Mark Mensik, 7/31/98

APPROVED BY:

Richard Burrows, Technical Reviewer	**Date** 8/3/98
Jim Travis, Technical Specialist	**Date** 8-25-98
Larry Penfold, Quality Assurance Manager	**Date** 9-2-98
Tim O'Shields, Laboratory Manager	**Date** 8/28/98

automated mercury systems do not require 100 mL of standard and therefore smaller volumes may be generated to reduce waste generation.

7.4. The initial calibration verification standard must be made from a different stock solution than that of the calibration standards.

7.5. 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.

7.6. 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.

7.7. Sulfuric acid (H_2SO_4), concentrated, trace metal grade or better.

7.7.1. Sulfuric acid, 0.5 N: Dilute 14.0 mL of concentrated H_2SO_4 to 1 liter with reagent water.

7.8. Hydrochloric acid (HCl), concentrated, trace metal grade or better.

7.9. Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3.

7.10. 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.

7.11. 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.

7.12. Potassium permanganate, 5% solution (w/v): Dissolve 5 g of potassium permanganate for every 100 mL of reagent water.

7.13. 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 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 Quanterra 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) 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. 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. The CCV results can be reported as the LCS results since all CCVs (as well as all other standards) are processed through the sample preparation step with the field samples. No more than

20 samples can be associated with one CCV used for the purpose of reporting LCS data.

- 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 be within 10% of the true value.* 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 associated samples.

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. If a mid-run CCV or CCB fails, the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV or CCB failure was not directly instrument related the corrective action will include repreparation of the associated samples.

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 Quanterra 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, 2.0, 5.0 and 10.0 mL aliquots of the working standard (7.5) into a series of sample digestion bottles.

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 or 11.1.6 below.

11.1.4. Transfer triplicate 0.2 g portions of a well mixed sample into a clean sample digestion bottle. Continue preparation as described under 11.2.2 or 11.2.3 below.

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 5 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. Cool.

11.1.5.4. Add 50 mL of distilled water.

11.1.5.5. Add 15 mL of potassium permanganate solution.

11.1.5.6. Add 8 mL of potassium persulfate solution, mix thoroughly.

11.1.5.7. Heat for 30 minutes in the water bath at 90 - 95°C.

11.1.5.8. Cool.

11.1.5.9. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.

11.1.5.10. To each **standard** bottle: Add 50 mL of reagent water.
To each **sample** bottle: Add 55 mL of reagent water.

11.1.5.11. Continue as described under Section 11.2.

11.1.6. Autoclave protocol:

11.1.6.1. Add 5 mL concentrated of H_2SO_4 and 2 mL of concentrated HNO_3.

11.1.6.2. Add 5 mL of saturated potassium permanganate solution.

11.1.6.3. Add 8 mL of potassium persulfate solution.

11.1.6.4. Cover digestion bottle with aluminum foil or screw cap loosely applied.

11.1.6.5. Heat samples at 121°C and 15 lbs. pressure for 15 minutes.

11.1.6.6. Cool.

11.1.6.7. Make up to volume of 100 mL with reagent water.

Note: Alternate final volumes may be used as long as the standards and sample are treated the same way.

11.1.6.8. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce excess permanganate.

11.1.6.9. 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. *Record the maximum absorbance value. When the signal levels off, remove the aerator from the BOD bottle.*

11.2.3.4. Place the aeration device into 100 mL of 1% HNO_3 and allow to bubble rinse until the next sample is analyzed.

11.2.4. 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. µg 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 (µg/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 (µg) 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 Quanterra 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 blank, laboratory control sample, 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

This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

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, Quanterra 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.

17.1.2.3. *This section has been deleted.*

17.1.3. Modifications from 245.5

Method 245.5 Section 9.3 states concentrations should be reported as follows: Between 0.1 and 1 µg/g, to the nearest 0.01 µg; between 1 and 10 µg/g, to the nearest 0.1µg; above 10 µg/g, to the nearest µg. Quanterra reports all Hg results under this SOP to two significant figures.

17.2. Modifications from previous SOP

None.

17.3. 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 - Autoclave Procedure

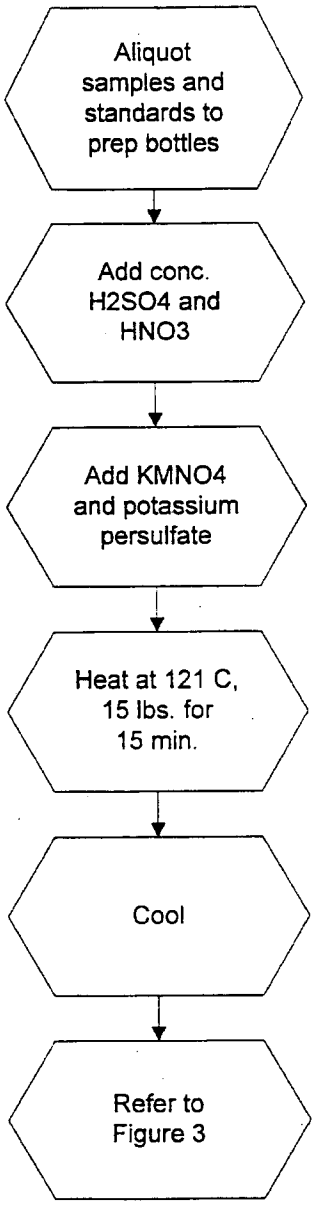


Figure 2. Solid Sample Preparation for Mercury - Water Bath Procedure

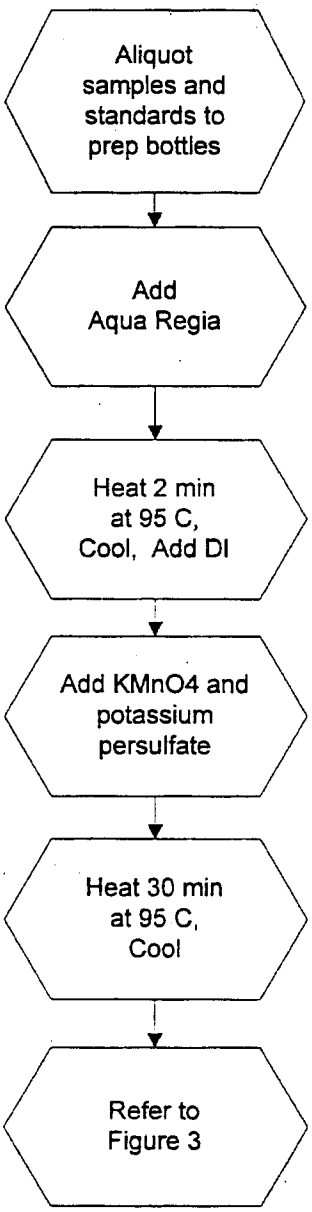
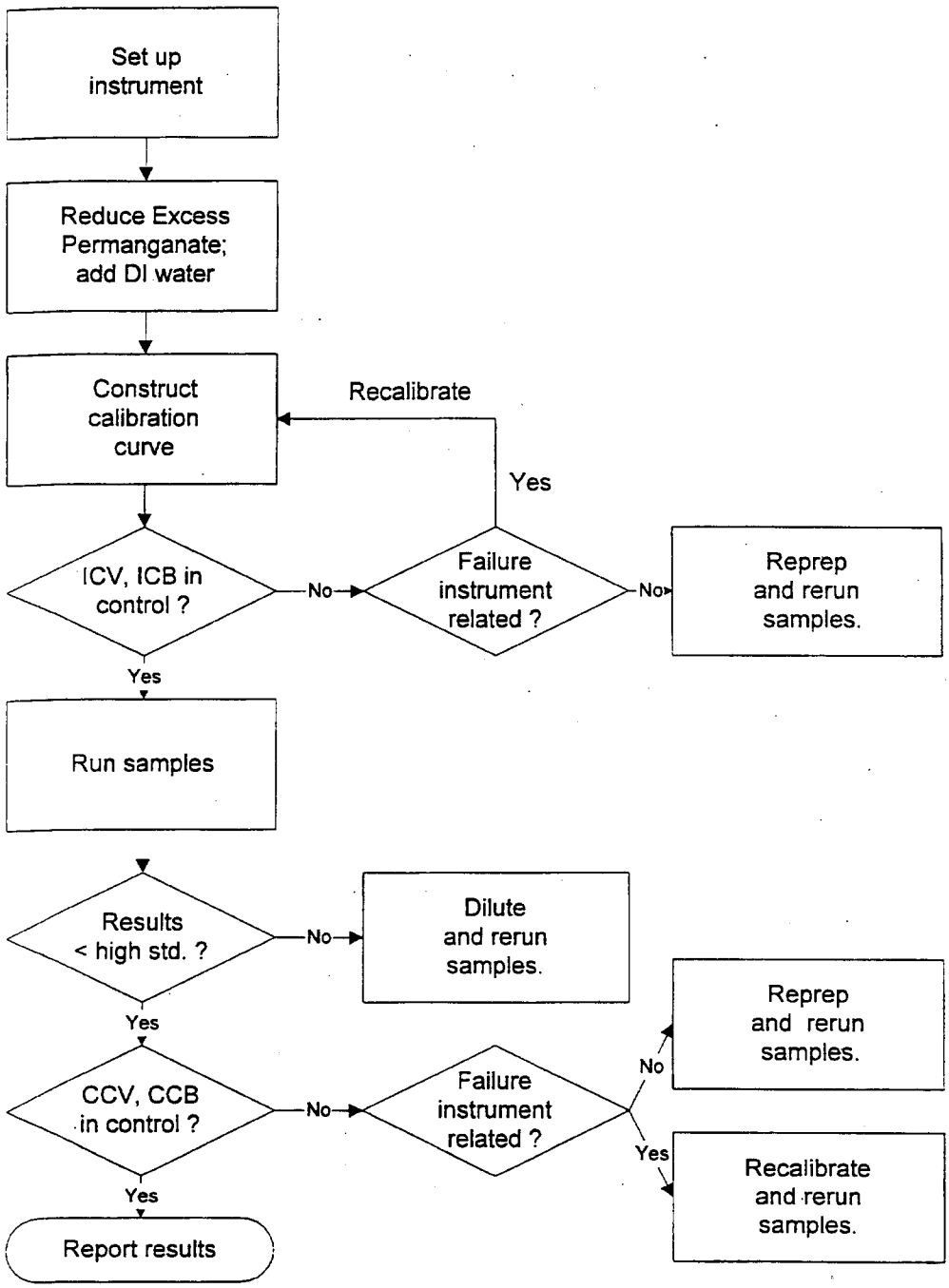


Figure 3. CVAA Mercury Analysis



APPENDIX A

TABLES

TABLE I . MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC STANDARD AND SPIKING LEVELS (µg/L)

Revised 9/4/98

	Other Hg Instruments	*Spectra Instrument*
Soil RL	*0.2*	*0.2*
Std 0	*0*	*0*
Std 1	*0.2*	*0.2*
Std 2	*0.5*	*0.5*
Std 3	*1.0*	*1.0*
Std 4)	*2.0*	*1.5*
Std 5	*5.0*	*2.0*
Std 6 **	*10*	---
ICV	*1.0 or 2.5* ***	*0.5*
CCV/LCS	*2.5 or 5.0* ***	*1.0*
MS	*1.0*	*1.0*

* **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.**

QUANTERRA ENVIRONMENTAL SERVICES
Interim SOP Change - This is a controlled document

SOP NUMBER:	CORP-MT-0007DEN
SOP TITLE:	**Preparation and Analysis of Mercury in Solid Samples by Cold Vapor Atomic Absorption, SW846 7471A and MCAWW 245.5**
SOP SECTION(S) AFFECTED BY CHANGE:	**Table I**
REASON FOR CHANGES:	Discrepancies discovered during follow-up to Quanterra corporate audit of 9/97.
SUBMITTED BY/DATE:	Mark Mensik, 7/31/98
APPROVED BY:	
Richard Burrows, Technical Reviewer	**Date** 8/31/98
Jim Travis, Technical Specialist	**Date** 8-25-98
Larry Penfold, Quality Assurance Manager	**Date** 9-2-98
Tim O'Shields, Laboratory Manager	**Date** 8/28/98

TABLE I. MERCURY REPORTING LIMITS, CALIBRATION STANDARD*, QC STANDARD AND SPIKING LEVELS (µg/L)

	Leeman Labs PS 200	*Spectro Instrument*
Soil RL	*0.2*	*0.2*
Std 0	*0*	*0*
Std 1	*0.2*	*0.2*
Std 2	*0.5*	*0.5*
Std 3	*1.0*	*1.0*
Std 4	*5.0*	*1.5*
Std 5	*10*	*2.0*
ICV	*2.0***	*0.5*
CCV/LCS	*5.0***	*1.0*
MS	*1.0*	*1.0*

* **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.**

** **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 batch (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 batch (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 batch (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 batch (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
QUANTERRA Hg CHECKLISTS AND BENCHSHEETS

Quanterra 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:**_____

_____

_____

Example Mercury Prep/Analysis Benchsheet

Seal# Corp-MT-0003
Revision 1
12-18-95

Quanterra - Denver
Inorganic Mercury Prep/Analysis
Aqueous

QC Lot 14FEB97-SA | Prep by AALBAN | Analyst AALBAN | Prep Date 14-Feb-97 | Analysis Date 14-Feb-97

Bottle Number		Std. (ppm)	Reading (mV)
50	13:10	0.0000	0.0048
333	:11	0.0002	0.0214
287	:12	0.0005	0.0459
7	:13	0.0010	0.0879
247	:14	0.0015	0.1230
48	:15	0.0020	0.1582

Time on	10:00	Time off	12:00
ICV/LCS Lot #	10354	Exp. Date	Feb-97
Calib. Lot#	10444	Exp. Date	Jul-97
Corr. Coeff.	0.9992 (>0.995)		
Y Intercept	0.005774		
Instrument	Hg-A		
slope	77.038445	X=(Y-B)/M	

Sequence Number	Matrix	Sample	Sample Mass (g)	Sample Volume	Prep Dilution	Dilution Aliquot	Reading (mV)	Initial Concentration (ppm)	D/F	Final Conc. (ppm)	Final Rep. Lt.	% Rec.	Time
49	H2O	ICV	100	136.5	1		0.1255	0.00154	1.000	0.001541	0.0005	308%	13:16
25	H2O	ICB	100	136.5	1		0.0048	-0.00003	1.000	ND	0.0002		:17
44	H2O	CRA	100	136.5	1		0.0224	0.00020	1.000	0.000203	0.0002	101%	:18
121	H2O	CRA	100	136.5	1		0.0390	0.00042	1.000	0.000418	0.0002	209%	:19
280	H2O	CCV	100	136.5	1		0.0840	0.00100	1.000	0.001002	0.001	100%	:20
260	H2O	CCB	50	136.5	1		0.0019	-0.00006	1.000	ND	0.0002		:21
156	H2O	PBW	50	136.5	1		0.0000	-0.00009	1.000	ND	0.0002		:22
12	H2O	LCS	50	136.5	1		0.0840	0.00100	1.000	0.001002	0.001	100%	:23
292	H2O	53616-01	100	136.5	1		0.0024	-0.00006	1.000	ND	0.0002		:24
59	H2O	53616-01DU	100	136.5	1		0.0000	-0.00009	1.000	ND	0.0002		:25
60	H2O	53616-01MS	100	136.5	1		0.0801	0.00095	1.000	0.000952	0.0002		:26
295	H2O	53616-02	100	136.5	1		0.0009	-0.00008	1.000	ND	0.0002		:27
341	H2O	53616-03	100	136.5	1		0.0019	-0.00006	1.000	ND	0.0002		:28
30	H2O	CCV	100	136.5	1		0.0801	0.00095	1.000	0.000952	0.001	95%	:29
263	H2O	CCB	100	136.5	1		0.0000	-0.00009	1.000	ND	0.0002		:30
159	H2O	PBW	100	136.5	1		0.0000	-0.00009	1.000	ND	0.0002		:31
167	H2O	LCS	100	136.5	1		0.0849	0.00101	1.000	0.001014	0.001	101%	:32
84	H2O	53653-01	100	136.5	1		0.0000	-0.00009	1.000	ND	0.0002		:33
107	H2O	53636-01DU	100	136.5	1		0.0000	-0.00009	1.000	ND	0.0002		:34
118	H2O	53653-01MS	100	136.5	1		0.0849	0.00101	1.000	0.001014	0.0002		:35
300	H2O	CCV	100	136.5	1		0.0801	0.00095	1.000	0.000952	0.001	95%	:36
329	H2O	CCB	100	136.5	1		0.0000	-0.00009	1.000	ND	0.0002		:37
41	H2O	BF1	100	136.5	1		0.0000	-0.00009	1.000	ND	0.0002		:38
384	H2O	DCS	100	136.5	1		0.0859	0.00103	1.000	0.001027	0.001	103%	:39
358	H2O	DCS	100	136.5	1		0.0840	0.00100	1.000	0.001002	0.001	100%	:40
383	H2O	53666-01	100	136.5	1		0.0000	-0.00009	1.000	ND	0.0002		:41
8	H2O	53668-01	100	136.5	1		0.0000	-0.00009	1.000	ND	0.0002		:42
302	H2O	53668-01MS	100	136.5	1		0.0493	0.00055	1.000	0.000552	0.0002		:43
129	H2O	53668-01SD	100	136.5	1		0.0488	0.00055	1.000	0.000546	0.0002		:44
326	H2O	CCV	100	136.5	1		0.0781	0.00093	1.000	0.000926	0.001	93%	:45
359	H2O	CCB	100	136.5	1		0.0000	-0.00009	1.000	ND	0.0002		:46
	H2O		100	136.5									
	H2O		100	136.5									

Additional Comments/Anomalies: _____

Hgbench.ALS
Revision 0
2/12/97

QUANTERRA ENVIRONMENTAL SERVICES, INC.

Industrial Mercury Analysis Raw Data Checklist

Revision 0 -March 25, 1997

Analyst's Checklist

		yes	no	n/a
1.	Were the special instructions for prep and/or analysis followed?	☐	☐	☐
2.	Is the correlation coefficient ≥ 0.995?	☐	☐	☐
3.	Is the blank less than 2 times the reporting limit or properly anomalized?	☐	☐	☐
4.	Are the DCSs within limits or properly anomalized?	☐	☐	☐
5.	Is the ICV and all CCVs within limits?	☐	☐	☐
6.	Are all CCBs within ± one reporting limit from zero?	☐	☐	☐
7.	Were the CCVs and CCBs run with only 10 samples between each set?	☐	☐	☐
8.	Are the reporting limits correct and reflect any dilutions?	☐	☐	☐
9.	Are the number of significant figures correctly reported?	☐	☐	☐
10.	Are the benchsheets complete (including calibration and standard verification #'s)?	☐	☐	☐
11.	Are all comments, footnotes, and anomalies properly documented?	☐	☐	☐
12.	Are holding time violation and rerun forms completed and attached?	☐	☐	☐
13.	Has all sample data been entered into LIMS?	☐	☐	☐
14.	Has all DCS and/or LCS Prep Blank data been entered into LIMS?	☐	☐	☐
15.	Has the data entered into LIMS been checked for errors?	☐	☐	☐
16.	For TCLP results, is the sample data within 20% of Regulatory Level (0.2 mg/L)?	☐	☐	☐

Analyst's Name:_____ Date:_____

Data Review's Checklist

		yes	no	n/a
1.	Have the calculations been checked?	☐	☐	☐
2.	Is the correlation coefficient ≥ 0.995?	☐	☐	☐
3.	Is all the QC data within the control limits and/or properly anomalized?	☐	☐	☐
4.	Are all the significant figures and reporting limits correct?	☐	☐	☐
5.	Have any comments, footnotes, and anomalies been properly documented?	☐	☐	☐
6.	Have any data errors been documented and entered into LIMS?	☐	☐	☐
7.	Has the raw data been copied and filed?	☐	☐	☐
8.	Has the data package been copied and filed?	☐	☐	☐
9.	If TCLP result ≤ 20% of Reg. Level and matrix spike < 50%, was MSA performed?	☐	☐	☐

Reviewed by:_____ Date:_____

Comments:_____

_____

_____

Anomalies:_____

_____

_____

APPENDIX C

MSA GUIDANCE

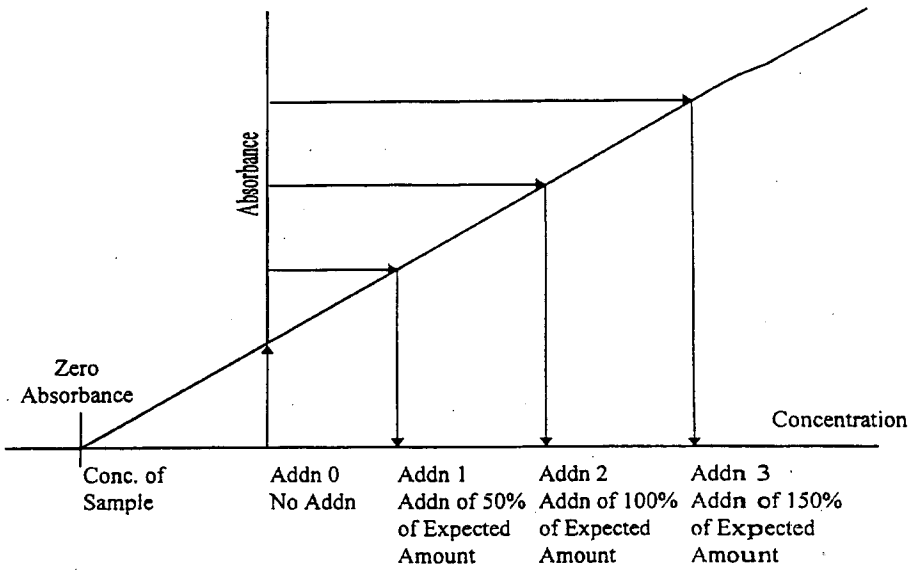
APPENDIX C. MSA GUIDANCE

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 pre-warmed 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.	

STL STANDARD OPERATING PROCEDURE

TITLE: CYANIDE PREPARATION METHOD

(SUPERSEDES: REVISION 8, DATED 02/04/99)

Prepared by:	Laurel E. Wood	3/20/00 Date
Reviewed by:	Deborah N Marcum Technology Specialist	3/20/00 Date
Approved by:	Quality Assurance Manager	3/21/00 Date
Approved by:	Environmental Health and Safety Coordinator	3-22-00 Date
Approved by:	Laboratory Director	3-22-00 Date

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17. MISCELLANEOUS (TABLES, APPENDICES, ETC...) 15

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 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. 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 7.2.8.)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:

$$\textit{Cyanide, mg/L} = \frac{\textit{(A) (1000)}}{\textit{mL cyanide solution (10)}}$$

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, MS, MSD, 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 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 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 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.

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. Summary

11.1.1. 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.

11.2. Sample Preparation Procedure applicable for use with the Midi distillation unit.

11.2.1. Checking for Interferences (For non-CLP samples)

11.2.1.1. 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.**

11.2.1.2.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.

11.2.2. 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.

11.2.3. Amenable Cyanide (Chlorinated Aliquot) [THIS PROCESS IS PERFORMED ONLY WHEN REQUESTED BY THE CLIENT]

11.2.3.1.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.

11.2.4. Total Cyanide

11.2.4.1. 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.

11.2.4.2.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.

11.2.4.3.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.

11.2.4.4.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.

11.2.4.5.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.

11.2.5. Free Cyanide (Weak and Dissociable) [THIS PROCESS IS PERFORMED ONLY WHEN REQUESTED BY THE CLIENT]

11.2.5.1.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.

11.2.5.2. 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.

11.2.5.3.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.

11.2.5.4. 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.

11.2.6. Cyanide Unit Clean-up

11.2.6.1. Cyanide distillation unit glassware is very fragile and expensive. It must be handled with care at all times.

11.2.6.2. 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.

11.2.6.3. 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.

11.2.6.4. Re-assemble the set-up.

11.2.6.5. Be sure to wash each set up as a separate unit and replace in the same position.

11.2.6.6. If a sample of known high cyanide concentration was distilled in a certain position, be sure to change the appropriate tubing on that position.

11.3. Analytical Documentation

11.3.6. 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.

11.3.7. 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.

11.3.8. 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.

11.3.9. 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.

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

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. STL® Quality Assurance Management Plan, current revision

16.2. Associated SOPs

16.2.1. 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			—	—	—	" 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	D8516 MS	T	C	F			—	—	—	" "
*10	D8516 MSD	T	C	F			—	—	—	" "
*1A	D8516	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	D83R1	T	C	F			✓	✓	✓	

leaches

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 I _II
_Calibration _QC
_Calculation _Complete
Date: 2-7-00 Sign: [signature]

Level Review _I X II
✓Calibration ✓QC
✓Calculation ✓Complete
Date: 2.8.00 Sign: [signature]

APPENDIX A

N:\WET-LOG\CYANIDE

QUANTERRA- NORTH CANTON
CYANIDE DISTILLATION LOGSHEET

PAGE 2 OF 2

Batch No. 0040131 water / 0040130 leaches
Prep/Anal. Date 000208 / 000209
Start Time: 2:30 pm

LCS&MS/MSD No. CY0117
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
1B 1A	D83R1e	(T) C F	50 ml	50 ml	✓	✓	✓	
2	D83R8	(T) C F	"	"	✓	✓	✓	
3	D85LeF	(T) C F	"	"	✓	✓	✓	
4	D85LeQ	(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 I _II
_Calibration _QC
_Calculation _Complete
Date: 2-8-00 Sign: M. Fuller

Level Review _I X II
✓Calibration ✓QC
✓Calculation ✓Complete
Date: 2-8-00 Sign: M. Fuller

Rev. 5-16-97

QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: CYANIDE AUTOMATED, PYRIDINE-BARBITURIC ACID METHOD

(SUPERSEDES: REVISION5, DATED 06/24/99)

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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), 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 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. Sulfides interfere, but can be eliminated by treating sodium hydroxide distillate 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 Quanterra 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: 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. 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. 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. 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.

9.2.3. 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 aqueous 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 samplesSOP NC-WC-0032 for distillation instruction.

9.3.3. The acceptance limits for drinking water samples is 90 – 110%.

9.3.4. Corrective Action for LCS

9.3.4.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.4.2. 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.

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.

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 ≥ 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 ± 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.

10.5. Normal Tray Protocol

10.5.1. P,5C>2, H@2, 2L@0, G@2, XS>7, H@2, 2L@0, G@2, E

NOTE: X = number of sample cups minus the six calibration cups.

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. 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. Non-CLP Samples: For samples containing sulfide, calibrate the instrument using the calibration curve specially prepared for samples containing sulfide.

11.3.2.2. 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. 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.

11.5. 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***

$$D = \text{Dilution Factor} = \frac{\text{Final Volume of Dilution}}{\text{Volume of Sample Distillate Used}}$$

NOTE: Free cyanide has the same calculations as Total cyanide

12.4. LCS Recovery:

$$\frac{\text{Instrument Value}}{0.04(\text{true})} \times 100 = \%\ \text{Recovery}$$

12.5. CCV Recovery:

$$\frac{\text{Instrument Value}}{0.025\ (\text{true})} \times 100 = \%\ \text{Recovery}$$

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:***

$$\frac{A - B}{0.040\ (\text{true})} \times 100 = \%\ \text{Recovery}$$

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.

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 Quanterra. 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. Quanterra® Quality Assurance Management Plan, current revision

16.2. Associated SOPs

16.2.1. 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 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.

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: SULFIDE

(SUPERSEDES: ENSECO SOP #LM-WALN-1330 (REVISION 1.0))

Prepared by: _____

Reviewed by: _____
Technology Specialist

Approved by: _____
Quality Assurance Manager

Approved by: _____
Environmental Health and Safety Coordinator

Approved by: _____
Laboratory Director

Proprietary Information Statement:

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of the concentration of Sulfide in waters, liquids, solids, and sludges. It is based on SW846 Method 9030A and Methods for Chemical Analysis of Water and Wastes (MCAWW) 376.1. The working range is 1 to 30 mg/L for waters and 50 to 1500 mg/kg for solids and sludges.

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.

2. SUMMARY OF METHOD

2.1. An excess of iodine is added to a sample which oxidizes the Sulfide to sulfur under acidic conditions. The excess iodine is back titrated with sodium thiosulfate.

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. Reducing substances such as thiosulfite, sulfites, and various organic compounds cause interferences, but treatment with zinc acetate solution will eliminate some of these interferences. (Use approximately 15 drops of 2 N zinc acetate per 500 mL of sample if not already preserved with it.)

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 (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 Aicd, 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 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. Volumetric pipets: various

6.2. Buret: 25 mL Class A

6.3. Erlenmeyer flasks: 500 mL

6.4. Graduated cylinder: 250 mL

6.5. Top loading balance: capable of accurately weighing ± 0.01 g

6.6. Volumetric flasks: various

6.7. Vacuum pump, filter and flask

6.8. Whatman 934-AH filters

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. (1:1) Hydrochloric Acid: Add 250 mL concentrated hydrochloric acid (HCl) to 250 mL of reagent water.

7.1.2. Starch Indicator: Add 10 mL of reagent water to 5 g starch (potato) and mix. Add starch mixture to 500 mL of boiling reagent water. Mix, cool, and store in a well-labeled squirt bottle. Alternately, use purchased starch solution.

7.1.3. 0.025 N Sodium Thiosulfate: Add 0.4 g NaOH and 6.205 g of sodium thiosulfate ($Na_2S_2O_3 \bullet 5H_2O$) to 500 mL of reagent water in a 1 liter volumetric. Dilute to volume with reagent water. Store in a dark container. Also available commercially.

7.1.3.1. Standardization of 0.025 N Sodium Thiosulfate Solution: To make 0.025N Biodate Solution, dissolve 0.462 g $KH(IO_3)_2$ in 500 mL with reagent water. Weigh 2 g KI in a 500 mL Erlenmeyer flask. Add 100 to 150 mL reagent water, 5 drops H_2SO_4 and 20 mL biodate solution using a volumetric pipet. Dilute to 200 mL with reagent water. Titrate with Sodium Thiosulfate. When a pale straw yellow color is reached, add 1-2 mL starch. Continue titrating from a blue to a clear end point.

Calculation

$$Na_xS_2O_3\ Normality = \frac{(a)\,(b)}{c}$$

a = ***mLs Biodate (20 mL)***

b = ***Normality Biodate (0.025N)***

c = ***mLs of $Na_2S_2O_3$ used to titrate***

Repeat two more times

7.1.4. 0.025 N Iodine Solution: Add 20 g KI (potassium iodide) and 3.2 g iodine to a 1 liter volumetric flask. Add 500 - 700 mL of reagent water and dissolve. Dilute to volume with reagent water. Store in a dark container. Also available commercially.

7.1.4.1. Standardization 0.025 N Iodine Solution: Perform three method blanks. Refer to method blank section in SOP.

Calculation

$$Normality\ Iodine = \frac{(Normality\ Na_2S_2O_3)(mL\ of\ titrant\ Na_2S_2O_3)}{20\ mL\ Iodine}$$

7.1.5. 2N Zinc Acetate: Dissolve 220 g of zinc acetate in 870 mL of reagent water and dilute to 1 liter with reagent water.

7.2. Standards

7.2.1. Laboratory Control Sample

7.2.1.1. 100 mg/L Sulfide: Add 0.35 g of sodium sulfide to 500 mL of reagent water in a 1 liter volumetric flask. Dilute to volume with reagent water. Store at 4°C ± 2°C. Stable for one week.

7.2.2. Matrix Spike Standard

7.2.2.1. Prepare a midrange matrix spike standard as described in 7.2.1 for use as a MS/MSD.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Waters are preserved to a pH > 9 with NaOH and zinc acetate. Non-water samples are unpreserved. All matrices are stored at 4°C ± 2°C in plastic or glass containers.

8.2. The holding time is seven 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, 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. A reagent water blank consisting of 250 mL of reagent water must be analyzed 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 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 midrange LCS consisting of 10.0 L of 100 mg/L sulfide standard and 240 mL of reagent water must be 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. 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 5 g or 250 mL of the sample and 50 mL of the 100 mg/L sulfide standard should be analyzed with every 20 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.

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. Sample Preparation

11.3.1. Water Samples

11.3.1.1. Using the vacuum apparatus, filter 250 mL (or a lesser aliquot) of well homogenized sample. Discard the filtrate and analyze the pad and solids following section 11.4. Record volume filtered on the analytical logsheet.

11.3.2. Non-Water Samples

11.3.2.1. No preparation necessary.

11.4. Sample Analysis

11.4.1. Summary

11.4.1.1. Excess iodine is added to a sample and under acidic conditions is back titrated with sodium thiosulfate from a blue to a clear color.

11.4.2. Sample Analysis Procedure

11.4.2.1. Place the pad from the filtered water sample (section 11.3.1) or 5 g of non-water sample in a 500 mL Erlenmeyer flask. At this time, add any spiking solutions if necessary. Add 20.0 mL .025 Iodine solution and 1-2 mL 1:1 HCl solution (watch for fumes). Add 250 mL reagent water and mix. Add 1 squirt (1-2 mL) of starch indicator and mix. Titrate from blue to clear with .025 N sodium thiosulfate titrant. Record the amount of titrant used on the analytical logsheet.

NOTE: Some matrices may be turbid or colored and the color change from blue to clear may not be easily seen. In this case, look for a shade change.

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. Calculations

$$\textit{Sulfide, mg / L or mg / kg} = \frac{[(A \times B) - (C \times D)] \times 16{,}000}{\textit{mL or g of sample used}}$$

Where:

A = *mL of iodine solution*

B = *Normality of iodine solution*

C = *mL of sodium thiosulfate titrant*

D = *Normality of sodium thiosulfate titrant*

NOTE: If the normality of both the iodine solution and sodium thiosulfate solution equal 0.025, the following calculation may be used

$$\textit{Sulfide, mg / L or mg / kg} = \frac{(20 - \textit{mL titrant}) \times 400}{\textit{mL or g of sample used}}$$

12.1.1.

$$\textit{LCS \% Recovery} = \frac{\textit{mg / L (from 12.1.1)}}{\textit{4.0 (true)}} \times 100$$

12.1.2.

$$\textit{MS / MSD \% Recovery} = \frac{A - B}{\textit{5000 (true)}} \times 100$$

Where:

A = *(20 - mL titrant for MS/MSD) x 400*

B = *Concentration from 12.1.1 x mL or g of sample used*

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. All aqueous sample preparations can be rinsed down the drain with copious amounts of water.

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 Quanterra. 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 of Evaluating Solid Waste, Third Edition, Sulfide, Method 9030A, Update I, September 1994.

16.1.2. EPA 600, Methods for Chemical Analysis of Waters and Wastes, Sulfide (Titrimetric, Iodine), Method 376.1

16.1.3. Standard Methods Eighteenth Edition, Sulfide Method 4500-S^2-E

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Reporting limits

17.1.1. The lower reporting limits (RL) are 1 mg/L for waters and 50 mg/kg for solids.

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. Samples that contain strong oxidizers or reducers will interfere with this method.

17.2.2. Samples that are not water miscible (oils, various solvents) cannot be analyzed using this method.

17.2.3. The iodine should turn a yellow-orange color when added to the sample after the addition of the reagent water. If it does not, the sample may be high in sulfide and less sample should be used.

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Richland-Operation Specific Standard Operating Procedure

TITLE: Determination of Hexavalent Chromium (CrVI) in Solid Matrices with Alkaline Digestion by Method 3060A and Colorimetric Determination by Method 7196A

(SUPERSEDES: RICHWC5005 Revision 3)

Prepared by: _____

Reviewed by: _____
Technical Specialist

Approved by: _____
Quality Assurance Manager

Approved by: _____
Environmental Health and Safety Coordinator

Approved by: _____
Laboratory Director

Proprietary Information Statement:

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1. SCOPE AND APPLICATION

1.1. Method 3060A is an alkaline digestion procedure for extracting hexavalent chromium [Cr(VI)] from soluble, adsorbed, and precipitated forms of chromium compounds in soils, sludges, sediments, and some industrial waste materials. To quantify total Cr(VI) in a solid matrix, three criteria must be satisfied: (a) the extracting solution must solubilize all forms of Cr(VI), (b) the conditions of the extraction must not induce reduction of native Cr(VI) to Cr(III), and (c) the method must not cause oxidation of native Cr(III) contained in the sample to Cr(VI). Method 3060A meets these criteria for a wide spectrum of solid matrices. Under the alkaline conditions of the extraction, minimal reduction of Cr(VI) or oxidation of native Cr(III) occurs. The addition of Mg^{2+} in a phosphate buffer to the alkaline solution has been shown to suppress oxidation if observed. The accuracy of the extraction procedure is assessed using spike recovery data for soluble and insoluble forms of Cr(VI) (e.g., $K_2Cr_2O_7$ and $PbCrO_4$). Recovery of an insoluble Cr(VI) spike can be used to assess the first two criteria listed above, and method-induced oxidation is minimal except in soils high in Mn and amended with soluble Cr(III) salts or freshly precipitated $Cr(OH)_3$.

1.2. The quantification of Cr(VI) shall be performed using SW-846 Method 7196A (colorimetric).

2. SUMMARY OF METHOD

2.1. This method uses an alkaline digestion to solublize both water-insoluble and water-soluble Cr(VI) compounds in solid waste samples. The pH of the digestate must be carefully adjusted during the digestion procedure. Failure to meet the pH specifications will necessitate redigestion of the samples.

2.2. The sample is digested using 0.28M Na_2CO_3/0.5M NaOH solution and heating at 90 - 95°C for 60 minutes to dissolve the Cr(VI) and stabilize it against reduction to Cr(III).

2.3. After filtration, the pH of the filtrate is adjusted to 7 and the colorimetric determination of Cr(VI) with the diphenylcarbazide complex at 540 nm is completed.

3. DEFINITIONS

3.1. None.

4. INTERFERENCES

4.1. When analyzing a sample digestate for total Cr(VI), it is appropriate to determine the reducing/oxidizing tendency of each sample matrix. This can be accomplished by characterization of each sample for additional analytical parameters, such as pH, ferrous iron, sulfides, and oxidation reduction potential. Other indirect indicators of reducing/oxidizing tendency include Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), and Biochemical Oxygen Demand (BOD). Analysis of these additional parameters establishes the tendency of Cr(VI) to exist in the unspiked sample(s) and assists in the interpretation of QC data for matrix spike recoveries outside conventionally accepted criteria.

4.2. Certain substances, not typically found in the alkaline digests of soils, any interfere in the analytical methods for Cr(VI) following alkaline extraction if the concentration of these interfeneces are high and the Cr(VI) concentration is low.

4.3. For waste materials suspected of containing soluble Cr(III) concentrations greater than four times the laboratory Cr(VI) reporting limit, Cr(VI) result obtained using alkaline digestion may be biased high due to method induced oxidation. The addition of Mg^{2+}, in a phosphate buffer, to the alkaline extraction solution has been shown to suppress oxidation and is added if oxidation is observed or suspected. The presence of soluble Cr(III) can be approximated by extracting sample with reagent water and analyzing the resultant digestate for both Cr(VI) and total Cr. The difference between the two values approximates soluble Cr(III).

4.4. One of the most insoluble forms of chromate in alkaline solution, barium chromate, may require additional heating time to effect complete dissolution in some soil matrices.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra 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:

5.1.2.1. The following materials are known to be **corrosive**: Nitric acid, sodium hydroxide and sulfuric acid.

5.1.2.2. The following materials are known to be an **oxidizer**: Nitric acid and potassium dichromate.

5.1.2.3. The following material is known to be **flammable**: Acetone.

5.1.2.4. The following materials are known be **carcinogens**: Diphenylcarbazide, lead chromate and potassium dichromate.

6. EQUIPMENT AND SUPPLIES

6.1. Aluminum dishes - various sizes.

6.2. Balance - capable of weighing 0.1 mg.

6.3. Beakers - glass, various sizes.

6.4. Centrifuge.

6.5. Centrifuge tubes - plastic, with caps.

6.6. Filtration Apparatus with filters of 0.45 µm pore size.

6.7. Graduated cylinders - Class A, various sizes.

6.8. Hot plate with stirrer and Teflon stir bars.

6.9. Oven - capable of 105 ± 2°C or moisture content analyzer (Mettler LP16M or equivalent).

6.10. pH paper or pH meter (calibrated) with buffer solutions.

6.11. Pipet bulb.

6.12. Pipettors - air-displacement type, (critical volume) with disposable tips.

6.13. Polystyrene graduated pipet - 5 mL volume with accuracy of 99% or better.

6.14. Spectrophotometer - for use at 540 nm.

6.15. Spectrophotometer cells - 5-cm and 1-cm path length.

6.16. Volumetric flasks - Class A, 1000, 200, 100 and 50 mL.

6.17. Watch glass covers.

7. REAGENTS AND STANDARDS

7.1. Reagents are prepared from analytical reagent grade chemicals unless otherwise specified below. Reagent water, which must have an electrical resistivity of 16.7 megohm-cm or greater when obtained, is used throughout. Reagent water is obtained from the Nano-poure system. Label all reagents as outlined in procedure RICHQA5002.

NOTE: Consult the Material Safety Data Sheets for the properties of these reagents and how to work with them.

NOTE: Reagents and standards may be prepared in volumes other than those specified providing the relative amounts of ingredients remain constant.

7.2. Acetone - WARNING: Flammable.

7.3. Digestion Reagent: Dissolve 20.0 g of sodium hydroxide and 30.0 g of sodium carbonate in about 800 mL of reagent water in a 1-L volumetric flask. Dilute to the mark with reagent water and mix well. Store in a tightly capped polyethylene bottle at 20 - 25°C and discard after 1 month. The pH of the digestion reagent must be checked before using. The pH must be 11.5 or greater.

7.4. 1,5-diphenylcarbazide. NOTE: Light sensitive. WARNING: Carcinogen.

7.5. Diphenylcarbazide solution - Dissolve 250 mg 1,5-diphenlycarbazide in 50 mL acetone. Store in a brown bottle. Prepare the day of use.

7.6. Lead chromate ($PbCrO_4$) - The insoluble matrix spike is prepared by adding 10 - 20 mg $PbCrO_4$ to a separate sample aliquot. Store under dry conditions in a tightly sealed container. WARNING: Carcinogen.

7.7. Magnesium chloride ($MgCl_2$) - anhydrous, 392.2 mg is equivalent to 100 mg Mg^{2+}. Store in a tightly sealed container.

7.8. 16M Nitric acid (16M HNO_3) - Concentrated. WARNING: Corrosive and oxidizer.

7.9. Potassium dichromate ($K_2Cr_2O_7$) - WARNING: Carcinogen and oxidizer.

7.10. Potassium dichromate stock solution (1000 mg/L Cr(VI)): Dissolve 0.2829 g of dried (105°C) potassium dichromate, $K_2Cr_2O_7$ (analytical reagent grade), in reagent water in a 100 mL volumetric flask and dilute to volume with reagent water. Mix well.

NOTE: A 1000 mg/L Cr(VI) commercial standard may be used.

7.11. Potassium dichromate standard solution (Spike Solution) (50 mg/L Cr(VI)) - Dilute 5.00 mL potassium dichromate stock solution to 100 mL with reagent water and mix well. Prepare daily.

7.12. Phosphate buffer (0.5M K_2HPO_4/0.5M KH_2PO_4 buffer) at pH 7 - Dissolve 87.09 g K_2HPO_4 and 68.04 g KH_2PO_4 in approximately 700 mL of reagent water in a 1 L volumetric flask. Dilute to volume with reagent water and mix well.

7.13. Sodium Carbonate, Na_2CO_3, anhydrous. Store at 20 - 25°C in a tightly sealed container.

7.14. Sodium hydroxide (NaOH) - pellets -. WARNING: Corrosive.

7.15. 18M Sulfuric acid (18M H_2SO_4) - Concentrated. WARNING: Corrosive.

7.16. Sulfuric acid, 10% (v/v) - Add 10 mL concentrated sulfuric acid into about 80 mL of reagent water. Dilute to 100 mL with reagent water. CAUTION: Slowly add H_2SO_4 to water with stirring and cooling to avoid splattering.

8. SAMPLE COLLECTION, PRESERVATIVES AND STORAGE

8.1. The sample may be collected in glass or plastic containers using no preservatives. Storage of the sample prior to analysis should be at 4 ± 2°C and not exceed one month from sample collection.

8.2. Sample digestates shall be analyzed within 96 hours after extraction.

9. QUALITY CONTROL

9.1. All quality control data shall be maintained and available for easy reference.

9.2. Dilute samples and matrix spikes if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve. Dilutions prior to the colorimetric step use reagent water as diluent. Dilutions done after the colorimetric step use the calibration blank as diluent.

9.3. Employ a minimum of one preparation blank, one laboratory control sample (LCS), and one duplicate per sample batch.

9.4. Verify calibration with an independently prepared check standard before running sample, after every 10 (or fewer) samples and at the end of an analytical run. Verification standard recoveries must be 100 ± 10% or the verification standard should be reprepared and reanalyzed. If the verification standard recovery is still out of limits, the batch must be reprepared and reanalyzed.

9.5. Preparation blanks, LCS, duplicates, and matrix spikes are brought through the whole sample preparation and analytical process. Preparation blank results must be less than the MDL or the batch should be reprepared and reanalyzed.

9.6. Duplicate samples must have a Relative Percent Difference (RPD) of ≤ 20%, if both the original and the duplicate are ≥ four times the MDL. A control limit of ± the MDL is used when either the original or the duplicate sample is < four times the MDL.

9.7. Both soluble and insoluble pre-digestion matrix spikes must be analyzed at a frequency of one per batch of ≤ 20 samples. The soluble matrix spike should be spiked with 2.0 mL of the spiking solution (40 mg/kg Cr(VI)) or at twice the sample concentration, whichever is greater. The insoluble matrix spike is prepared by adding 10 - 20 mg of $PbCrO_4$ to a separate sample aliquot, and is used to evaluate the dissolution during the digestion process. Both matrix spikes are then carried through the digestion process. More frequent matrix spikes must be analyzed if the soil characteristics within the analytical batch appear to have significant variability based on visual observation. The recovery limits for matrix spikes are 75 - 125%. If the matrix spike recoveries are not within these limits, the entire batch must be redigested/reanalyzed. If upon reanalysis, the matrix spike is not within the recovery limits, but the LCS is within the recovery limits of 80 - 120%, Cr(VI) data may be valid for use despite the perceived "QC failure".

9.7.1. Pre-digestion matrix spike recoveries for Cr(VI) less than the minimum recovery criterion (75%), are indicative of highly reducing samples. If a low or zero percent pre-digestion matrix spike recovery is obtained, when requested by the client, the following alternate approach can be used to determine the potential contribution of the sample matrix to Cr(VI) reduction. This approach consists of performing a mass balance, whereby total chromium (Method 3050A) is analyzed for two samples: (1) a separate unspiked aliquot of the sample previously used for spiking, and (2) the digested solids remaining after the alkaline digestion and filtration of the matrix spike The difference between the total chromium measurements should be approximately equal to the amount of the spike added to the matrix spike. If the LCS recovery is within the recovery limits and the Cr(VI) spike is accounted for in the filtered solids as total chromium, it is likely that the reduction of the Cr(VI) to insoluble Cr(III)

resulted from the reducing matrix of the original sample subjected to Cr(VI) spiking.

9.7.2. When low matrix spike recoveries are obtained, a post-digestion Cr(VI) matrix spike must be analyzed per batch. The post-digestion matrix spike concentration should be equivalent to 40 mg/kg (1 mg/L for 2.5 g sample size) or twice the sample concentration observed in the unspiked aliquot of the test sample, whichever is greater.

9.7.2.1. Dilute the sample aliquot to a minimum extent, if necessary, so that the absorbance reading for both the unspiked sample aliquot and spiked aliquot are within the initial calibration curve.

9.7.2.2. A guideline for the post-digestion matrix spike recovery is 85-115% recovery. If not achieved, consider the corrective actions/guidance on data. These digestates may contain soluble reducing agents for Cr(VI), such as fulvic acids.

10. CALIBRATION

10.1. Spectrophotometer calibration shall be done after sample digestion has been completed.

10.2. To compensate for possible slight losses of chromium during digestion or other operations of the analysis, treat the chromium standards by the same colorimetric preparation procedure as the sample. Accordingly, pipet aliquots of a chromium standard solution to prepare calibration standard concentrations ranging from 0.0 to 0.5 mg/L (if using a 5-cm absorption cell) or 0.0 to 1.5 mg/L (if using a 1-cm absorption cell) Cr(VI) when diluted to the appropriate volume. Mix well.

10.3. Transfer 95 mL of each calibration standard solution to a 100 mL volumetric flask. For the calibration blank, transfer 180 mL of reagent water to a 200 mL volumetric flask. Add 2 mL of diphenylcarbazide solution (4 mL for the calibration blank) to each standard flask and mix. Adjust the pH of each standard to 2 ± 0.5 with 10% H_2SO_4 and dilute to volume with reagent water. Mix well. Wait 10 minutes for full color development.

NOTE: Lower room temperatures may extend the time needed for color development.

10.4. Transfer a suitable portion of each colored solution to a 1-cm or a 5-cm absorption cell and measure the absorbance at 540 nm. As reference, use the calibration blank. Determine the equation for the calibration curve relating absorbance to mg/L of Cr(VI). (The spectrophotometer may have the capability to calculate the equation for the calibration curve or it may be done manually.)

10.5. A calibration curve shall be generated prior to the analysis of samples each day of analysis.

11. PROCEDURE

NOTE: If any parameter is found to be out of limits a Nonconformance Memo will be issued to the Quality Assurance Group.

NOTE: One time procedural variations are allowed if deemed necessary by the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size or other parameters. Any variation in procedure shall require approval by supervision and immediate notification of the Quality Assurance Group. If contractually required, the client shall be notified prior to any procedure changes. A Nonconformance Memo shall be completed and forwarded to the Quality Assurance Group within one day of the supervisor's approval. The Nonconformance Memo will be filed in the project file.

11.1. % Solid Determination

11.1.1. Weigh out a suitable aliquot of well mixed sample into a pre-dried, tared aluminum dish. If a drying oven is to be used, weigh approximately 5 g of sample into the dish. If a moisture content analyzer is to be used, follow the manufacturer's recommendation regarding sample size. Record the tare weight of the dish and the sample size on the appropriate analytical worksheet.

11.1.2. If a drying oven is used, place the dish in an oven at 105 ± 2°C for 12 to 24 hours. Remove from oven and cool in a dessicator. Weigh the cooled dish and record the weight on the appropriate analytical worksheet.

11.1.3. If a moisture content analyzer is used, follow the manufacturer's instructions. Record information on the appropriate analytical worksheet.

11.2. Sample Digestion

11.2.1. Adjust the temperature of the hot plate to be used in the alkaline digestion by preparing and monitoring a temperature blank (a 150 mL beaker containing approximately 50 mL of digestion solution). Maintain a temperature of 90 - 95°C.

11.2.2. Mix the sample and transfer a 2.5 ± 0.10 g of the sample into a clean labeled beaker (150 mL recommended).

11.2.3. Add 50 ± 1.0 mL of digestion solution to each sample. Add 392.2 ± 0.2 mg of $MgCl_2$ and 0.5 mL of 1.0M phosphate buffer. Cover the sample with a watch glass. The Mg^{2+} is used to suppress oxidation of certain forms of Cr(III) (such as soluble forms) that can be oxidized to Cr(VI) during the procedure.

11.2.4. Stir the samples continuously (unheated) for at least 5 minutes using a stirring bar.

11.2.5. Heat the samples and maintain a temperature range of 90 - 95°C with constant stirring for at least 60 minutes after the correct temperature range has been reached. Record the digestion on and digestion off time.

11.2.6. Remove the sample from the hot plate and allow to cool to room temperature. Quantitatively transfer the sample to the filtration apparatus using reagent water to rinse the beaker and add the rinses to the filtration apparatus. Filter the sample through a 0.45 µm filter. Rinse the inside of the filter holder and filter pad with reagent water.

NOTE: The sample digestate may be centrifuged prior to filtration in order to facilitate filtration. Quantitatively transfer the sample to a clean labeled centrifuge tube and centrifuge. Filter the supernate. Wash any remaining solids in the centrifuge tube with reagent water, centrifuge and filter the wash supernate.

NOTE: The remaining solids after filtration of the matrix spike should be saved for possible use in assessing low Cr(VI) matrix spike recoveries. If the matrix spike was centrifuged, place the filter containing solids in the centrifuge tube containing the matrix spike solids. Store the filtered solids at 4 ± 2°C. See section 9.7.1 for additional details.

11.2.7. While stirring, slowly add concentrated nitric acid solution to the sample vessel dropwise. Monitor the pH with either pH paper or a pH meter. Adjust the pH of the solution to 7.5 ± 0.5. If the pH of the digest should drop below 7.0, discard the solution and redigest. If overshooting the pH of 7.5 ± 0.5 occurs repeatedly, prepare a diluted nitric acid solution and repeat the digestion (11.2).

CAUTION: Carbon dioxide will evolve from the sample.

11.2.8. Quantitatively transfer the contents of the sample vessel to a 100 mL volumetric flask using reagent water. Dilute to volume with reagent water and mix well.

11.3. Colorimetric Determination

11.3.1. Transfer 95 mL of solution from 11.2.8 to a 100 mL volumetric flask. Add 2.0 mL diphenylcarbazide solution and mix. Add 10% H_2SO_4 to give a pH of 2 ± 0.5, dilute to 100 mL with reagent water, mix, and let stand 10 minutes for full color development. Transfer an appropriate portion of the solution to either a 1-cm or 5-cm absorption cell and measure its absorbance at 540 nm. Use the calibration blank as a reference.

NOTE: If the solution is turbid or colored after dilution to 100 mL in step 11.2.8, use the remaining 5 mL to obtain an absorbance reading for the color blank and correct the result of the final colored solution by subtracting the measured result for the color blank.

12. DATA ANALYSIS AND CALCULATIONS

12.1. % Solids:

$$\% \text{ Solids} = \frac{B}{A} \times 100$$

where:

A = initial weight of moist soil in grams.

B = final weight of dried soil in grams.

12.2. Sample concentration:

$$\text{Concentration in ng/kg} = \frac{A \times D \times E}{B \times C}$$

where:

A = Concentration observed in the digest (mg/L).

B = Initial moist sample weight (kg).

C = % solids/100.

D = Dilution factor.

E = Final digest volume (L).

12.3. Relative Percent Difference (RPD)

$$RPD = \frac{(S - D)}{[(S + D) / 2]} \times 100$$

where:

S = Initial sample result.

D = Duplicate sample result.

12.4. Spike Recovery

$$\text{Percent recovery} = \frac{(SSR - SR)}{SA} \times 100$$

where:

SSR = Spike sample result.

SR = Sample (unspiked) result.

SA = Spike added.

13. METHOD PERFORMANCE

13.1. 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. All training shall be conducted and documented in accordance with RICHQA0022.

14. POLLUTION PREVENTION

14.1. None.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure will be segregated, and disposed according to the facility hazardous waste procedures. The Health and Safety Coordinator should be contacted if additional information is required.

16. REFERENCES

16.1. SW-846, Method 3050A, Revision 1, July 1992, Acid Digestion of Sediments, Sludges and Soils.

16.2. Quanterra, Quality Management Program, latest revision.

16.3. Quanterra, Richland Site-Specific Quality Assurance Management Plan, latest revision.

16.4. Quanterra, Richland Site-Specific Quality Assurance Summary, latest revision.

16.5. Associated SOPs

16.5.1. RICHQA0022 - Training.

16.5.2. RICHQA5002 - Chemical/Reagent Labeling.

17. MISCELLANEOUS

17.1. Method Differences

17.1.1. In case of low spike recoveries, a total chromium analysis on the solids from the matrix spike and on an unspiked sample aliquot will only be done if requested by the client.

17.2. Responsibilities

Analyst: Implements SOP as written.

Peer Reviewer/Team Leader: Performs calculation and first level of data checking 100% of all data.

Group/Team Leader: Performs final data review checking 20% of all data. Schedules analysts.

Project Manager: Confirms final review and prepares data for reporting to client.

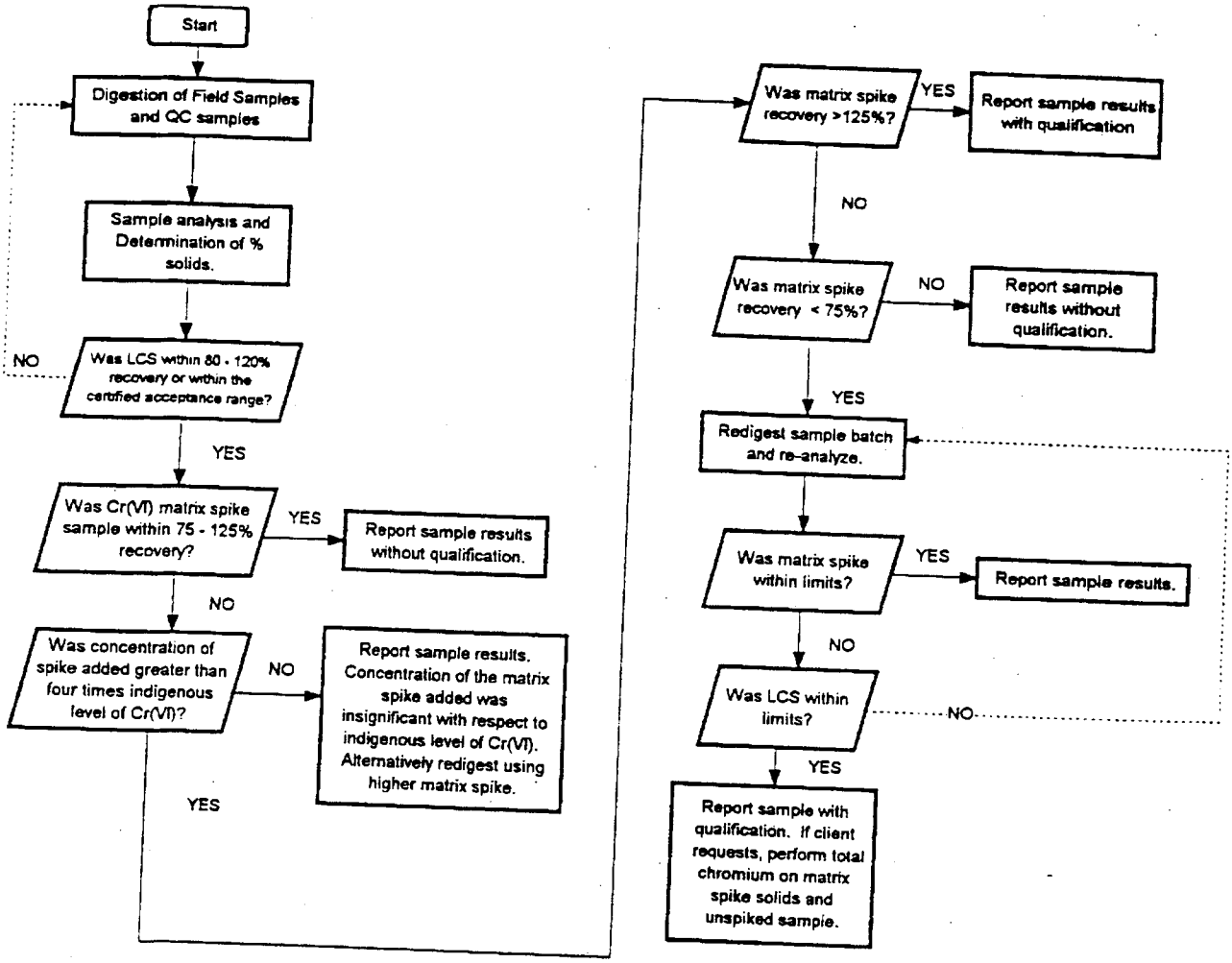
QA Manager: Performs product quality assessments as defined in the facilities Quality Assurance procedures.

17.3. Records Management/Documentation

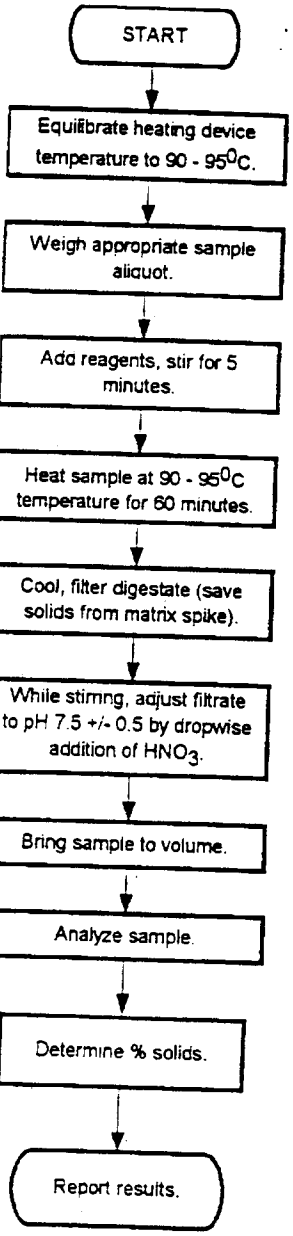
17.3.1. All records generated by this analysis will be filed and kept in accordance with Quanterra SOPs for records management and maintenance.

17.4. Procedural Flow Chart

QUALITY CONTROL FLOW CHART



ALKALINE DIGESTION AND SAMPLE ANALYSIS



QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: CHROMIUM, HEXAVALENT (COLORIMETRIC)

(SUPERSEDES: REVISION 1.1, DATED 06/03/97)

Prepared by:	Patrick O'Meara	3/9/99 Date
Reviewed by:	Technology Specialist	3/9/99 Date
Approved by:	Quality Assurance Manager	3/10/99 Date
Approved by:	Environmental Health and Safety Coordinator	3-12-99 Date
Approved by:	Laboratory Director	3-21-99 Date

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1. SCOPE AND APPLICATION

1.1. This method is used to determine the concentration of dissolved Hexavalent Chromium (Cr[VI]) in Extraction Procedure (EP) toxicity characteristic extracts, groundwaters, waters and solids. This method may also be applicable to certain domestic and industrial wastes, provided that no interfering substances are present. It is based on SW846 Methods 7196A, and Standard Methods 3500-CR. The working linear range is 0.02 - 0.5 mg/L for waters and 1 - 25 mg/kg for solids.

1.2. The associated method codes are EA (7196A) and H4 (3500-CR).

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. Dissolved Hexavalent Chromium, in the absence of interfering amounts of substances such as molybdenum, vanadium, and mercury, is determined colorimetrically by reaction with diphenylcarbazide in acid solution and measuring the resulting red-violet color at 540 nm.

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. The Chromium reaction with diphenylcarbazide is usually free from interferences. However, certain substances may interfere if the Chromium concentration is relatively low. Hexavalent molybdenum and mercury salts also react to form color with the reagent; however, the red-violet intensities produced are much lower than those for Chromium at the specified pH. Concentrations of up to 200 mg/L of molybdenum and mercury can be tolerated. Vanadium interferes strongly, but concentrations up to ten times that of Chromium will not cause trouble.

4.3. Iron in concentrations greater than 1 mg/L may produce a yellow color, but the ferric iron color is not strong and no difficulty is encountered normally if the absorbance is measured photometrically at the appropriate wavelength.

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 (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. Chemicals known to be **flammable** are: **Acetone**

5.3.2. The following materials are known to be **corrosive: 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. Spectrophotometer for use at 540 nm

6.2. 1 cm Spectrophotometer Cell

6.3. Hot plate

6.4. Beakers: glass, plastic

6.5. Volumetric flasks: various

6.6. Pipettes: various

6.7. Autopipettor with disposable tips

6.8. Funnels: various

6.9. Watch glass: various

6.10. Analytical balance capable of accurately weighing ± 0.0001 g

6.11. Top load balance capable of accurately weighing ± 0.01 g

6.12. Narrow range pH paper (0-3 ± 0.25)

6.13. Snapseal containers

6.14. Graduated cylinders: various

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Sulfuric Acid (H_2SO_4): concentrated

7.1.2. Acetone: reagent grade. Avoid material that comes in containers with metal or metal-lined caps.

7.1.3. 1, 5 Diphenylcarbazide: reagent grade

7.1.4. Diphenylcarbazide Solution: Dissolve 0.25 g phenycarbazide in 50 mL acetone. Store in a brown bottle. Prepare daily.

7.1.5. Sodium Hydroxide (NaOH): reagent grade

7.1.6. Sodium Carbonate (Na_2CO_3): reagent grade

7.1.7. Alkaline Digestion Solution: Dissolve 20 g of sodium hydroxide and 30 g of sodium carbonate in water. Dilute to 1000 mL. Prepare fresh monthly.

7.1.8. Potassium Dichromate ($K_2Cr_2O_7$): primary standard grade

7.2. Standards

7.2.1. Primary Chromium Stock Solution, 50 mg/L: Dissolve 0.1414 g of dried potassium dichromate in 800 mL reagent water. Dilute to 1 liter in a volumetric flask with reagent water. Stable 6 months.

7.2.1.1. Calibration Standards: Add the indicated volume of the appropriate standard and dilute to volume with reagent water.

Conc., mg/L	mLs	Source, mg/L	Final Vol., mL
0.5	0.5	50	50
0.25	0.25	50	50
0.1	0.10	50	50
0.02	0.02	50	50

7.2.1.2. Secondary Chromium Stock Solution, 50 mg/L: Dissolve 0.1414 g dried potassium dichromate secondary source, in 800 mL reagent water. Dilute 1 liter in a volumetric flask with reagent water. Stable 6 months.

7.2.1.3. Verification Standards: Add the indicated volume of the appropriate standard and dilute to volume with reagent water.

Use	Conc., mg/L	mLs	Source, mg/L	Final Vol., mL
MS/MSD (waters)	0.25	0.25	50	50
MS/MSD (solids)	0.25	0.50	50	100
CCV	0.25	0.25	50	50
LCS (waters)	0.25	0.25	50	50
LCS (solids)	0.10	0.20	50	100

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. To retard the chemical activity of Hexavalent Chromium, the samples and extracts should be stored at 4°C ± 2°C until analyzed.

8.2. The maximum holding time prior to analysis for water samples is one day.

8.3. The holding time for solid samples is 30 days prior to digestion and 96 hours after digestion.

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. A reagent water blank consisting of 50 mL reagent water or 40 mL of digestion reagent must be prepared and analyzed 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 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. An LCS for waters, consisting of 50 mL of the 0.25 mg/L standard, is analyzed with each analytical batch of samples. An LCS for solids, consisting of 0.2 mL of the 50 mg/L secondary standard and 40 mL of digestion reagent is prepared and 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. 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 of sample and 0.25 mL (waters) or 2 g of sample and 0.5 mL (solids) of 50 mg/L secondary standard should be prepared and analyzed with every 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. For soil samples only: If the MS/MSD is not diluted out and the percent recovery of either the MS or MSD fails acceptance criteria, a post digestion spike must be analyzed. Prepare a post digestion spike at 0.1 mg/L by adding 0.1 mL of 50 mg/L secondary standard to 50 mL of sample.

NOTE: Sample may require dilution prior to addition of the post digestion spike.

9.4.3.4. 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.

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. A calibration curve is prepared daily prior to the analysis of the samples.

10.2. Initial Calibration

10.2.1. See Section11.4.3.6 for calibration procedure.

10.3. Continuing Calibration

10.3.1. The calibration is verified at the beginning, 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 ± 10% or recalibration is required.

10.3.2. The system cleanliness is verified at the beginning, every ten samples and at the end of the run using a CCB. The CCB is a 50 mL of reagent water or 40 mL of digestion reagent which is processed through all corresponding preparation and analysis steps like a method blank. It cannot contain the analyte of interest above the reporting limit or recalibration is required.

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. Water samples require minimal sample preparation. Solid samples require an alkaline digestion procedure after which the sample can be treated as a water throughout the remainder of the analysis.

11.3.1.1. Solids

11.3.1.1.1. Weigh 2.0 g of sample into a beaker and add 40 mL alkaline digestion solution. Record sample weight on analytical logsheet. Cover with a watch glass and reflux for two hours at 95°C. Cool beaker and filter through a Whatman #4 into 100 mL volumetric flask or equivalent. Dilute to volume with reagent water.

11.3.1.1.2. Prepare appropriate calibration standards and digest in the same manner as the samples.

11.3.1.2.Water Samples

11.3.1.2.1.No preparation is necessary.

11.4. Sample Analysis

11.4.1. An aliquot of prepared sample is acidified to a pH of < 2 and colorized with diphenylcarbizide solution. The samples are then analyzed on a spectrophotometer at 540 nm.

11.4.2. Recommended Instrument Conditions

11.4.2.1.Turn on the spectrophotometer and adjust the wavelength to 540 nm. Set the instrument to the absorbance mode.

11.4.3. Preliminary Evaluation

11.4.3.1.Preliminary evaluation can consist of visual determination by comparison to the standards to determine if dilutions are required.

11.4.3.2.Transfer 50 mL, or aliquot diluted to 50 mL with reagent water, of calibration standards, CCV, CCB, LCS's blanks, and water samples or solid digestion solution to suitable containers.

11.4.3.3.Add concentrated sulfuric acid (drop by drop) to each flask until the pH is 2.0 ± 0.5. Measure the pH by placing a drop of sample or digestate on narrow range pH paper (0 - 3 ± 0.25). Solid digests should take approximately 1 mL and all other samples (standards and waters) should take approximately 5 - 10 drops.

NOTE: For samples that may cause color interferences, a second acidified aliquot should be analyzed without the diphenylcarbizide solution. Record the absorbance on the analytical logsheet.

11.4.3.4.Add 1.0 mL diphenylcarbizide solution to each flask and swirl to mix.

11.4.3.5.Allow 10 minutes for color development.

11.4.3.6.Zero the instrument. Analyze initial calibration standards for both water and solid matrices as needed and record all absorbances on the analytical logsheet. Derive the correlation coefficient (r), slope (a), and y-intercept (b). The correlation

coefficient must be $\geq$ 0.995 for waters and $\geq$ 0.992 for solids to continue or reanalysis is required.

11.4.3.7. Analyze each sample on absorbance and record the value on the analytical logsheet.

11.4.3.8. A CCV and CCB are analyzed at the beginning and end of the run and every ten samples to verify linearity. The CCV must be ± 10% or recalibration is required. The CCB must be less than the reporting limit.

11.4.3.9. Absorbance values greater than the highest calibration standard must be diluted and reanalyzed. Follow procedures 11.4.3.2 - 11.4.3.5.

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, analysis time, 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. Use linear regression or the following equation to calculate sample results.

$$\frac{\text{Absorbance - a}}{\text{b}} = \text{mg / L } Cr^{6+}$$

Where:

a = y-intercept

b = slope

12.2. ***Waters, mg/L Cr^{+6} = (A - B) x C***

Solids, mg/kg Cr^{+6} = (A - B) x C x D

Where:

A = mg/L of sample colorized

B = mg/L of non-colorized sample

C = Dilution Factor = Final Volume / Initial Volume

D = Weight/Volume Factor - = Final Volume / Sample Weight

12.3. $$LCS\ Recovery = \frac{mg/L}{0.25\ (true)} \times 100$$

12.4. $$CCV\ Recovery = \frac{mg/L}{0.25\ (true)} \times 100$$

12.5. $$MS\ /\ MSD\ Recovery = \frac{E - F}{0.25\ (true)} \times 100$$

Where:

E = MS/MSD, mg/L

F = Sample, mg/L

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. Solvent waste must be disposed of in clearly labeled waste cans.

15.2. Acid waste must be collected in clearly labeled acid waste containers.

15.3. Solid materials (gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.4. Refer to the Laboratory Sample and Waste Disposal plan.

15.5. 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 an annual refresher training.

16. REFERENCES

16.1. References

16.1.1. SW846 Test Methods for Evaluating Solid Waste, Third Edition, Chromium, Hexavalent (Colorimetric), Method 7196A.

16.1.2. Standard Methods 3500-CR, 18th Edition

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Reporting limits

17.1.1. The lower reporting limit (RL) is 0.02 mg/L for waters and 1.0 mg/kg for solids.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Method deviations

17.2.1. SW-846 Method 7196A and Standard Methods 3500-CR, 18th edition, specify different pH requirements for the sample prior to color development (Section 11.4.3.3). The pH requirement from 7196A (2.0 ± 0.5) was selected for implementation in this SOP rather than the 3500-CR requirement (1.0 ± 0.3).

LABORATORY-SPECIFIC

QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: PH ELECTROMETRIC METHOD

(SUPERSEDES: REVISION 3)

Prepared by: Patrick O'Meara 2-16-99
Date

Reviewed by: Laurel E. Wood 2-16-99
Technology Specialist Date

Approved by: [signature] 2-11-99
Quality Assurance Manager Date

Approved by: [signature] 2-17-99
Environmental Health and Safety Coordinator Date

Approved by: [signature] 2-18-99
Laboratory Director Date

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1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of pH in waters, wastewaters, and solids. It is based on SW846 Methods 9040B and 9045C and EPA Method 150.1. The approximate working range is 1 - 14 pH units. Samples with a pH of < 1 are reported as < 1.

1.2. The associated method codes are PU (9040B), OZ (9045C), and AJ (150.1). The preparation codes are 88 and 1C.

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. The pH is determined electrometrically by using an electrode. The pH meter is calibrated with a series of known pH buffers.

3. DEFINITIONS

3.1. Refer to the glossary in the Quality Assurance Management Plan (QAMP), current version.

4. INTERFERENCES

4.1. Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode.

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 (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 Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. pH meter with electrode(s) and temperature compensation

6.2. Beakers: various

6.3. Top loading balance: Capable of accurately weighing ±0.01 g

6.4. Stir plate and stir bars

6.5. Shaker or mechanical tumbler

7. REAGENTS AND STANDARDS

7.1. Standards

7.1.1. Target Calibration Standards

7.1.1.1. pH 4, 7, and pH 10 buffers, purchased

7.1.1.2.Fresh buffers are poured and used each working day.

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. Samples should be analyzed as soon as possible after sampling, but not to exceed twenty-four hours.

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. Duplicates

9.2.1. Sample duplicates are performed at a minimum frequency one per analytical batch and must meet laboratory-specific limits for precision.

10. CALIBRATION AND STANDARDIZATION

10.1. Initial Calibration

10.1.1. Refer to the manufacturer's manual for instrumental calibration.

10.1.2. The following procedure is applicable for use with the Orion 240 pH meter.

10.1.2.1. Rinse the electrodes with reagent water and place in the pH 4.0 buffer. Press "Cal". Allow the value to stabilize and then, using the arrow keys, adjust the value up or down to read 4.00. Press Enter.

10.1.2.2. Rinse the electrodes and place in the pH 7.0 buffer. Allow the value to stabilize and then, using the arrow keys, adjust the value up or down to read 7.00. Press Enter.

10.1.2.3.Calibration Check: Rinse the electrodes and place in the pH 10.0 buffer. Allow value to stabilize. The pH should be between 9.95 and 10.05 or recalibration is necessary.

NOTE: When analyzing drinking water samples, calibrate as described in section 10.1.2, using the pH 7.0 and pH 10.0 buffers for calibration and the pH 4.0 buffer for the calibration check.

10.1.3. The pH meter should be calibrated daily. The calibration is recorded on the analytical logsheet.

10.1.4. If the pH meter has been turned off, it must be calibrated prior to use.

10.2. Continuing Calibration

10.2.1. A pH 7 buffer is analyzed before analysis and every ten samples to ensure the calibration remains linear.

10.2.2. The pH meter must be recalibrated if the buffer deviates by more than ± 0.05 s.u. If this range is exceeded, reanalyze all samples analyzed since the last pH buffer that met criteria.

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. Waters

11.3.1.1.No preparation necessary for waters and wastewaters.

11.3.2. Solids and Soils

11.3.2.1. Place 20 g (± 0.5 g) of sample in a beaker or other suitable container.

11.3.2.2. Add 20 mL of reagent water and mix for five minutes.

11.3.2.3. Allow sample to stand for one hour to allow the solids to settle out.

11.4. Sample Analysis

11.4.1. Waters

11.4.1.1. Place the sample in a clean beaker using a sufficient volume to cover the sensing elements of the electrode(s). Allow the pH to stabilize (swirling or stirring may quicken stabilization). Record the pH on the analytical logsheet. Remove the electrodes from the sample. Rinse and gently dab off the electrodes between each measurement. Store the electrodes in pH 7 buffer when not in use.

11.4.2. Solids

11.4.2.1. Immerse the pH electrodes in the supernatant layer of the sample - be careful not to stir up solids. Allow pH to stabilize and record it on the analytical logsheet. Remove and rinse the electrodes between each measurement. Store electrodes in the pH 7.0 buffer.

NOTE: If the sample contains oil or other substances that will coat or damage the electrodes, the pH should be analyzed following SOP# NC-WC-0009, pH - Paper Method.

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards 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.

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 Quanterra. 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, 3rd Edition, pH Electrometric Measurement, Method 9040B

16.1.2. EPA 600, Methods for Chemical Analysis of Water and Wastes, pH (Electrometric), Method 150.1

16.1.3. SW846, Test Methods for Evaluating Solid Waste, 3rd Edition, Soil pH, method 9045C.

16.1.4. Quanterra® Quality Assurance Management Plan (QAMP), current version.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Reporting limits

17.1.1. Values of < 1 will be reported as less than 1. pH units are reported as s.u.

STL Refere .ce Data Summary

Target Analyte List: All Analytes

Matrix: WATER
Extraction: LIQ/LIQ, CONT w/ACID STRIP (PCB) - Nominal
Method: PCBs (8082)
QC Program: MICHIGAN
Location: STL North Canton

Analyte List		Detection Limits				Check List 928					Spike List 929				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
PCB-1016	0.2	ug/L	0.054	ug/L	20000229										
Aroclor 1016	0.2	ug/L	0.054	ug/L	20000229	10	ug/L	61	118	20	10	ug/L	56	119	20
PCB-1221	0.2	ug/L	0.11	ug/L	20000229										
Aroclor 1221	0.2	ug/L	0.11	ug/L	20000229										
PCB-1232	0.4	ug/L	0.056	ug/L	20000229										
Aroclor 1232	0.4	ug/L	0.056	ug/L	20000229										
PCB-1242	0.2	ug/L	0.075	ug/L	20000229										
Aroclor 1242	0.2	ug/L	0.075	ug/L	20000229										
PCB-1248	0.2	ug/L	0.061	ug/L	20000229										
Aroclor 1248	0.2	ug/L	0.061	ug/L	20000229										
PCB-1254	0.2	ug/L	0.082	ug/L	20000229										
Aroclor 1254	0.2	ug/L	0.082	ug/L	20000229										
PCB-1260	0.2	ug/L	0.044	ug/L	20000229										
Aroclor 1260	0.2	ug/L	0.044	ug/L	20000229	10	ug/L	61	124	27	10	ug/L	31	138	27
Decachlorobiphenyl						0.2	ug/L	24	128	0	0.2	ug/L	24	128	0
Tetrachloro-m-xylene						0.2	ug/L	45	120	0	0.2	ug/L	45	120	0

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: NO SAMPLE PREPARATION PERFORMED / DIRECT INJE
Method: Sulfide (9030A)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits				Check List 1204					Spike List 1205				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Acid-insoluble Sulfide	50	mg/kg	21.5	mg/kg	19990419	20	mg/kg	80	120	20	20	mg/kg	80	120	20

STL Refere ce Data Summary

Target Analyte List: All Analytes

Matrix: WATER
Extraction: NO SAMPLE PREPARATION PERFORMED / DIRECT INJE
Method: Chromium, Hexavalent (7196A)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits				Check List 1225					Spike List 1226				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Dissolved Hexavalent Chromium	0.02	mg/L	0.0038	mg/L	19990312	0.25	mg/L	90	110	20	0.25	mg/L	90	110	20
Hexavalent Chromium	0.02	mg/L	0.0038	mg/L	19990312	0.25	mg/L	90	110	20	0.25	mg/L	90	110	20

STL Reference Data Summary

Target Analyte List: All Analytes

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

Analyte List		Detection Limits				Check List 1112					Spike List 1113				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Acetone	10	ug/L	0.86	ug/L	19990112										
Acetonitrile	20	ug/L	2.6	ug/L	19990113										
Acrolein	20	ug/L	2.5	ug/L	19990112										
Acrylonitrile	20	ug/L	0.84	ug/L	19990111										
Benzene	1	ug/L	0.06	ug/L	19990111	22	ug/L	80	116	20	28	ug/L	76	118	20
Benzyl chloride	10	ug/L	0.618	ug/L	19990521										
Bromobenzene	1	ug/L	0.054	ug/L	19990113										
Bromochloromethane	1	ug/L	0.15	ug/L	19990111										
Bromodichloromethane	1	ug/L	0.049	ug/L	19990118										
Dichlorobromomethane	1	ug/L	0.049	ug/L	19990118										
Bromoform	1	ug/L	0.13	ug/L	19990121										
Bromomethane	2	ug/L	0.14	ug/L	19990111										
Methyl bromide	2	ug/L	0.14	ug/L	19990111										
n-Butanol	50	ug/L	7.9	ug/L	19990119										
2-Butanone	10	ug/L	0.64	ug/L	19990112										
Methyl ethyl ketone	10	ug/L	0.64	ug/L	19990112										
2-Butanone (MEK)	10	ug/L	0.64	ug/L	19990112										
n-Butyl alcohol	50	ug/L	7.9	ug/L	19990119										
tert-Butyl alcohol	50	ug/L	50	ug/L	20000419										
n-Butylbenzene	1	ug/L	0.11	ug/L	19990113										
sec-Butylbenzene	1	ug/L	0.12	ug/L	19990113										
tert-Butylbenzene	1	ug/L	0.12	ug/L	19990113										
Carbon disulfide	1	ug/L	0.11	ug/L	19990113										
Carbon tetrachloride	1	ug/L	0.17	ug/L	19990113										
Chlorobenzene	1	ug/L	0.062	ug/L	19990118	22	ug/L	76	117	20	28	ug/L	76	117	20
Chloroprene	2	ug/L	0.36	ug/L	19990119										
Chlorodibromomethane	1	ug/L	0.06	ug/L	19990111										
Dibromochloromethane	1	ug/L	0.06	ug/L	19990111										
Chloroethane	2	ug/L	0.096	ug/L	19990111										
2-Chloroethyl vinyl ether	10	ug/L	0.29	ug/L	19990112										
Chloroform	1	ug/L	0.083	ug/L	19990111										
1-Chlorohexane	1	ug/L	0.10	ug/L	19950703										
Chloromethane	2	ug/L	0.085	ug/L	19990111										
Methyl chloride	2	ug/L	0.085	ug/L	19990111										
Allyl chloride	2	ug/L	0.33	ug/L	19990113										
3-Chloropropene	2	ug/L	0.33	ug/L	19990113										

Target Analyte List: All Analytes

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

Analyte List		Detection Limits				Check List 1112					Spike List 1113				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
2-Chlorotoluene	1	ug/L	0.063	ug/L	19990113										
4-Chlorotoluene	1	ug/L	0.074	ug/L	19990111										
Crotononitrile	10	ug/L	1.4	ug/L	19990125										
Cyclohexane	1	ug/L	0.14	ug/L	20000304										
Cyclohexanone	--	ug/L	7.1	ug/L	19990121										
1,2-Dibromo-3-chloropropane	2	ug/L	0.17	ug/L	19990112										
1,2-Dibromoethane	1	ug/L	0.046	ug/L	19990111										
1,2-Dibromoethane (EDB)	1	ug/L	0.046	ug/L	19990111										
Dibromomethane	1	ug/L	0.13	ug/L	19990111										
Methylene bromide	1	ug/L	0.13	ug/L	19990111										
1,2-Dichlorobenzene	1	ug/L	0.049	ug/L	19990111										
o-Dichlorobenzene	1	ug/L	0.049	ug/L	19990111										
1,3-Dichlorobenzene	1	ug/L	0.053	ug/L	19990113										
m-Dichlorobenzene	1	ug/L	0.053	ug/L	19990113										
1,4-Dichlorobenzene	1	ug/L	0.059	ug/L	19990113										
p-Dichlorobenzene	1	ug/L	0.059	ug/L	19990113										
trans-1,4-Dichloro-2-butene	1	ug/L	0.56	ug/L	19990112										
Dichlorodifluoromethane	2	ug/L	0.210	ug/L	19990113										
1,1-Dichloroethane	1	ug/L	0.096	ug/L	19990111										
1,2-Dichloroethane	1	ug/L	0.08	ug/L	19990111										
cis-1,2-Dichloroethene	0.5	ug/L	0.1	ug/L	19990111										
cis-1,2-Dichloroethylene	0.5	ug/L	0.1	ug/L	19990111										
trans-1,2-Dichloroethene	0.5	ug/L	0.07	ug/L	19990113										
trans-1,2-Dichloroethylene	0.5	ug/L	0.07	ug/L	19990113										
1,1-Dichloroethene	1	ug/L	0.12	ug/L	19990113	22	ug/L	63	130	20	28	ug/L	62	130	20
1,1-Dichloroethylene	1	ug/L	0.12	ug/L	19990113										
1,2-Dichloroethene (total)	1	ug/L	0.83	ug/L	19990120										
Dichlorofluoromethane	2	ug/L	0.4	ug/L	19990119										
1,2-Dichloropropane	1	ug/L	0.031	ug/L	19990119										
1,3-Dichloropropane	1	ug/L	0.054	ug/L	19990113										
2,2-Dichloropropane	1	ug/L	0.11	ug/L	19990111										
cis-1,3-Dichloropropene	1	ug/L	0.015	ug/L	19990120										
trans-1,3-Dichloropropene	1	ug/L	0.062	ug/L	19990111										
1,1-Dichloropropene	1	ug/L	0.1	ug/L	19990113										
1,3-Dichloropropene (total)	0.5	ug/L	0.19	ug/L	19970204										
Diisobutyl ketone	20	ug/L	20	ug/L	19990911										
1,4-Dioxane	200	ug/L	19	ug/L	19990112										
Ethyl acetate	4	ug/L	0.4	ug/L	19990119										
Ethylbenzene	1	ug/L	0.048	ug/L	19990108										

Target Analyte List: All Analytes

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

Analyte List		Detection Limits				Check List 1112					Spike List 1113				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Ethyl ether	2	ug/L	0.33	ug/L	19990121										
Diethyl ether	2	ug/L	0.33	ug/L	19990121										
Ethyl methacrylate	1	ug/L	0.62	ug/L	19990112										
Hexachlorobutadiene	1	ug/L	0.18	ug/L	19990113										
Hexane	1	ug/L	0.42	ug/L	19990120										
n-Hexane	1	ug/L	0.42	ug/L	19990120										
2-Hexanone	10	ug/L	0.51	ug/L	19990112										
Iodomethane	1	ug/L	0.094	ug/L	19990111										
Methyl iodide	1	ug/L	0.094	ug/L	19990111										
Isobutanol	50	ug/L	13	ug/L	19990121										
Isobutyl alcohol	50	ug/L	13	ug/L	19990121										
Isopropylbenzene	1	ug/L	0.064	ug/L	19990113										
Isopropyl ether	10	ug/L	0.88	ug/L	19990119										
p-Isopropyltoluene	1	ug/L	0.097	ug/L	19990113										
Methacrylonitrile	2	ug/L	0.74	ug/L	19990121										
Methyl acetate	10	ug/L	2.4	ug/L	20000304										
Methylcyclohexane	1	ug/L	0.083	ug/L	20000304										
Methylene chloride	1	ug/L	0.097	ug/L	19990111										
Methyl methacrylate	2	ug/L	0.44	ug/L	19990119										
4-Methyl-2-pentanone	10	ug/L	0.47	ug/L	19990112										
4-Methyl-2-pentanone (MIBK)	10	ug/L	0.47	ug/L	19990112										
Methyl tert-butyl ether	5	ug/L	0.35	ug/L	19990120										
Methyl tert-butyl ether (MTBE)	5	ug/L	0.35	ug/L	19990120										
Naphthalene	1	ug/L	0.083	ug/L	19990108										
2-Nitropropane	2	ug/L	0.12	ug/L	19990121										
Paraldehyde	10	ug/L	1.2	ug/L	19990125										
Propionitrile	4	ug/L	0.49	ug/L	19990119										
n-Propylbenzene	1	ug/L	0.089	ug/L	19990113										
Styrene	1	ug/L	0.3	ug/L	19990120										
1,1,1,2-Tetrachloroethane	1	ug/L	0.05	ug/L	19990111										
1,1,2,2-Tetrachloroethane	1	ug/L	0.077	ug/L	19990111										
Tetrachloroethene	1	ug/L	0.12	ug/L	19990113										
Tetrachloroethylene	1	ug/L	0.12	ug/L	19990113										
Tetrahydrofuran	5	ug/L	0.35	ug/L	19990112										
Thiophene	1	ug/L	0.084	ug/L	19990125										
Toluene	1	ug/L	0.041	ug/L	19990118	22	ug/L	74	119	20	28	ug/L	70	119	20
1,3,5-Trichlorobenzene	1	ug/L	1	ug/L	19990911										
1,2,3-Trichlorobenzene	1	ug/L	0.077	ug/L	19990113										
1,2,4-Trichlorobenzene	1	ug/L	0.06	ug/L	19990111										

Target Analyte List: All Analytes

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

Analyte List		Detection Limits				Check List 1112					Spike List 1113				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
1,1,1-Trichloroethane	1	ug/L	0.11	ug/L	19990113										
1,1,2-Trichloroethane	1	ug/L	0.075	ug/L	19990108										
Trichloroethene	2	ug/L	0.3	ug/L	19990113										
Trichloroethylene	2	ug/L	0.3	ug/L	19990113	22	ug/L	75	122	20	28	ug/L	62	130	20
Trichlorofluoromethane	2	ug/L	0.3	ug/L	19990113										
1,2,3-Trichloropropane	1	ug/L	0.11	ug/L	19990120										
1,1,2-Trichloro-1,2,2-trifluoroethane	1	ug/L	0.260	ug/L	19990113										
Trichlorotrifluoroethane	1	ug/L	0.260	ug/L	19990113										
1,2,4-Trimethylbenzene	1	ug/L	0.06	ug/L	19990111										
1,3,5-Trimethylbenzene	1	ug/L	0.065	ug/L	19990111										
Vinyl acetate	2	ug/L	0.25	ug/L	19990112										
Vinyl chloride	2	ug/L	0.12	ug/L	19990111										
m-Xylene & p-Xylene	1	ug/L	0.78	ug/L	19990120										
o-Xylene	0.5	ug/L	0.044	ug/L	19990108										
Xylenes (total)	1	ug/L	0.36	ug/L	19980223										
4-Bromofluorobenzene						22	ug/L	74	116	0	28	ug/L	74	116	0
1,2-Dichloroethane-d4						22	ug/L	61	128	0	28	ug/L	61	128	0
Toluene-d8						22	ug/L	76	110	0	28	ug/L	76	110	0
Dibromofluoromethane						22	ug/L	73	122	0	28	ug/L	73	122	0

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: WATER
Extraction: LIQ/LIQ, CONT (Acids,Phenols) - Acid
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits				Check List 1114					Spike List 1115				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
a,a-Dimethylphenethylamine	50	ug/L	12	ug/L	19980309										
Acenaphthene	10	ug/L	1.1	ug/L	19990217	50	ug/L	39	118	35	50	ug/L	26	118	35
Acenaphthylene	10	ug/L	1.0	ug/L	19990119			48	101	21			48	96	21
Acetophenone	10	ug/L	1.7	ug/L	19990121										
2-Acetylaminofluorene	100	ug/L	1.2	ug/L	19990121										
4-Aminobiphenyl	50	ug/L	1.3	ug/L	19990121										
Aniline	10	ug/L	0.880	ug/L	19990217										
Anthracene	10	ug/L	0.93	ug/L	19990119			56	105	18			52	101	18
Aramite	20	ug/L	1.5	ug/L	19990121										
Atrazine	10	ug/L	2.3	ug/L	20000217										
Benzaldehyde	10	ug/L	1.6	ug/L	20000226										
Benz(a)anthracene	10	ug/L	1.00	ug/L	19990119										
Benzenethiol	10	ug/L	2.3	ug/L	19990629										
Benzidine	100	ug/L	19	ug/L	19990126										
Benzo(a)anthracene	10	ug/L	1.00	ug/L	19990119			56	109	16			52	110	16
Benzo(b)fluoranthene	10	ug/L	1.3	ug/L	19990119			52	108	20			48	107	20
Benzo(k)fluoranthene	10	ug/L	1.3	ug/L	19990119			53	112	20			53	109	20
Benzoic acid	50	ug/L	6.8	ug/L	19990619										
Benzo(ghi)perylene	10	ug/L	1.1	ug/L	19990119			45	115	17			48	109	17
Benzo(a)pyrene	10	ug/L	1.0	ug/L	19990217			50	100	18			47	98	18
Benzothiazole	10	ug/L	2.4	ug/L	19980527										
Benzyl Mercaptan	10	ug/L	3.5	ug/L	19980527										
o-Benzyl Phenol	10	ug/L	2.4	ug/L	19980527										
Benzyl alcohol	10	ug/L	0.94	ug/L	19990119										
1,1'-Biphenyl	10	ug/L	2.1	ug/L	20000217										
bis(2-Chloroethoxy)methane	10	ug/L	0.97	ug/L	19990119			39	109	26			40	101	40
bis(2-Chloroethyl) ether	10	ug/L	1.1	ug/L	19990217			45	103	26			36	104	26
bis(2-Chloroisopropyl) ether	10	ug/L	0.92	ug/L	19990119			49	136	25			43	133	25
bis(2-Chloro-1-methylethyl) ether	10	ug/L	0.92	ug/L	19990119										
bis(2-Ethylhexyl) phthalate	10	ug/L	3.8	ug/L	19990126			56	127	23			44	133	23
4-Bromophenyl phenyl ether	10	ug/L	1.10	ug/L	19990119			57	114	17			56	110	17
2,3-Butanedithiol	10	ug/L	4.8	ug/L	19980527										
1,4-Butanedithiol	10	ug/L	4.2	ug/L	19980527										
Butyl benzyl phthalate	10	ug/L	1.2	ug/L	19990119			53	113	18			46	115	18
Caprolactam	10	ug/L	1	ug/L	20000217										
Carbazole	10	ug/L	1.2	ug/L	19990217			37	114	21			42	115	21

Target Analyte List: All Analytes

Matrix: WATER
Extraction: LIQ/LIQ, CONT (Acids,Phenols) - Acid
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List	Detection Limits					Check List 1114					Spike List 1115				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
p-Chloroaniline	10	ug/L	1.7	ug/L	19990217										
4-Chloroaniline	10	ug/L	1.7	ug/L	19990217			19	82	41			13	71	41
Chlorobenzilate	10	ug/L	1.7	ug/L	19990121										
4-Chloro-3-methylphenol	10	ug/L	0.96	ug/L	19990217	75	ug/L	29	124	55	75	ug/L	21	124	55
p-Chloro-m-cresol	10	ug/L	0.96	ug/L	19990217										
2-Chloronaphthalene	10	ug/L	1.1	ug/L	19990217			51	106	25			46	104	25
2-Chlorophenol	10	ug/L	1	ug/L	19990217	75	ug/L	19	124	43	75	ug/L	19	124	43
4-Chlorophenyl phenyl ether	10	ug/L	1	ug/L	19990119			57	114	19			55	110	19
3-Chloropropionitrile	10	ug/L	1	ug/L	19990217										
Chrysene	10	ug/L	1.1	ug/L	19990217			59	112	16			54	115	16
6-Methylchrysene	20	ug/L	2.9	ug/L	19990629										
m-Cresol	10	ug/L	1.3	ug/L	19990121										
p-Cresol	10	ug/L	2.4	ug/L	19990217										
m-Cresol & p-Cresol	20	ug/L	20	ug/L	19991202										
Cresols (total)	10	ug/L	2.4	ug/L	19990217										
Cyclohexylamine	200	ug/L	9.07	ug/L	19980529										
Diallate	20	ug/L	1.8	ug/L	19990121										
2,4-Diaminotoluene	50	ug/L	50	ug/L	20000126										
Dibenz(a,h)acridine	50	ug/L	2.5	ug/L	19990629										
Dibenz(a,j)acridine	50	ug/L	2.5	ug/L	19990629										
Dibenz(a,h)anthracene	10	ug/L	1.2	ug/L	19990119			50	112	18			49	110	18
Dibenzofuran	10	ug/L	1.1	ug/L	19990119			55	107	20			53	104	20
1,3-Dibutyl-2-Thiourea	20	ug/L	12	ug/L	19980527										
Di-n-butyl phthalate	10	ug/L	1.3	ug/L	19990119			59	108	17			53	109	17
1,2-Dichlorobenzene	10	ug/L	0.92	ug/L	19990119			39	90	31			33	91	29
o-Dichlorobenzene	10	ug/L	0.92	ug/L	19990119										
1,3-Dichlorobenzene	10	ug/L	0.920	ug/L	19990119			34	85	31			30	86	31
m-Dichlorobenzene	10	ug/L	0.920	ug/L	19990119										
1,4-Dichlorobenzene	10	ug/L	1.00	ug/L	19990119	50	ug/L	28	110	36	50	ug/L	18	110	36
p-Dichlorobenzene	10	ug/L	1.00	ug/L	19990119										
3,3'-Dichlorobenzidine	50	ug/L	0.96	ug/L	19990217			20	76	36			10	71	36
2,4-Dichlorophenol	10	ug/L	1.4	ug/L	19990217			48	101	26			43	103	26
2,6-Dichlorophenol	10	ug/L	1.3	ug/L	19990121										
1,3-Diethyl-2-Thiourea	10	ug/L	8.9	ug/L	19980527										
Diethyl phthalate	10	ug/L	1.3	ug/L	19990119			48	112	20			36	117	20
O,O-Diethyl-O-(2-pyrazinyl) phosphoro	50	ug/L	1.6	ug/L	19990121										
2,3-Dimercapto-1-Propanol	10	ug/L	10	ug/L	19991202										
Dimethoate	20	ug/L	1.6	ug/L	19990121										
1,3-Dimethyl-2-Thiourea	10	ug/L	7.6	ug/L	19980527										

Target Analyte List: All Analytes

Matrix: WATER
Extraction: LIQ/LIQ, CONT (Acids,Phenols) - Acid
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits				Check List 1114					Spike List 1115				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
N,N-Dimethylacetamide	10	ug/L	1.5	ug/L	19990120										
p-Dimethylaminoazobenzene	20	ug/L	1.6	ug/L	19990121										
7,12-Dimethylbenz(a)anthracene	20	ug/L	1.5	ug/L	19990121										
3,3'-Dimethylbenzidine	50	ug/L	1.4	ug/L	19990121										
2,4-Dimethylphenol	10	ug/L	2.1	ug/L	19990217			10	88	28			10	88	28
Dimethyl phthalate	10	ug/L	1	ug/L	19990217			46	117	22			32	124	22
1,2-Dinitrobenzene	10	ug/L	0.740	ug/L	19990217										
m-Dinitrobenzene	10	ug/L	1.5	ug/L	19990121										
1,3-Dinitrobenzene	10	ug/L	1.5	ug/L	19990121										
p-Dinitrobenzene	10	ug/L	1.2	ug/L	19990121										
1,4-Dinitrobenzene	10	ug/L	1.2	ug/L	19990121										
4,6-Dinitro-2-methylphenol	50	ug/L	0.68	ug/L	19990217			37	137	24			46	123	24
4,6-Dinitro-o-cresol	50	ug/L	0.68	ug/L	19990217										
2,4-Dinitrophenol	50	ug/L	3.3	ug/L	19980305			21	143	32			30	133	32
2,4-Dinitrotoluene	10	ug/L	0.89	ug/L	19990119	50	ug/L	47	131	32	50	ug/L	31	131	32
2,6-Dinitrotoluene	10	ug/L	0.86	ug/L	19990119			62	114	16			58	109	16
Dinoseb	20	ug/L	0.93	ug/L	19990121										
2-sec-Butyl-4,6-dinitrophenol	20	ug/L	0.93	ug/L	19990121										
Di-n-octyl phthalate	10	ug/L	1.3	ug/L	19990119			49	127	22			46	124	22
1,4-Dioxane	10	ug/L	1.7	ug/L	19990121										
Diphenyl	10	ug/L	2.1	ug/L	20000217										
Diphenyl Thiourea	10	ug/L	5.7	ug/L	19980527										
Diphenylamine	10	ug/L	1.1	ug/L	19990119										
Diphenyl ether	10	ug/L	0.48	ug/L	19990222										
1,2-Diphenylhydrazine	10	ug/L	1.0	ug/L	19990119										
Disulfoton	50	ug/L	2.2	ug/L	19990121										
1,2-Ethanedithiol	50	ug/L	8.3	ug/L	19980527										
Ethyl methacrylate	10	ug/L	1	ug/L	19990119										
Ethyl methanesulfonate	10	ug/L	1.7	ug/L	19990121										
Famphur	100	ug/L	9.8	ug/L	19980306										
Fluoranthene	10	ug/L	1.1	ug/L	19990119			53	116	19			51	113	19
Fluorene	10	ug/L	1.2	ug/L	19990217			57	107	18			54	105	19
Hexachlorobenzene	10	ug/L	1.3	ug/L	19990217			57	128	22			36	132	22
Hexachlorobutadiene	10	ug/L	1.2	ug/L	19990119			36	116	32			18	116	32
Hexachlorocyclopentadiene	50	ug/L	2.1	ug/L	19990420			10	81	59			10	45	59
Hexachloroethane	10	ug/L	1.1	ug/L	19990217			30	110	33			18	110	33
Hexachloropropene	100	ug/L	1.3	ug/L	19990121										
Hexamethylphosphoramide	0.5	ug/L			0										
Indene	20	ug/L	2.7	ug/L	19990629										

Target Analyte List: All Analytes

Matrix: WATER
Extraction: LIQ/LIQ, CONT (Acids,Phenols) - Acid
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits				Check List 1114					Spike List 1115				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Indeno(1,2,3-cd)pyrene	10	ug/L	1.4	ug/L	19990217			49	114	19			48	113	19
Isophorone	10	ug/L	0.9	ug/L	19990119			48	103	25			42	102	25
Isosafrole	20	ug/L	2	ug/L	19990120										
Malononitrile	10	ug/L	1.1	ug/L	19990217										
Methapyrilene	50	ug/L	6.2	ug/L	19990421										
3-Methylcholanthrene	20	ug/L	2.16	ug/L	19990421										
4,4'-Methylenebis(2-chloroaniline) (1)	10	ug/L	1.5	ug/L	19990119										
Methyl methanesulfonate	10	ug/L	1.7	ug/L	19990121										
2-Methylnaphthalene	10	ug/L	1.10	ug/L	19990119			49	98	28			39	102	28
1-Methylnaphthalene	10	ug/L	1	ug/L	19990119										
Methyl parathion	50	ug/L	NA	ug/L	19800101										
2-Methylphenol	10	ug/L	2.4	ug/L	19990217			33	115	31			29	115	31
o-Cresol	10	ug/L	2.4	ug/L	19990217										
3-Methylphenol	10	ug/L	1.3	ug/L	19990121										
4-Methylphenol	10	ug/L	2.4	ug/L	19990217			29	144	33			25	144	33
3-Methylphenol & 4-Methylphenol	20	ug/L	20	ug/L	19991202										
Naphthalene	10	ug/L	1.1	ug/L	19990119			46	95	26			39	96	26
1,4-Naphthoquinone	50	ug/L	12	ug/L	19990421										
1-Naphthylamine	10	ug/L	1.3	ug/L	19990121										
2-Naphthylamine	10	ug/L	1.7	ug/L	19990121										
2-Nitroaniline	50	ug/L	0.730	ug/L	19990217			55	119	17			44	116	17
o-Nitroaniline	50	ug/L	0.730	ug/L	19990217										
3-Nitroaniline	50	ug/L	1.3	ug/L	19990217			33	107	23			20	102	23
m-Nitroaniline	50	ug/L	1.3	ug/L	19990217										
4-Nitroaniline	50	ug/L	1.2	ug/L	19990119			32	106	26			25	95	26
p-Nitroaniline	50	ug/L	1.2	ug/L	19990119										
Nitrobenzene	10	ug/L	1.0	ug/L	19990217			45	130	50			10	211	50
2-Nitrophenol	10	ug/L	1.2	ug/L	19990217			43	104	26			35	104	26
o-Nitrophenol	10	ug/L	1.2	ug/L	19990217										
4-Nitrophenol	50	ug/L	0.560	ug/L	19990217	75	ug/L	19	144	34	75	ug/L	10	145	34
p-Nitrophenol	50	ug/L	0.560	ug/L	19990217										
4-Nitroquinoline-1-oxide	100	ug/L	5.2	ug/L	19990421										
N-Nitrosodi-n-butylamine	10	ug/L	2.9	ug/L	19990121										
N-Nitrosodiethylamine	10	ug/L	1.3	ug/L	19990121										
N-Nitrosodimethylamine	10	ug/L	1	ug/L	19990217										
N-Nitrosodiphenylamine	10	ug/L	1.1	ug/L	19990119			47	112	18			53	99	18
N-Nitrosodi-n-propylamine	10	ug/L	0.9	ug/L	19990217	50	ug/L	30	115	36	50	ug/L	18	115	36
N-Nitrosomethylethylamine	10	ug/L	1.2	ug/L	19990121										
N-Nitrosomorpholine	10	ug/L	1.1	ug/L	19990121										

Target Analyte List: All Analytes

Matrix: WATER
Extraction: LIQ/LIQ CONT (Acids,Phenols) - Acid
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits				Check List 1114					Spike List 1115				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
N-Nitrosopiperidine	10	ug/L	1.1	ug/L	19990121										
N-Nitrosopyrrolidine	10	ug/L	0.9	ug/L	19990121										
5-Nitro-o-toluidine	20	ug/L	1.6	ug/L	19990121										
2,2'-oxybis(1-Chloropropane)	10	ug/L	0.92	ug/L	19990119										
Pentachlorobenzene	10	ug/L	1.5	ug/L	19990121										
Pentachloroethane	50	ug/L	1.7	ug/L	19990121										
Pentachloronitrobenzene	50	ug/L	1.6	ug/L	19990121										
Pentachlorophenol	10	ug/L	0.65	ug/L	19990217	75	ug/L	10	140	56	75	ug/L	10	140	56
Phenacetin	20	ug/L	1.6	ug/L	19990121										
Phenanthrene	10	ug/L	1.1	ug/L	19990119			58	110	18			55	109	18
Phenol	10	ug/L	1.1	ug/L	19990217	75	ug/L	10	131	43	75	ug/L	10	131	43
p-Phenylene diamine	100	ug/L	2	ug/L	19990421										
Phenyl ether	10	ug/L	0.48	ug/L	19990222										
Phorate	50	ug/L	1.8	ug/L	19990121										
2-Picoline	20	ug/L	1.9	ug/L	19990120										
Pronamide	20	ug/L	1.6	ug/L	19990121										
1,3-Propanedithiol	10	ug/L	4.9	ug/L	19980527										
2,2'-Oxybis(1-Chloropropane)	10	ug/L	0.92	ug/L	19990119										
Pyrene	10	ug/L	1.4	ug/L	19990217	50	ug/L	46	130	31	50	ug/L	27	138	31
Pyridine	20	ug/L	7.1	ug/L	19990126										
Quinoline	10	ug/L	0.87	ug/L	19990222										
Safrole	20	ug/L	1.4	ug/L	19990121										
Sulfotepp	50	ug/L	1.7	ug/L	19990121										
1,2,3,5-Tetrachlorobenzene	10	ug/L	0.97	ug/L	19990119										
1,2,4,5-Tetrachlorobenzene	10	ug/L	1.4	ug/L	19990121										
1,2,3,4-Tetrachlorobenzene	10	ug/L	1.3	ug/L	19990217										
2,3,4,6-Tetrachlorophenol	50	ug/L	1.3	ug/L	19990121										
2,3,5,6-Tetrachlorophenol	10	ug/L	1.1	ug/L	19990217										
Tetraethyldithiopyrophosphate	50	ug/L	1.7	ug/L	19990121										
1,1,3,3-Tetramethyl-2-Thiourea	10	ug/L	2.5	ug/L	19980527										
Thionazin	50	ug/L	1.6	ug/L	19990121										
Thiourea	1000	ug/L	9.48	ug/L	19980529										
2,4-Toluenediamine	50	ug/L	50	ug/L	20000126										
o-Toluidine	20	ug/L	4.5	ug/L	19990121										
1,3,5-Trichlorobenzene	10	ug/L	1.1	ug/L	19990119										
1,2,3-Trichlorobenzene	10	ug/L	1.3	ug/L	19990217										
1,2,4-Trichlorobenzene	10	ug/L	1.1	ug/L	19990119	50	ug/L	31	110	37	50	ug/L	22	110	37
2,4,5-Trichlorophenol	10	ug/L	1.10	ug/L	19990217			41	125	22			24	143	22
2,4,6-Trichlorophenol	10	ug/L	0.72	ug/L	19990217			46	135	27			36	135	27

Target Analyte List: All Analytes

Matrix: WATER
Extraction: LIQ/LIQ, CONT (Acids,Phenols) - Acid
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits				Check List 1114					Spike List 1115				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
O,O,O-Triethyl phosphorothioate	50	ug/L	1.6	ug/L	19990121										
sym-Trinitrobenzene	50	ug/L	0.920	ug/L	19990121										
1,3,5-Trinitrobenzene	50	ug/L	0.920	ug/L	19990121										
3-Picoline	10	ug/L	2.5	ug/L	19990120										
2-Fluorobiphenyl						50	ug/L	30	110	0	50	ug/L	30	110	0
2-Fluorophenol						75	ug/L	13	110	0	75	ug/L	13	110	0
2,4,6-Tribromophenol						75	ug/L	21	122	0	75	ug/L	21	122	0
Nitrobenzene-d5						50	ug/L	32	112	0	50	ug/L	32	112	0
Phenol-d5						75	ug/L	10	113	0	75	ug/L	10	113	0
Terphenyl-d14						50	ug/L	10	144	0	50	ug/L	10	144	0

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: WATER
Extraction: LIQ/LIQ, CONT w/ACID STRIP (PCB) - Nominal
Method: PCBs (8082)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits				Check List 928					Spike List 929				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
PCB-1016	1	ug/L	0.08	ug/L	19990406										
Aroclor 1016	1	ug/L	0.08	ug/L	19990406	10	ug/L	61	118	20	10	ug/L	56	119	20
PCB-1221	1	ug/L	0.062	ug/L	19990406										
Aroclor 1221	1	ug/L	0.062	ug/L	19990406										
PCB-1232	1	ug/L	0.34	ug/L	19990406										
Aroclor 1232	1	ug/L	0.34	ug/L	19990406										
PCB-1242	1	ug/L	0.31	ug/L	19990406										
Aroclor 1242	1	ug/L	0.31	ug/L	19990406										
PCB-1248	1	ug/L	0.22	ug/L	19990406										
Aroclor 1248	1	ug/L	0.22	ug/L	19990406										
PCB-1254	1	ug/L	0.054	ug/L	19990406										
Aroclor 1254	1	ug/L	0.054	ug/L	19990406										
PCB-1260	1	ug/L	0.093	ug/L	19990406										
Aroclor 1260	1	ug/L	0.093	ug/L	19990406	10	ug/L	61	124	27	10	ug/L	31	138	27
PCB-1262	1	ug/L	1	ug/L	19991207										
Aroclor 1262	1	ug/L	1	ug/L	19991207										
PCB-1268	1	ug/L	0.41	ug/L	19980319										
Aroclor 1268	1	ug/L	0.41	ug/L	19980319										
PCBs (total)	0.5	ug/L	0.5	ug/L	19991207										
Aroclors (Total)	0.5	ug/L	0.5	ug/L	19991207										
Decachlorobiphenyl						0.2	ug/L	24	128	0	0.2	ug/L	24	128	0
Tetrachloro-m-xylene						0.2	ug/L	45	120	0	0.2	ug/L	45	120	0

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: PURGE AND TRAP - Lab Preserved Low Level - Solids
Method: Volatile Organics, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits				Check List 924					Spike List 925				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Acetone	20	ug/kg	6.6	ug/kg	19980302	20	ug/kg	80	120	18	50	ug/kg	80	120	18
Acetonitrile	100	ug/kg	100	ug/kg	19990924										
Acrolein	100	ug/kg	25	ug/kg	19990104										
Acrylonitrile	100	ug/kg	9	ug/kg	19990104										
Benzene	5	ug/kg	0.18	ug/kg	19990105	20	ug/kg	75	129	20	50	ug/kg	55	138	20
Bromobenzene	5	ug/kg	0.29	ug/kg	19990104										
Bromochloromethane	5	ug/kg	0.22	ug/kg	19990104										
Bromodichloromethane	5	ug/kg	0.15	ug/kg	19990104	20	ug/kg	35	155	21	50	ug/kg	35	155	21
Dichlorobromomethane	5	ug/kg	0.15	ug/kg	19990104										
Bromoform	5	ug/kg	0.22	ug/kg	19990104	20	ug/kg	45	169	22	50	ug/kg	45	169	22
Tribromomethane	5	ug/kg	0.22	ug/kg	19990104										
Bromomethane	10	ug/kg	0.66	ug/kg	19990104	20	ug/kg	10	242	11	50	ug/kg	10	242	11
Methyl bromide	10	ug/kg	0.66	ug/kg	19990104										
2-Butanone	20	ug/kg	0.99	ug/kg	19990104	20	ug/kg	20	155	47	50	ug/kg	10	187	47
Methyl ethyl ketone	20	ug/kg	0.99	ug/kg	19990104										
2-Butanone (MEK)	20	ug/kg	0.99	ug/kg	19990104										
n-Butyl alcohol	1000	ug/kg			0										
tert-Butyl alcohol	200	ug/kg	78	ug/kg	19980302										
n-Butylbenzene	5	ug/kg	0.27	ug/kg	19990105										
sec-Butylbenzene	5	ug/kg	0.24	ug/kg	19990105										
tert-Butylbenzene	5	ug/kg	0.43	ug/kg	19990104										
Carbon disulfide	5	ug/kg	0.17	ug/kg	19990104	20	ug/kg	81	125	19	50	ug/kg	81	125	19
Carbon tetrachloride	5	ug/kg	0.27	ug/kg	19990104	20	ug/kg	66	141	55	50	ug/kg	39	149	55
Chlorobenzene	5	ug/kg	0.2	ug/kg	19990104	20	ug/kg	75	127	22	50	ug/kg	49	139	22
Chlorobromomethane	5	ug/kg	0.22	ug/kg	19990104										
Chloroprene	5	ug/kg	5	ug/kg	19990924										
2-Chloro-1,3-butadiene	5	ug/kg	5	ug/kg	19990924										
Chlorodibromomethane	5	ug/kg	0.24	ug/kg	19990104										
Dibromochloromethane	5	ug/kg	0.24	ug/kg	19990104	20	ug/kg	53	149	16	50	ug/kg	53	149	16
Chloroethane	10	ug/kg	0.6	ug/kg	19990104	20	ug/kg	82	114	11	50	ug/kg	82	114	11
2-Chloroethyl vinyl ether	50	ug/kg	50	ug/kg	19990924										
Chloroform	5	ug/kg	0.26	ug/kg	19990105	20	ug/kg	77	126	17	50	ug/kg	52	140	17
Chloromethane	10	ug/kg	0.5	ug/kg	19990104	20	ug/kg	10	273	18	50	ug/kg	10	273	18
Methyl chloride	10	ug/kg	0.5	ug/kg	19990104										
Allyl chloride	10	ug/kg	10	ug/kg	19990924										
3-Chloro-1-propene	10	ug/kg	10	ug/kg	19990924										

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: PURGE AND TRAP - Lab Preserved Low Level - Solids
Method: Volatile Organics, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits				Check List 924					Spike List 925				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
2-Chlorotoluene	5	ug/kg	0.21	ug/kg	19990104										
4-Chlorotoluene	5	ug/kg	0.42	ug/kg	19990104										
Cyclohexane	10	ug/kg	10	ug/kg	20000310										
1,2-Dibromo-3-chloropropane	10	ug/kg	0.38	ug/kg	19990104										
1,2-Dibromoethane	5	ug/kg	0.32	ug/kg	19990104										
1,2-Dibromoethane (EDB)	5	ug/kg	0.32	ug/kg	19990104										
Dibromomethane	5	ug/kg	0.46	ug/kg	19990104										
Methylene bromide	5	ug/kg	0.46	ug/kg	19990104										
1,2-Dichlorobenzene	5	ug/kg	0.32	ug/kg	19990105										
o-Dichlorobenzene	5	ug/kg	0.32	ug/kg	19990105										
1,3-Dichlorobenzene	5	ug/kg	0.13	ug/kg	19990104										
m-Dichlorobenzene	5	ug/kg	0.13	ug/kg	19990104										
1,4-Dichlorobenzene	5	ug/kg	0.61	ug/kg	19990105										
p-Dichlorobenzene	5	ug/kg	0.61	ug/kg	19990105										
trans-1,4-Dichloro-2-butene	5	ug/kg	1	ug/kg	19990104										
Dichlorodifluoromethane	10	ug/kg	0.18	ug/kg	19990105										
1,1-Dichloroethane	5	ug/kg	0.16	ug/kg	19990105	20	ug/kg	59	155	12	50	ug/kg	33	137	14
1,2-Dichloroethane	5	ug/kg	0.29	ug/kg	19990105	20	ug/kg	76	127	41	50	ug/kg	44	145	41
cis-1,2-Dichloroethene	2.5	ug/kg	0.18	ug/kg	19990104	20	ug/kg	50	150	50	50	ug/kg	50	150	50
cis-1,2-Dichloroethylene	2.5	ug/kg	0.18	ug/kg	19990104										
trans-1,2-Dichloroethene	2.5	ug/kg	0.2	ug/kg	19990105	20	ug/kg	54	156	10	50	ug/kg	54	156	10
trans-1,2-Dichloroethylene	2.5	ug/kg	0.2	ug/kg	19990105										
1,1-Dichloroethene	5	ug/kg	0.68	ug/kg	19990105	20	ug/kg	55	142	27	50	ug/kg	43	147	27
Vinylidene chloride	5	ug/kg	0.68	ug/kg	19990105										
1,1-Dichloroethylene	5	ug/kg	0.68	ug/kg	19990105										
1,2-Dichloroethene (total)	5	ug/kg	0.37	ug/kg	19990104	40	ug/kg	50	150	50	100	ug/kg	50	150	50
Dichlorofluoromethane	10	ug/kg	10	ug/kg	19990924										
1,2-Dichloropropane	5	ug/kg	0.27	ug/kg	19990104	20	ug/kg	10	210	13	50	ug/kg	10	210	13
1,3-Dichloropropane	5	ug/kg	0.38	ug/kg	19990104										
2,2-Dichloropropane	5	ug/kg	0.19	ug/kg	19990104										
cis-1,3-Dichloropropene	5	ug/kg	0.24	ug/kg	19990104	20	ug/kg	10	227	14	50	ug/kg	10	227	14
trans-1,3-Dichloropropene	5	ug/kg	0.3	ug/kg	19990104	20	ug/kg	17	183	14	50	ug/kg	17	183	14
trans-1,3-Dichloropropylene	5	ug/kg	0.3	ug/kg	19990104										
1,1-Dichloropropene	5	ug/kg	0.22	ug/kg	19990105										
cis-1,3-Dichloropropylene	5	ug/kg	0.24	ug/kg	19990104										
Diisobutyl ketone	1000	ug/kg	2	ug/kg	19980725										
1,4-Dioxane	500	ug/kg	27	ug/kg	19990104										
Ethanol	5000	ug/kg	5000	ug/kg	19990924										
Ethyl acetate	20	ug/kg	6.4	ug/kg	19980725										

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: PURGE AND TRAP - Lab Preserved Low Level - Solids
Method: Volatile Organics, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List	Detection Limits					Check List 924					Spike List 925				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Ethylbenzene	5	ug/kg	0.28	ug/kg	19990104	20	ug/kg	37	162	14	50	ug/kg	37	162	14
Ethyl ether	5	ug/kg	5	ug/kg	19990924										
Ethyl methacrylate	5	ug/kg	0.28	ug/kg	19990104										
Freon 113	5	ug/kg	0.28	ug/kg	19990104										
Hexachlorobutadiene	5	ug/kg	0.52	ug/kg	19990104										
Hexane	5	ug/kg	0.31	ug/kg	19990105										
n-Hexane	5	ug/kg	0.31	ug/kg	19990105			98	117	20			66	110	20
2-Hexanone	20	ug/kg	0.48	ug/kg	19990104	20	ug/kg	87	129	17	50	ug/kg	87	129	17
Iodomethane	5	ug/kg	0.19	ug/kg	19990105										
Methyl iodide	5	ug/kg	0.19	ug/kg	19990105										
Isobutyl alcohol	200	ug/kg	200	ug/kg	19990924										
Isopropylbenzene	5	ug/kg	0.23	ug/kg	19990104										
Cumene	5	ug/kg	0.23	ug/kg	19990104										
4-Isopropyltoluene	5	ug/kg	0.24	ug/kg	19990105										
p-Isopropyltoluene	5	ug/kg	0.24	ug/kg	19990105										
Methacrylonitrile	5	ug/kg	5	ug/kg	19990924										
Methyl acetate	10	ug/kg	10	ug/kg	20000310										
Methylcyclohexane	10	ug/kg	10	ug/kg	20000310										
Methylene chloride	5	ug/kg	0.98	ug/kg	19990104	20	ug/kg	10	221	22	50	ug/kg	10	221	22
Methyl methacrylate	5	ug/kg	5	ug/kg	19990924										
4-Methyl-2-pentanone	20	ug/kg	0.44	ug/kg	19990104	20	ug/kg	90	125	12	50	ug/kg	90	125	12
4-Methyl-2-pentanone (MIBK)	20	ug/kg	0.44	ug/kg	19990104										
Methyl tert-butyl ether	20	ug/kg	0.19	ug/kg	19990105										
Naphthalene	5	ug/kg	0.29	ug/kg	19990105										
Propionitrile	20	ug/kg	20	ug/kg	19990924										
n-Propylbenzene	5	ug/kg	0.37	ug/kg	19990104										
Styrene	5	ug/kg	0.31	ug/kg	19990104	20	ug/kg	79	100	10	50	ug/kg	79	110	10
1,1,1,2-Tetrachloroethane	5	ug/kg	0.23	ug/kg	19990104										
1,1,2,2-Tetrachloroethane	5	ug/kg	0.61	ug/kg	19990104	20	ug/kg	46	157	24	50	ug/kg	46	157	24
Tetrachloroethene	5	ug/kg	0.29	ug/kg	19990104	20	ug/kg	68	136	22	50	ug/kg	39	154	22
Tetrachloroethylene	5	ug/kg	0.29	ug/kg	19990104										
Tetrahydrofuran	10	mg/kg	1.5	mg/kg	19990104										
Toluene	5	ug/kg	0.43	ug/kg	19990104	20	ug/kg	71	130	24	50	ug/kg	46	147	24
1,2,3-Trichlorobenzene	5	ug/kg	0.33	ug/kg	19990105										
1,2,4-Trichlorobenzene	5	ug/kg	0.42	ug/kg	19990104										
1,1,1-Trichloroethane	5	ug/kg	0.16	ug/kg	19990104	20	ug/kg	52	162	12	50	ug/kg	52	162	12
1,1,2-Trichloroethane	5	ug/kg	0.67	ug/kg	19990105	20	ug/kg	52	150	19	50	ug/kg	52	150	19
Trichloroethene	5	ug/kg	0.18	ug/kg	19990104	20	ug/kg	70	131	23	50	ug/kg	46	143	23
Trichloroethylene	5	ug/kg	0.18	ug/kg	19990104										

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: PURGE AND TRAP - Lab Preserved Low Level - Solids
Method: Volatile Organics, GC/MS (8260B)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits				Check List 924					Spike List 925				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Trichlorofluoromethane	10	ug/kg	0.21	ug/kg	19990105										
1,2,3-Trichloropropane	5	ug/kg	0.88	ug/kg	19990104										
1,1,2-Trichloro-1,2,2-trifluoroethane	5	ug/kg	0.28	ug/kg	19990104										
1,2,4-Trimethylbenzene	5	ug/kg	0.21	ug/kg	19990105										
1,3,5-Trimethylbenzene	5	ug/kg	0.16	ug/kg	19990105										
Vinyl acetate	10	ug/kg	2.3	ug/kg	19990104										
Vinyl chloride	10	ug/kg	0.28	ug/kg	19990105	20	ug/kg	41	138	43	50	ug/kg	29	150	43
m-Xylene & p-Xylene	5.0	ug/kg	0.64	ug/kg	19990104										
o-Xylene	2.5	ug/kg	0.2	ug/kg	19990105										
Xylenes (total)	10	ug/kg	0.93	ug/kg	19990104										
4-Bromofluorobenzene						60	ug/kg	83	129	10	150	ug/kg	83	129	10
1,2-Dichloroethane-d4						50	ug/kg	47	158	0	50	ug/kg	47	158	0
Toluene-d8						50	ug/kg	61	130	0	50	ug/kg	61	130	0
Dibromofluoromethane						50	ug/kg	60	143	0	50	ug/kg	60	143	0
						50	ug/kg	59	138	0	50	ug/kg	59	138	0

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: SONICATION - Low Level
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List	Detection Limits					Check List 1114					Spike List 1115				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
a,a-Dimethylphenethylamine	1600	ug/kg	210	ug/kg	19990412										
Acenaphthene	330	ug/kg	45	ug/kg	19990120	1700	ug/kg	44	108	44	1700	ug/kg	13	133	44
Acenaphthylene	330	ug/kg	37	ug/kg	19990120			48	107	36			35	111	36
Acetophenone	330	ug/kg	320	ug/kg	19990422										
2-Acetylaminofluorene	3300	ug/kg	56	ug/kg	19990121										
4-Aminobiphenyl	1600	ug/kg	55	ug/kg	19990202										
Aniline	330	ug/kg	130	ug/kg	19990619										
Anthracene	330	ug/kg	37	ug/kg	19990120			49	114	38			35	115	38
Aramite	660	ug/kg	40	ug/kg	19990202										
Atrazine	330	ug/kg	18	ug/kg	20000218										
Benzaldehyde	330	ug/kg	80	ug/kg	20000218										
Benz(a)anthracene	330	ug/kg	45	ug/kg	19990120										
Benzenethiol	330	ug/kg	25	ug/kg	19990629										
Benzidine	3300	ug/kg	640	ug/kg	19990413										
Benzo(a)anthracene	330	ug/kg	45	ug/kg	19990120			49	116	35			30	122	35
Benzo(b)fluoranthene	330	ug/kg	52	ug/kg	19990120			44	117	38			28	121	38
Benzo(k)fluoranthene	330	ug/kg	32	ug/kg	19990120			43	116	36			28	121	36
Benzoic acid	1600	ug/kg	56	ug/kg	19980122										
Benzo(ghi)perylene	330	ug/kg	41	ug/kg	19990128			44	121	38			17	126	38
Benzo(a)pyrene	330	ug/kg	37	ug/kg	19990120			46	109	36			26	114	36
Benzothiazole	0.33	mg/kg	77	ug/kg	19980518										
Benzyl Mercaptan	0.33	mg/kg	110	ug/kg	19980518										
o-Benzyl Phenol	0.33	mg/kg	76	ug/kg	19980518										
Benzyl alcohol	330	ug/kg	53	ug/kg	19990120										
1,1'-Biphenyl	330	ug/kg	17	ug/kg	20000218										
bis(2-Chloroethoxy)methane	330	ug/kg	40	ug/kg	19990120			42	109	40			34	109	40
bis(2-Chloroethyl) ether	330	ug/kg	69	ug/kg	19990120			45	100	52			29	104	52
bis(2-Chloroisopropyl) ether	330	ug/kg	39	ug/kg	19990120			51	132	38			36	135	38
bis(2-Chloro-1-methylethyl) ether	330	ug/kg	39	ug/kg	19990120										
bis(2-Ethylhexyl) phthalate	330	ug/kg	37	ug/kg	19990120			44	125	38			21	130	38
4-Bromophenyl phenyl ether	330	ug/kg	39	ug/kg	19990120			45	120	38			33	124	38
2,3-Butanedithiol	0.33	mg/kg	240	ug/kg	19980518										
1,4-Butanedithiol	0.33	mg/kg	110	ug/kg	19980518										
Butyl benzyl phthalate	330	ug/kg	39	ug/kg	19990120			46	111	37			26	119	37
Caprolactam	330	ug/kg	26	ug/kg	20000218										
Carbazole	330	ug/kg	42	ug/kg	19990120			48	133	37			38	126	37

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: SONICATION - Low Level
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits				Check List 1114					Spike List 1115				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
p-Chloroaniline	330	ug/kg	66	ug/kg	19990120										
4-Chloroaniline	330	ug/kg	66	ug/kg	19990120			14	70	67			10	73	67
Chlorobenzilate	330	ug/kg	22	ug/kg	19990202										
4-Chloro-3-methylphenol	330	ug/kg	50	ug/kg	19990120	2500	ug/kg	43	110	55	2500	ug/kg	17	128	55
p-Chloro-m-cresol	330	ug/kg	50	ug/kg	19990120										
2-Chloronaphthalene	330	ug/kg	37	ug/kg	19990120			49	111	36			37	114	36
2-Chlorophenol	330	ug/kg	41	ug/kg	19990120	2500	ug/kg	43	110	54	2500	ug/kg	17	116	54
4-Chlorophenyl phenyl ether	330	ug/kg	44	ug/kg	19990120			47	120	36			33	128	36
Chrysene	330	ug/kg	36	ug/kg	19990120			53	115	36			28	126	36
6-Methylchrysene	330	ug/kg	9.4	ug/kg	19990629										
m-Cresol	330	ug/kg	320	ug/kg	19990422										
p-Cresol	330	ug/kg	33	ug/kg	19990120										
m-Cresol & p-Cresol	660	ug/kg			0										
Cresols (total)	330	ug/kg	34	ug/kg	19990120										
Cyclohexylamine	1600	ug/kg	640	ug/kg	19980521										
Diallate	660	ug/kg	20	ug/kg	19990121										
Dibenz(a,h)acridine	330	ug/kg	7.6	ug/kg	19990629										
Dibenz(a,j)acridine	330	ug/kg	7.6	ug/kg	19990629										
Dibenz(a,h)anthracene	330	ug/kg	55	ug/kg	19990128			49	119	37			26	123	37
Dibenzofuran	330	ug/kg	49	ug/kg	19990120			48	113	36			36	119	36
1,3-Dibutyl-2-Thiourea	0.33	mg/kg	98	ug/kg	19980518										
Di-n-butyl phthalate	330	ug/kg	43	ug/kg	19990120			48	115	36			33	117	36
1,2-Dichlorobenzene	330	ug/kg	36	ug/kg	19990120			49	100	40			32	104	40
o-Dichlorobenzene	330	ug/kg	36	ug/kg	19990120										
1,3-Dichlorobenzene	330	ug/kg	35	ug/kg	19990120			47	96	44			29	99	44
m-Dichlorobenzene	330	ug/kg	35	ug/kg	19990120										
1,4-Dichlorobenzene	330	ug/kg	35	ug/kg	19990120	1700	ug/kg	38	100	59	1700	ug/kg	18	110	59
p-Dichlorobenzene	330	ug/kg	35	ug/kg	19990120										
3,3'-Dichlorobenzidine	1600	ug/kg	44	ug/kg	19990128			15	80	79			10	76	79
2,4-Dichlorophenol	330	ug/kg	43	ug/kg	19990120			46	111	42			31	120	42
2,6-Dichlorophenol	330	ug/kg	27	ug/kg	19990121										
1,3-Diethyl-2-Thiourea	0.33	mg/kg	80	ug/kg	19980518										
Diethyl phthalate	330	ug/kg	51	ug/kg	19990120			47	116	36			32	118	36
O,O-Diethyl-O-(2-pyrazinyl) phosphoro	1600	ug/kg	23	ug/kg	19990121										
2,3-Dimercapto-1-Propanol	0.33	mg/kg	0.33	mg/kg	19991202										
Dimethoate	660	ug/kg	390	ug/kg	19990422										
1,3-Dimethyl-2-Thiourea	0.33	mg/kg	81	ug/kg	19980518										
p-Dimethylaminoazobenzene	660	ug/kg	25	ug/kg	19990121										
7,12-Dimethylbenz(a)anthracene	660	ug/kg	36	ug/kg	19990202										

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: SONICATION - Low Level
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits				Check List 1114					Spike List 1115				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
3,3'-Dimethylbenzidine	1600	ug/kg	160	ug/kg	19990121										
2,4-Dimethylphenol	330	ug/kg	45	ug/kg	19990120			34	108	56			18	118	56
Dimethyl phthalate	330	ug/kg	39	ug/kg	19990120			48	118	38			34	120	38
1,2-Dinitrobenzene	330	ug/kg	57	ug/kg	19990120										
m-Dinitrobenzene	330	ug/kg	28	ug/kg	19990121										
1,3-Dinitrobenzene	330	ug/kg	28	ug/kg	19990121										
p-Dinitrobenzene	330	ug/kg	32	ug/kg	19990121										
1,4-Dinitrobenzene	330	ug/kg	32	ug/kg	19990121										
4,6-Dinitro-2-methylphenol	1600	ug/kg	46	ug/kg	19990128			25	137	45			13	126	45
4,6-Dinitro-o-cresol	1600	ug/kg	46	ug/kg	19990128										
2,4-Dinitrophenol	1600	ug/kg	71	ug/kg	19980311			10	143	50			10	141	50
2,4-Dinitrotoluene	330	ug/kg	46	ug/kg	19990120	1700	ug/kg	48	111	45	1700	ug/kg	10	171	45
2,6-Dinitrotoluene	330	ug/kg	44	ug/kg	19990120			50	122	38			36	123	38
Dinoseb	660	ug/kg	470	ug/kg	19990422										
2-sec-Butyl-4,6-dinitrophenol	660	ug/kg	470	ug/kg	19990422										
Di-n-octyl phthalate	330	ug/kg	37	ug/kg	19990120			40	121	39			21	130	39
1,4-Dioxane	330	ug/kg	22	ug/kg	19990121										
Diphenyl Thiourea	0.33	mg/kg	71	ug/kg	19980518										
Diphenylamine	330	ug/kg	44	ug/kg	19990120										
1,2-Diphenylhydrazine	330	ug/kg	36	ug/kg	19990120										
Disulfoton	1600	ug/kg	20	ug/kg	19990202										
1,2-Ethanedithiol	1.6	mg/kg	310	ug/kg	19980518										
Ethyl methanesulfonate	330	ug/kg	17	ug/kg	19990202										
Famphur	3300	ug/kg	640	ug/kg	19990412										
Fluoranthene	330	ug/kg	39	ug/kg	19990120			46	124	42			24	138	42
Fluorene	330	ug/kg	44	ug/kg	19990120			48	114	37			32	123	37
Hexachlorobenzene	330	ug/kg	38	ug/kg	19990120			44	126	29			39	127	29
Hexachlorobutadiene	330	ug/kg	38	ug/kg	19990120			36	110	41			31	110	41
Hexachlorocyclopentadiene	1600	ug/kg	26	ug/kg	19980122			10	126	90			10	102	90
Hexachloroethane	330	ug/kg	35	ug/kg	19990128			30	110	40			23	110	40
Hexachloropropene	3300	ug/kg	32	ug/kg	19990121										
Hexamethylphosphoramide	0.02	mg/kg	0.02	mg/kg	19991202										
Indene	330	ug/kg	39	ug/kg	19990629										
Indeno(1,2,3-cd)pyrene	330	ug/kg	51	ug/kg	19990128			47	125	41			27	123	41
Isophorone	330	ug/kg	39	ug/kg	19990120			41	102	37			27	107	37
Isosafrole	660	ug/kg	120	ug/kg	19980226										
Methapyrilene	1600	ug/kg	31	ug/kg	19990121										
3-Methylcholanthrene	660	ug/kg	19	ug/kg	19990202										
Methyl methanesulfonate	330	ug/kg	88	ug/kg	19980226										

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: SONICATION - Low Level
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List	Detection Limits					Check List 1114					Spike List 1115				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
2-Methylnaphthalene	330	ug/kg	37	ug/kg	19990120			46	105	43			33	112	43
1-Methylnaphthalene	330	ug/kg	37	ug/kg	19990120										
Methyl parathion	1600	ug/kg	NA	ug/kg	19950301										
2-Methylphenol	330	ug/kg	38	ug/kg	19990128			41	102	39			33	113	39
o-Cresol	330	ug/kg	38	ug/kg	19990128										
3-Methylphenol	330	ug/kg	320	ug/kg	19990422										
4-Methylphenol	330	ug/kg	33	ug/kg	19990120			40	110	34			33	118	34
3-Methylphenol & 4-Methylphenol	660	ug/kg			0										
Methylphenols (total)	330	ug/kg	34	ug/kg	19990120										
Naphthalene	330	ug/kg	39	ug/kg	19990120			48	101	38			34	107	38
1,4-Naphthoquinone	1600	ug/kg	350	ug/kg	19990422										
1-Naphthylamine	330	ug/kg	33	ug/kg	19990121										
2-Naphthylamine	330	ug/kg	47	ug/kg	19990221										
2-Nitroaniline	1600	ug/kg	38	ug/kg	19990120			45	123	33			30	124	33
o-Nitroaniline	1600	ug/kg	38	ug/kg	19990120										
3-Nitroaniline	1600	ug/kg	32	ug/kg	19990120			26	119	53			10	105	53
m-Nitroaniline	1600	ug/kg	32	ug/kg	19990120										
4-Nitroaniline	1600	ug/kg	55	ug/kg	19990128			34	122	69			10	105	69
p-Nitroaniline	1600	ug/kg	55	ug/kg	19990128										
Nitrobenzene	330	ug/kg	37	ug/kg	19990120			35	112	36			33	112	36
2-Nitrophenol	330	ug/kg	39	ug/kg	19990120			43	110	39			29	112	39
o-Nitrophenol	330	ug/kg	39	ug/kg	19990120										
4-Nitrophenol	1600	ug/kg	52	ug/kg	19990120	2500	ug/kg	22	128	64	2500	ug/kg	10	148	64
p-Nitrophenol	1600	ug/kg	52	ug/kg	19990120										
4-Nitroquinoline-1-oxide	3300	ug/kg	150	ug/kg	19990412										
N-Nitrosodi-n-butylamine	330	ug/kg	24	ug/kg	19990202										
N-Nitrosodiethylamine	330	ug/kg	23	ug/kg	19990202										
N-Nitrosodimethylamine	330	ug/kg	32	ug/kg	19990120										
N-Nitrosodiphenylamine	330	ug/kg	40	ug/kg	19990120			50	118	42			35	118	42
N-Nitrosodi-n-propylamine	330	ug/kg	35	ug/kg	19990120	1700	ug/kg	38	110	50	1700	ug/kg	12	128	50
N-Nitrosomethylethylamine	330	ug/kg	22	ug/kg	19990202										
N-Nitrosomorpholine	330	ug/kg	300	ug/kg	19990422										
N-Nitrosopiperidine	330	ug/kg	30	ug/kg	19990202										
N-Nitrosopyrrolidine	330	ug/kg	22	ug/kg	19990121										
5-Nitro-o-toluidine	660	ug/kg	320	ug/kg	19990422										
2,2'-oxybis(1-Chloropropane)	330	ug/kg	39	ug/kg	19990120										
Pentachlorobenzene	330	ug/kg	24	ug/kg	19990202										
Pentachloroethane	1600	ug/kg	21	ug/kg	19990202										
Pentachloronitrobenzene	1600	ug/kg	29	ug/kg	19990202										

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: SONICATION - Low Level
Method: Base/Neutrals and Acids (8270C)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits				Check List 1114					Spike List 1115				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Pentachlorophenol	330	ug/kg	44	ug/kg	19990120	2500	ug/kg	10	123	87	2500	ug/kg	10	144	87
Phenacetin	660	ug/kg	35	ug/kg	19990121										
Phenanthrene	330	ug/kg	39	ug/kg	19990120			50	117	39			32	126	39
Phenol	330	ug/kg	51	ug/kg	19990120	2500	ug/kg	35	110	50	2500	ug/kg	10	148	50
p-Phenylene diamine	3300	ug/kg	200	ug/kg	19990412										
Phenyl ether	0.33	mg/kg	30	ug/kg	19990322										
Phorate	1600	ug/kg	380	ug/kg	19990422										
2-Picoline	660	ug/kg	26	ug/kg	19990121										
Pronamide	660	ug/kg	370	ug/kg	19990422										
1,3-Propanedithiol	0.33	mg/kg	140	ug/kg	19980518										
2,2'-Oxybis(1-Chloropropane)	330	ug/kg	39	ug/kg	19990120										
Pyrene	330	ug/kg	41	ug/kg	19990120	1700	ug/kg	42	122	66	1700	ug/kg	10	218	66
Pyridine	660	ug/kg	65	ug/kg	19990128										
Quinoline	330	ug/kg	23	ug/kg	19990322										
Safrole	660	ug/kg	21	ug/kg	19990121										
Sulfotepp	1600	ug/kg	350	ug/kg	19990422										
1,2,4,5-Tetrachlorobenzene	330	ug/kg	310	ug/kg	19990422										
2,3,4,6-Tetrachlorophenol	1600	ug/kg	44	ug/kg	19990121										
2,3,5,6-Tetrachlorophenol	1600	ug/kg	59	ug/kg	19990120										
Tetraethyldithiopyrophosphate	1600	ug/kg	350	ug/kg	19990422										
1,1,3,3-Tetramethyl-2-Thiourea	0.33	mg/kg	76	ug/kg	19980518										
Thionazin	1600	ug/kg	23	ug/kg	19990121										
Thiourea	1600	ug/kg	670	ug/kg	19980518										
o-Toluidine	660	ug/kg	95	ug/kg	19990121										
1,2,4-Trichlorobenzene	330	ug/kg	34	ug/kg	19990120	1700	ug/kg	45	110	54	1700	ug/kg	16	121	54
2,4,5-Trichlorophenol	330	ug/kg	63	ug/kg	19990120			39	117	29			29	125	29
2,4,6-Trichlorophenol	330	ug/kg	63	ug/kg	19990120			40	110	60			21	126	60
O,O,O-Triethyl phosphorothioate	1600	ug/kg	22	ug/kg	19990202										
sym-Trinitrobenzene	1600	ug/kg	31	ug/kg	19990202										
1,3,5-Trinitrobenzene	1600	ug/kg	31	ug/kg	19990202										
2-Fluorobiphenyl						1660	ug/kg	43	110	0	1660	ug/kg	43	110	0
2-Fluorophenol						2500	ug/kg	11	116	0	2500	ug/kg	11	116	0
2,4,6-Tribromophenol						2500	ug/kg	35	116	0	2500	ug/kg	35	116	0
Nitrobenzene-d5						1660	ug/kg	42	110	0	1660	ug/kg	42	110	0
Phenol-d5						1500	ug/kg	25	115	0	2500	ug/kg	25	115	0
Terphenyl-d14						1660	ug/kg	37	137	0	1660	ug/kg	37	137	0

STL Reference Data Summary

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: SONICATION w/ACID STRIP (PCB)
Method: PCBs (8082)
QC Program: STANDARD TEST SET
Location: STL North Canton

Analyte List		Detection Limits				Check List 928					Spike List 929				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
PCB-1016	33	ug/kg	15	ug/kg	19990405										
Aroclor 1016	33	ug/kg	15	ug/kg	19990405	333	ug/kg	49	122	39	333	ug/kg	26	144	39
PCB-1221	33	ug/kg	19	ug/kg	19990405										
Aroclor 1221	33	ug/kg	19	ug/kg	19990405										
PCB-1232	33	ug/kg	20	ug/kg	19990405										
Aroclor 1232	33	ug/kg	20	ug/kg	19990405										
PCB-1242	33	ug/kg	24	ug/kg	19990405										
Aroclor 1242	33	ug/kg	24	ug/kg	19990405										
PCB-1248	33	ug/kg	8.1	ug/kg	19990405										
Aroclor 1248	33	ug/kg	8.1	ug/kg	19990405										
PCB-1254	33	ug/kg	20	ug/kg	19990405										
Aroclor 1254	33	ug/kg	20	ug/kg	19990405										
PCB-1260	33	ug/kg	14	ug/kg	19990405										
Aroclor 1260	33	ug/kg	14	ug/kg	19990405	333	ug/kg	51	127	33	333	ug/kg	37	138	33
PCB-1262	33	ug/kg	33	ug/kg	19991207										
Aroclor 1262	33	ug/kg	33	ug/kg	19991207										
PCB-1268	33	ug/kg	33	ug/kg	19991207										
Aroclor 1268	33	ug/kg	33	ug/kg	19991207										
PCBs (total)	1000	ug/kg	1000	ug/kg	19991207										
Aroclors (Total)	1000	ug/kg	1000	ug/kg	19991207										
Decachlorobiphenyl						6.67	ug/kg	23	141	0	6.67	ug/kg	23	141	0
Tetrachloro-m-xylene						6.67	ug/kg	31	127	0	6.67	ug/kg	31	127	0

APPENDIX D

DATA MANAGEMENT PLAN

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1.0 INTRODUCTION

This Data Management Plan (DMP) is submitted as an appendix to and forms part of the RCRA Facility Investigation (RFI) Work Plan prepared by Remediation and Liability Management Company, Inc. (REALM) for the Former Peregrine (US) Inc. Coldwater Road Facility (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 has been developed by Conestoga-Rovers & Associates (CRA) and 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 CRA possesses. These two tasks will be expanded upon in the following sections.

Mr. Glenn Turchan, the Project Manager for CRA, will be responsible for all aspects of this DMP.

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 data base. 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; and

viii) field decisions made and pertinent information associated with the decision.

The field logbooks reviewed will be numbered consecutively and maintained in accordance with the project QAPP (see Appendix C).

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 3 1/2-inch floppy discs in addition to the original laboratory reports. In all cases where Facility data is 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, 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

CRA's 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 data will be presented in its raw format as an appendix to the RFI Report. Summary tables of the raw analytical data will be developed to present the data in a useable format. Analytical data will be manipulated using CRA's data base 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 the data. Only personnel actively involved in the project will be permitted access to the data. The data will be accessed in accordance with CRA'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.

In addition to the document file maintained by CRA, a file of related documents (i.e., RFI Work Plan, analytical data) will be maintained by REALM.

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 MDEQ-WMD; 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 may consist of the following:

i) field logbooks;

ii) analytical reports;

iii) chain-of-custody forms;

iv) MDEQ-WMD 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 may include reports generated during the RFI and forwarded to MDEQ-WMD. These documents will be included in the document control system.

Documents will be maintained for a minimum of 6 years.

APPENDIX E

HEALTH AND SAFETY PLAN

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ATTACHMENT E.1 TRAINING ACKNOWLEDGMENT FORM

1.0 GENERAL

The RCRA Facility Investigation (RFI) program to be implemented, as presented in the RFI Work Plan, will involve drilling and sampling within limits of the Facility. During these operations, personnel may come in contact with potentially hazardous materials. This Facility-specific Health and Safety Plan (HASP) has been developed to ensure the following:

i) that Facility personnel are not adversely exposed to the compounds of concern;

ii) that public welfare or the environment are not adversely impacted by off-Facility migration of contaminated materials due to work activities at the Facility; and

iii) compliance with applicable governmental (Title 29 Code of Federal Regulations (CFR) Part 1910.120) and non-governmental (American Conference of Governmental Industrial Hygienist) regulations and guidelines will be implemented for all Facility work.

For the purpose of this HASP, all sampling and investigative activities carried out during the RFI at the Facility involving contact with potentially contaminated materials will be considered contaminated operations requiring personal protective equipment (PPE). A detailed description of the PPE required is presented in Section 9.0.

All sampling and investigative operations at the Facility will be conducted in accordance with the provisions of this HASP. Subcontractors performing work at the Facility will be responsible for providing a Facility-specific HASP to address hazards associated with the work they are performing. As a minimum, subcontractor HASPs are to be in accordance with the provisions of this HASP. Cost and/or scheduling considerations will not be considered as justification for modifying this HASP.

2.0 FACILITY CHARACTERIZATION AND HAZARD ANALYSIS AND POTENTIALLY HAZARDOUS COMPOUNDS

2.1 FACILITY CHARACTERIZATION

2.1.1 FACILITY BACKGROUND

The Site is located is located at G1245 East Coldwater Road, Genesee Township, Michigan. The plant was used as a plating facility and a stamping plant that produced interior components for cars and light trucks, principally door hinges and window regulators. Operations at the facility began in 1953. In 1996 the plant and its operations were sold to Peregrine, Inc. Operations at the Site ceased in 1998. General Motors Corporation subsidiary Remediation and Liability Management Company, Inc. (REALM) has reacquired the property and the plant and is in the process of decommissioning and demolishing the manufacturing building. The Site location is shown on Figure E.2.1.

The manufacturing facility is comprised of six main structures totaling approximately 1.97 million square feet:

- a manufacturing building (1.75 million square feet);
- an administration building (68,400 square feet);
- a storage building (69,000 square feet); and
- a gas and coal-fired power house (33,200 square feet).

Potentially hazardous materials that may be present on Site include asbestos, metals, polychlorinated biphenyls (PCBs), waste oil, heavy metals, acid salts, cyanide, and corrosives. A summary of the contaminants of concern and their related health effects are presented in Table E.1.

The scope of work (SOW) to be performed is limited to activities related to sampling. Activities to be performed as part of the SOW may include:

- sampling activities; and
- surveying activities.

TABLE E.1

FACILITY HAZARD ANALYSIS
RFI WORK PLAN
FORMER PEREGRINE (US) INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Site Activities	*Hazards*	*Prevention*
Drilling	• Contact with Chemicals of Concern	• Level D, Level C PPE (contingency level)
		• Upgrade to level C if exposed to dust
	• Overhead Hazards/Underground Utilities	• Be alert to current site activities and obtain clearance prior to intrusive field work
		• Perform an underground utilities search
	• Slip, Trip, and Fall	• Be alert to current site conditions
	• Cold Stress	• Wear appropriate clothing for the ambient temperature
Monitoring Well Installation	• Contact with Chemicals of Concern	• Level D, Level C PPE (contingency level)
	• Overhead Hazards	• Be alert to current site activities
	• Slip, Trip, and Fall	• Be alert to current site conditions
	• Cold Stress	• Wear appropriate clothing for the ambient temperature
Soil Sampling	• Contact with Chemicals of Concern	• Level D, or Level C PPE
		• Upgrade to level C if exposed to dust
	• Overhead Hazards	• Be alert to current site activities
	• Slip, Trip, and Fall	• Be alert to current site conditions
	• Cold Stress	• Wear appropriate clothing for the ambient temperature
Groundwater Sampling	• Contact with Chemicals of Concern	• Level D, or Level C PPE
	• Slip, Trip, and Fall	• Be alert to current site activities
	• Cold Stress	• Wear appropriate clothing for the ambient temperature
Sediment Sampling	• Contact with Chemicals of Concern	• Level D, Level C PPE (contingency level)
		• Upgrade to level C if exposed to dust
	• Slip, Trip, and Fall	• Be alert to current site activities
	• Cold Stress	• Wear appropriate clothing for the ambient temperature

TABLE E.2

TOXICITY PROFILE FOR SELECTED HAZARDOUS SUBSTANCES ON-SITE
FORMER PEREGRINE (US) INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Contaminant (synonym)	*Physical/Chemical Characteristics (target organs/route of entry)*	*OSHA PEL*	*ACGIH TLV*	*NIOSH REL*	*IDLH*	*Ionization Potential*
Asbestos	Appearance and Odor: White or greenish, blue, or gray-green fibrous, odorless solids. Target Organs: Respiratory system. Primary Entry Routes: Inhalation, ingestion.	TWA: 0.1 f/cm^3	TWA: 0.1 f/cm^3	TWA: 0.1 f/cm^3	1 f/cm^3	NA
Barium	Appearance and Odor: White odorless solids. Target Organs: Eyes, skin, respiratory system, heart, CNS. Primary Entry Routes: Inhalation, skin and/or eye contact, ingestion.	TWA: 0.5 mg/m^3	TWA: 0.5 mg/m^3	TWA: 0.5 mg/m3	50 mg/m^3	NA
Cadmium	Appearance and Odor: silver-white blue tinged metal dust, odorless solid Target Organs: Respiratory system, kidneys Primary Entry Routes: Inhalation, ingestion.	TWA: 0.005 mg/m^3	TWA: 0.002 mg/m^3	NA	9 mg/m^3	NA
Chromium	Appearance and Odor: blue-white to steel greylustrous,brittle, hard odorless solid Target Organs: Eyes, Skin, Respiratory system Primary Entry Routes: Inhalation, ingestion, skin and/or eye contact.	TWA: 1 mg/m^3	TWA: 0.5 mg/m^3	TWA: 0.5 mg/m^3	250 mg/m^3	NA
Cyanide	CNS, irritation, anoxia, lung, thyroid	TWA: 5 mg/m^3	TWA: 5 mg/m^3	TWA:5 mg/m^3	25 mg/m^3	13.6 ev
Lead	Appearance and Odor: Heavy, ductile, soft, gray, solid. Target Organs: Eyes, GI tract, CNS, kidneys, blood, gingival tissue. Primary Entry Routes: Inhalation, ingestion, contact.	TWA: 0.05 mg/m^3	TWA: 0.05 mg/m^3	TWA: 0.10 mg/m^3	100 mg/m^3	
Polychlorinated biphenyls (PCBs)	Appearance and Odor: Colorless to pale-yellow, viscous liquid or solid (below 50 degrees F) with a mild, hydrocarbon odor. Target Organs: Skin, eyes, liver, and reproductive system. Primary Entry Routes: Inhalation, ingestion, contact.	TWA: 0.5 mg/m^3	NA	TWA: 0.001 mg/m^3	5 mg/m^3	NA
Trichloroethylene (TCE)	Appearance and Odor: Colorless liquid with a chloroform odor. Target Organs: Skin, eyes, respiratory system, heart, liver and kidneys. Primary Entry Routes: Inhalation, absorption,ingestion, contact.	TWA: 100 ppm	50 ppm	NA	1000 ppm	9.45ev

Risks associated with these activities will be minimized by implementing engineering controls, safe work practices, and the proper use of personal protective equipment. Table E.2 summarizes the potential hazards associated with activities at the Site.

2.1.2 CURRENT OPERATIONS

The Facility is undergoing a complete and systematic environmental decommissioning and demolition program that began during the summer of 1999 and is expected to be completed in 2001. The Facility location is presented on Figure E.1.

2.2 HAZARD ANALYSIS

The RFI activities proposed in the RFI Work Plan consist of drilling and sampling. The hazards associated with these activities are summarized in Table E.1.

2.3 POTENTIALLY HAZARDOUS COMPOUNDS

Potential hazards associated with the Facility include heavy equipment and residual chemical constituents from plant operations.

Table E.2 lists selected hazardous substances that are known or suspected to be present on Site based on laboratory analyses of samples obtained during previous Facility investigations. The Immediately Dangerous to Life and Health (IDLH) limits, Threshold Limit Value (TLV), and Permissible Exposure Limits (PELs) for these compounds are also presented in Table E.2.

2.4 CHEMICAL EXPOSURE

Preventing exposure to toxic chemicals is a primary concern. Chemical substances may enter the unprotected body by inhalation, skin absorption, ingestion, or through a injection (puncture wound). A contaminant may cause damage at the point of contact or may act systematically, causing a toxic effect in a part of the body distant from the point of initial contact.

Chemical exposures are generally divided into two categories, acute and chronic. Symptoms resulting from acute exposures usually occur during or shortly after

exposure to a sufficiently high concentration of a contaminant. The concentration required to produce such effects varies widely from chemical to chemical. The term "chronic exposure" generally refers to exposures to "low" concentrations of a contaminant over a long period of time. The "low" concentrations required to produce symptoms of chronic exposure depend upon the chemical, the duration of each exposure, and the number of exposures. For a given contaminant, the symptoms of an acute exposure may be completely different from those resulting from chronic exposure.

For either chronic or acute exposure, the toxic effect may be temporary and reversible, or may be permanent (disability or death). For example, acute exposure effects associated with exposure to high concentrations of chromium include severe eye and skin burns. Chronic inhalation of dust/mists may cause irritation to respirator tract, nasal septum drainage and asthmatic bronchitis. Liver and kidney drainage may also occur.

The effects of exposure not only depend on the chemical, its concentration, route of entry, and duration of exposure, but may also be influenced by personal factors such as the individual's smoking habits, alcohol consumption, medication use, nutrition, age, and sex.

An important exposure route of concern at the Facility is inhalation. The lungs are extremely vulnerable to chemical agents. Even substances that do not directly affect the lungs may pass through lung tissue into the bloodstream, where they are transported to other vulnerable areas of the body. Some toxic chemicals present in the atmosphere may not be detected by human senses (i.e., they may be colorless, odorless, and their toxic effects may not produce any immediate symptoms). Respiratory protection is therefore extremely important if there is a possibility that the work site atmosphere may contain hazardous substances. Chemicals may also enter the respiratory tract through punctured eardrums. Where this is a hazard, individuals with punctured eardrums should be medically evaluated specifically to determine if such a condition would place them at an unacceptable risk and preclude their working at the task in question.

Direct contact of the skin and eyes with hazardous substances is another important route of exposure. Some chemicals directly injure the skin and some pass through the skin into the bloodstream where they are transported to vulnerable organs. Skin absorption is enhanced by abrasions, cuts, heat, and moisture. The eye is particularly vulnerable because airborne chemicals can dissolve in its moist surface and be carried to the rest of the body through the bloodstream (capillaries are very close to the surface of the eye). Wearing protective equipment, not using contact lenses in contaminated

atmospheres (since they may trap chemicals against the eye surface), keeping hands away from the face, and minimizing contact with liquid and solid chemicals will help protect against skin and eye contact.

Although ingestion should be the least significant route of exposure at the Facility, it is important to be aware of how this type of exposure occurs. Deliberate ingestion of chemicals is unlikely, however, personal habits such as chewing gum or tobacco, drinking, eating, smoking cigarettes, and applying cosmetics may provide a route of entry for chemicals.

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) and current Threshold Limit Values (TLVs) as provided by the American Conference of Governmental Industrial Hygienists (ACGIH) provide the basis for this HASP. Some of the specifications within this HASP are in addition to OSHA regulations and reflect the positions of the United States Environmental Protection Agency (U.S. EPA), and the National Institute for Occupational Safety and Health (NIOSH) regarding procedures required to ensure safe operations at potential hazardous waste sites.

The safety and health of the public, Facility personnel, and the protection of the environment will take precedence over cost and schedule considerations for all project work. Conestoga-Rovers & Associates (CRA) will designate an on-Facility individual as Site Health and Safety Officer (HSO). The HSO will be responsible for decisions regarding when work will be stopped or started for health or safety considerations.

4.0 RESPONSIBILITIES AND ADMINISTRATION

The HSO will supervise the implementation of this 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 Site access;

ii) be responsible for implementation of this HASP at the initiation of Facility work;

iii) conduct the initial briefing sessions for all on-Facility personnel involved with investigative activities with regard to this HASP and other safety requirements to be observed during field activities, including:

   a) potential hazards,

   b) personal hygiene principles,

   c) personal protective equipment,

   d) respiratory protection equipment usage, and

   e) emergency procedures dealing with fire and medical situations;

iv) review and modify this HASP as more information becomes available concerning the hazardous materials involved, and review all monitoring reports;

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) maintain the on-Facility hazard communication program and inform workers of the nature of chemical exposure risk as required by the "Right-to-Know" Law;

xi) recommend medical examination when worker appears to require it;

xii) coordination of Emergency Response Plan;

xiii) assure that safety equipment is provided, maintained, and accessible to Site personnel;

xiv) maintain a log with a sign in/out sheet for personnel performing monitoring activities and visitors entering the Site;

xv) assure that employees comply with the "buddy system" while working on Site;

xvi) maintain the temporary exclusion zone (EZ) and contaminant reduction zone (CRZ) work areas; and

xvii) assure that all utilities are cleared.

5.0 FACILITY HEALTH AND SAFETY PLAN

This Facility HASP will be executed for work performed at the Facility. A copy of this plan will be maintained at the Facility at all times and available to all Facility employees and visitors.

As previously mentioned, subcontractors will be responsible for providing a Facility-specific HASP in accordance with provisions of the HASP. A copy of the subcontractor's HASP must also be maintained at the Facility.

6.0 MEDICAL SURVEILLANCE

In accordance with requirements detailed in 29 CFR 1910.120 and 29 CFR 1910.134, all Facility personnel who will come in contact with potentially contaminated materials will have received, within 1 year prior to starting field activities, medical surveillance by a licensed physician or physician's group.

Medical records for all on-Facility sampling personnel will be maintained by their respective employers. 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.

The medical records will be available to the employee or his designated representative upon written request, as outlined in 29 CFR 1910.20.

Each employer will ensure that its personnel involved in on-Facility 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. Proof of medical certification will be provided to CRA's HSO prior to commencement of work.

Interim medical surveillance will be completed if an individual exhibits poor health or high stress responses due to on-Facility activity or when accidental exposure to elevated concentrations of contaminants occurs.

7.0 TRAINING

Prior to entering the Site, all personnel performing RFI activities will have completed training sessions in accordance with 29 CFR 1910.120. This training shall consist of a minimum of 40 hours of instruction and 3 days of actual field experience under direct supervision of a trained, experienced supervisor. Each employer will maintain documentation stating that its on-Facility personnel have complied with this regulation. Each contractor will be required to maintain a HASP meeting the requirements of this HASP, as a minimum.

Prior to commencing Facility activities, a Facility-specific initiation session will be conducted. This session will be used to instruct the on-Facility personnel as to the potential Facility hazards, level of PPE required, Facility-specific requirements, and the basis of the Health and Safety Plan. Proof of 29 CFR 1910.120 training will be provided to CRA's HSO prior to commencement of work. All personnel who attend the Facility-specific initiation session will sign a Training Acknowledgment Form, an example of which is presented as Attachment E.1.

TABLE E.3

SPECIFIC PERSONAL PROTECTION LEVELS
RFI WORK PLAN
FORMER PEREGRINE (US) INC. COLDWATER FACILITY
GENESEE TOWNSHIP, MICHIGAN

Work Task	*Maximum Protection Level*[1]	*Alternate Protection Level*[2]
Mobilization and Demobilization of Labor, Materials, and Equipment to and from Site	Modified D	D
Surveying Activities	Modified D	D
Site Setup Activities Including Construction of a Decontamination Pad	Modified D	D
Drilling and Sampling Activities		
• Activities with Potential Contact to Waste Materials	C[3]	Modified D
• Activities with No Potential Contact to Waste Materials	Modified D[3]	D
Decontamination Activities	C	Modified D

Notes:

Specific requirements of protection levels are detailed in Section 9.0.

(1) Level C: To be worn when the criterion for using air-purifying respirators (APRs) are met and a lesser level of skin protection is needed.
Modified Level D: To be worn when dermal protection is required, however, no respiratory hazards are present. It provides minimal protection against chemical hazards.

(2) Alternate protection levels will be used if monitoring indicates that conditions are appropriate and the HSO and Site Engineer agree that there is a reduced potential of exposure.

(3) May require additional personal protective equipment. See Confined Space Entry Permit.

8.0 WORK AREAS

Specific work areas will be delineated by fence or a flagged line as outlined below:

a) Temporary Exclusion Zone (EZ) - This zone will include all areas where potentially contaminated soils or materials are to be sampled, and all areas where contaminated equipment or personnel travel. When drilling or sampling is being conducted, the EZ will encompass a circle 20 feet in diameter around the drilling or sampling location. This area may vary depending on field conditions.

The EZ will be clearly delineated in the field prior to commencing Facility work, by temporary fencing with warning signs or other appropriate warning delineation spaced around the perimeter of the Zone warning of a hazardous work area.

b) Contaminant Reduction Zone (CRZ) - This zone will occur at the interface of the EZ and Support Zone and will provide access for the transfer of materials and Facility dedicated equipment to the EZ, the decontamination of equipment handling contaminated soil prior to leaving the EZ, the decontamination of personnel and clothing prior to entering the Support Zone, and for the physical segregation of the Support Zone and EZ.

c) Support Zone (SZ) - This area is the portion of the Facility defined as the area outside the zone of significant soil contamination. The Support Zone will be clearly delineated and procedures implemented to prevent active or passive migration of contamination from the work area. The function of the Support Zone includes:

1) an entry area for personnel, material, and equipment to the CRZ,

2) an exit area for decontaminated personnel, materials, and equipment from the EZ,

3) the housing of Facility special services, and

4) a storage area for clean safety and work equipment.

9.0 PERSONAL PROTECTIVE EQUIPMENT

CRA will require that all personnel performing RFI activities are equipped with PPE appropriate for the nature of work being completed. CRA will require that all safety equipment and protective clothing are kept clean, well maintained, and that their integrity is intact.

Safety equipment and apparel as required for general work at the Facility will be Level D, modified Level D, and Level C protective equipment within the Exclusion Zone. The anticipated PPE requirements for various Facility activities are presented in Table E.3.

The Level D protective equipment consists of the following:

i) work boots with steel toe and shank;
ii) hard hat;
iii) nitrile and/or cotton gloves; and
iv) safety glasses and/or goggles.

The modified Level D protective equipment consists of the following:

i) tyvek® coveralls (polycoated tyvek® when handling or working with liquids);
ii) work boots with steel toe and shank;
iii) disposable nitrile inner gloves - chemical resistant;
iv) outer nitrile work gloves - chemical resistant;
v) safety glasses and/or goggles;
vi) splash shields as necessary; and
vii) hard hat.

Level C protective equipment consists of the following:

i) individually assigned half- or full-facepiece air-purifying respirators (NIOSH approved), with appropriate cartridges for organic vapors and particulates. Respirators should be available at all times and donned when required as indicated by air monitoring;
ii) tyvek® coveralls (polycoated tyvek® when handling or working with liquids);

iii) disposable nitrile inner gloves - chemical resistant;

iv) outer nitrile work gloves - chemical resistant;

v) work boots with steel toe and shank;

vi) chemical-resistant overboots or booties;

vii) hard hat; and

viii) safety glasses and/or chemical-resistant goggles.

Additional protective equipment guidelines to be implemented include:

i) prescription eyeglasses in use at the Facility will be safety glasses;

ii) contact lenses will not be permitted in the EZ;

iii) all disposable or reusable nitrile, and/or cotton gloves worn on the Facility will be changed, decontaminated or discarded at the end of each day;

iv) during periods of respirator usage, respirator cartridges and filters will be changed daily, or upon breakthrough, whichever occurs first;

v) personnel performing RFI activities who have not passed a respirator fit test will not be permitted to enter or work in the EZ. Personnel will not be permitted to have beards, or long sideburns or mustaches as these interfere with a proper fit of the respirator;

vi) all PPE worn at the Facility will be decontaminated or discarded at the end of each work day;

vii) duct tape will be used to ensure that disposable coveralls and gloves are tightly secured when personnel are working within the EZ; and

viii) no watches, rings, or other jewelry accessories will be permitted during drilling or sampling activities.

10.0 RESPIRATOR PROGRAM

Prior to arriving at the Facility, all personnel performing RFI activities will have received training in the use of, and have been fit tested for, either half- or full-facepiece respirators.

At each sampling location, a background air monitoring reading will be taken using a Photoionization Detector (PID) to determine if organic vapors are present.

Action levels of organic vapor monitoring to determine the level of respiratory protection necessary during field activities will be:

Sustained Photoionization Organic Vapor Reading Above Background	*Protection Level*
0 ppm	half or full facepiece air purifying respirator available
>0 ppm	half or full-facepiece air purifying respirator (Level C)
>5 ppm	Supplied Air or Self-Contained Breathing Apparatus (SCBA) (Level B)

Work will be stopped and the work area will be allowed to vent if monitoring indicates that any of the following conditions exist:

i) toxic gases are present at concentrations which present IDLH conditions, or in excess of the protection factor afforded by the air purifying respirator (whichever is lower); or

ii) the oxygen content of the air is less than 19.5 percent.

If operations are stopped due to action level exceedances, air monitoring should continue at a safe distance, to determine if a threat to the surrounding community exists.

11.0 PERSONAL HYGIENE

CRA will require that all personnel performing or supervising work within the EZ observe and adhere to the personal hygiene-related provisions of this section.

Personnel performing RFI activities found to be disregarding the personal hygiene-related provisions of this HASP will, at the discretion of the HSO, be barred from the Facility.

CRA will ensure that the following equipment/facilities are available for the personal hygiene of all on-Facility personnel:

i) suitable disposable outerwear, gloves, respiratory protection, and footwear on a daily basis for the use of personnel performing RFI activities;

ii) disposal containers for used disposable outerwear; and

iii) potable water and a suitable sanitation facility.

CRA will enforce the following regulations for personnel actively participating in the field sampling program:

i) personnel performing RFI activities 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 the designated area of the Support Zone; and

iv) personnel performing RFI activities will thoroughly cleanse their hands, face, neck area, and other exposed areas before smoking, eating, or drinking and before leaving the Facility.

12.0 AIR MONITORING

During the progress of active work, air quality at the Facility will be monitored. Monitoring will be conducted on a daily basis and additionally as required by special or work-related conditions. The air monitoring instruments and frequency of their use is presented in Table E.4.

The daily monitoring program will consist of monitoring with the PID, calibrated daily for volatile organic vapors.

All monitoring equipment will be calibrated on a daily basis in accordance with the manufacturer's guidelines, and such calibrations will be recorded in the Facility daily log book. Results of all daily air monitoring will be recorded in the Facility daily log book and copies will be provided to the Facility Engineer.

Air monitoring will be conducted initially prior to conducting intrusive field work at the source of the potential contaminant, and periodically thereafter in the breathing zone of workers.

Immediately upon identifying sustained elevated levels of volatile vapors in the breathing zone, results shall be reported to the HSO, who will determine when operations be shut down and restarted.

The results of the air monitoring program will be recorded in a log book which will be maintained at the Facility.

TABLE E.4

AIR MONITORING INSTRUMENTS AND FREQUENCY FOR SITE ACTIVITIES FORMER PEREGRINE (US) INC. COLDWATER ROAD FACILITY GENESSE TOWNSHIP, MICHIGAN

Site Activity	*PID*
Drilling	X initial and periodic
Monitoring Well Installation	X initial and periodic
Soil Sampling	X initial and periodic
Ground Water Sampling	X initial and periodic
Sediment Sampling	X initial and periodic

13.0 COMMUNICATIONS

Emergency numbers including police, fire, ambulance, hospital, and appropriate regulatory agencies (Table E.5) will be maintained at the Facility as well as a route map to the nearest emergency medical facility (see Figure E.13.1).

Prior to initiating Facility activities, the emergency medical facility will be notified of Facility activities to ensure preparedness to respond to any RFI activity-related injuries.

TABLE E.5

EMERGENCY CONTACTS
FORMER PEREGRINE (US) INC. COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Agency/Firm	*Emergency Telephone Number*	*Business Telephone Number*
Local Emergency Services		
Fire Department	911	
Police Department	911	
Ambulance	911	
McLaren Regional Medical Center 401 South Ballenger Highway Flint, Michigan 48532		(810) 342-2300
National Poison Center		(800) 942-5969
National Response Center		(800) 424-8802
CRA Industrial Hygiene - Chicago (Matthew Lazaric) - Corporate (Mitch Bergner)		 (773) 380-9933 (651) 639-0913
CRA Project Manager - Glenn Turchan		(519) 884-0510

Hospital Route:

East on Coldwater Road (0.2 miles)
Left onto Horton Avenue (0.5 miles)
Continue on to Selby Street (1.0 miles)
Slight Left to I-475 South (3.6 miles)
Exit No. 7 towards Court Street/Downtown (0.3 miles)
Merge onto SB Chavez Drive (0.2 miles)
Right onto E. Court St/MI-21 (1.6 miles)
Continue onto W. Court Street (0.8 miles)
Slight Right onto Beecher Road (0.6 miles)
Right onto S. Ballenger Hwy. (0.2 miles)

14.0 EMERGENCY AND FIRST AID EQUIPMENT

Safety equipment will be available for use by personnel performing RFI activities and will be located and maintained at the Facility. The safety equipment will include, but is not limited to, the following:

i) portable emergency eye wash;

ii) two ABC type dry chemical fire extinguishers; and

iii) first-aid kit for a minimum of ten personnel.

15.0 EMERGENCY RESPONSE PLAN

Prior to commencing work, CRA will coordinate the development of an emergency contingency plan. The plan is intended to provide immediate response to a serious Facility occurrence such as injury, explosion or fire.

In the event of injury to personnel, the following protocol will be followed:

i) notify the Health and Safety Officer;

ii) contact Plant Security;

iii) contact the designated hospital and describe the injury, if necessary;

iv) decontaminate personnel if necessary, and administer appropriate first aid. If personnel cannot be decontaminated, alert hospital to possible problems of contamination; and

v) transport personnel to the medical facility along a predefined route.

16.0 EQUIPMENT AND PERSONNEL DECONTAMINATION

During the initiation of the field investigative program, procedures will be implemented to reduce the amount of contact of both personnel and equipment with the waste constituents. These procedures include the following:

i) proper work practices that would lead to minimal direct contact with potentially contaminated material; and

ii) use of disposable equipment and clothing as much as practicable.

All equipment leaving the EZ which came in contact with potentially contaminated material will be decontaminated. Sampling equipment and tools will be cleaned using the following protocols:

i) wash thoroughly with non-phosphate detergent;

ii) tap water rinse and steam clean;

iii) thorough rinse with deionized distilled water;

iv) air dry; and

v) wrap in aluminum foil until required and during transport to the sampling site.

Tap water may be used from any municipal water treatment system. The use of an untreated potable water supply is not an acceptable substitute.

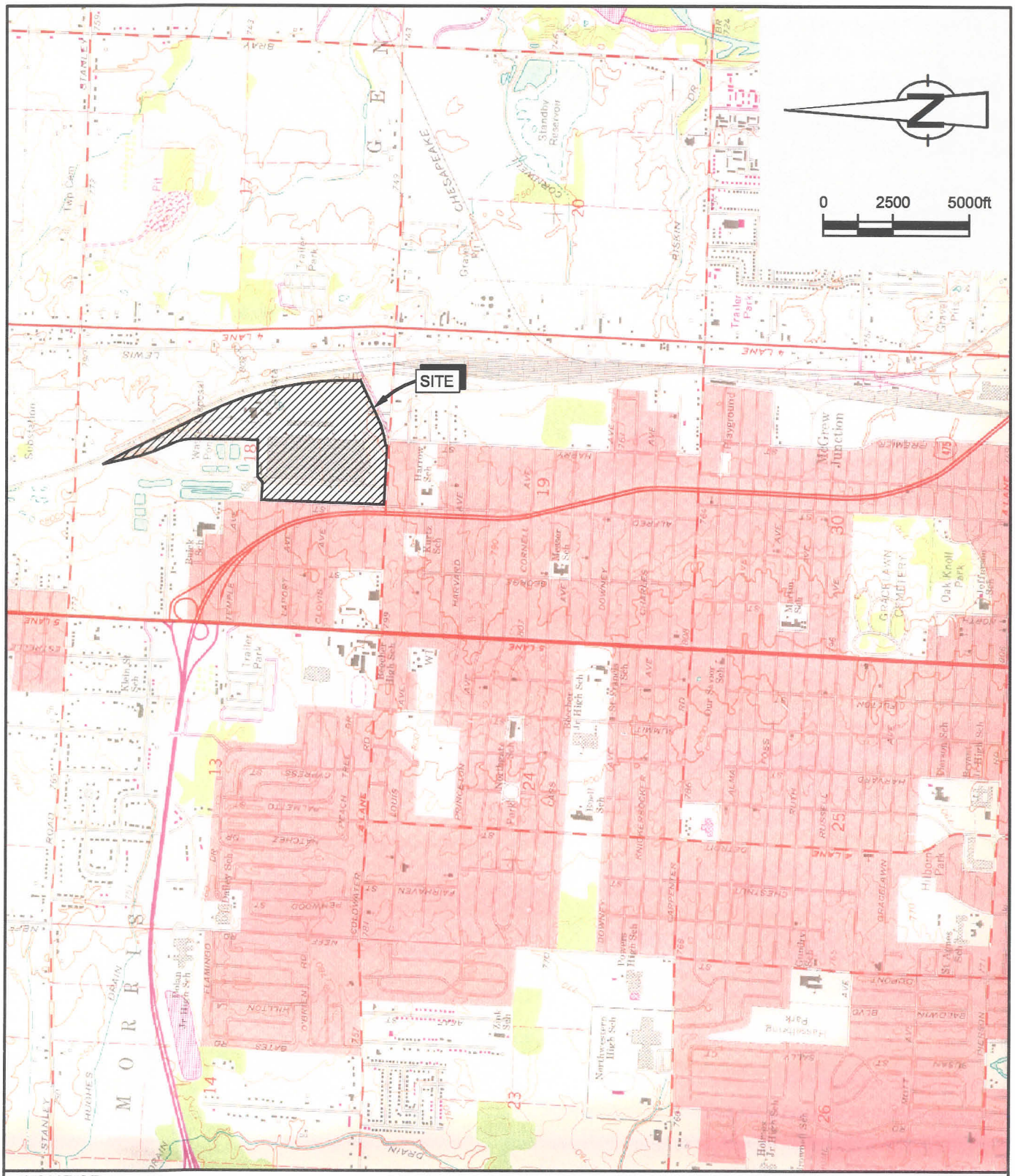
Personnel decontamination will take place at the exit from the EZ and will, as a minimum, consist of removal of gloves, disposable suit, and hard hat.

All personnel will remove their protective clothing 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 Exclusion Zone will be decontaminated prior to leaving the Facility as determined necessary by the HSO. 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 is clean or has been decontaminated prior to removal from the Facility.

Personnel engaged in vehicle decontamination will wear suitable protective equipment.



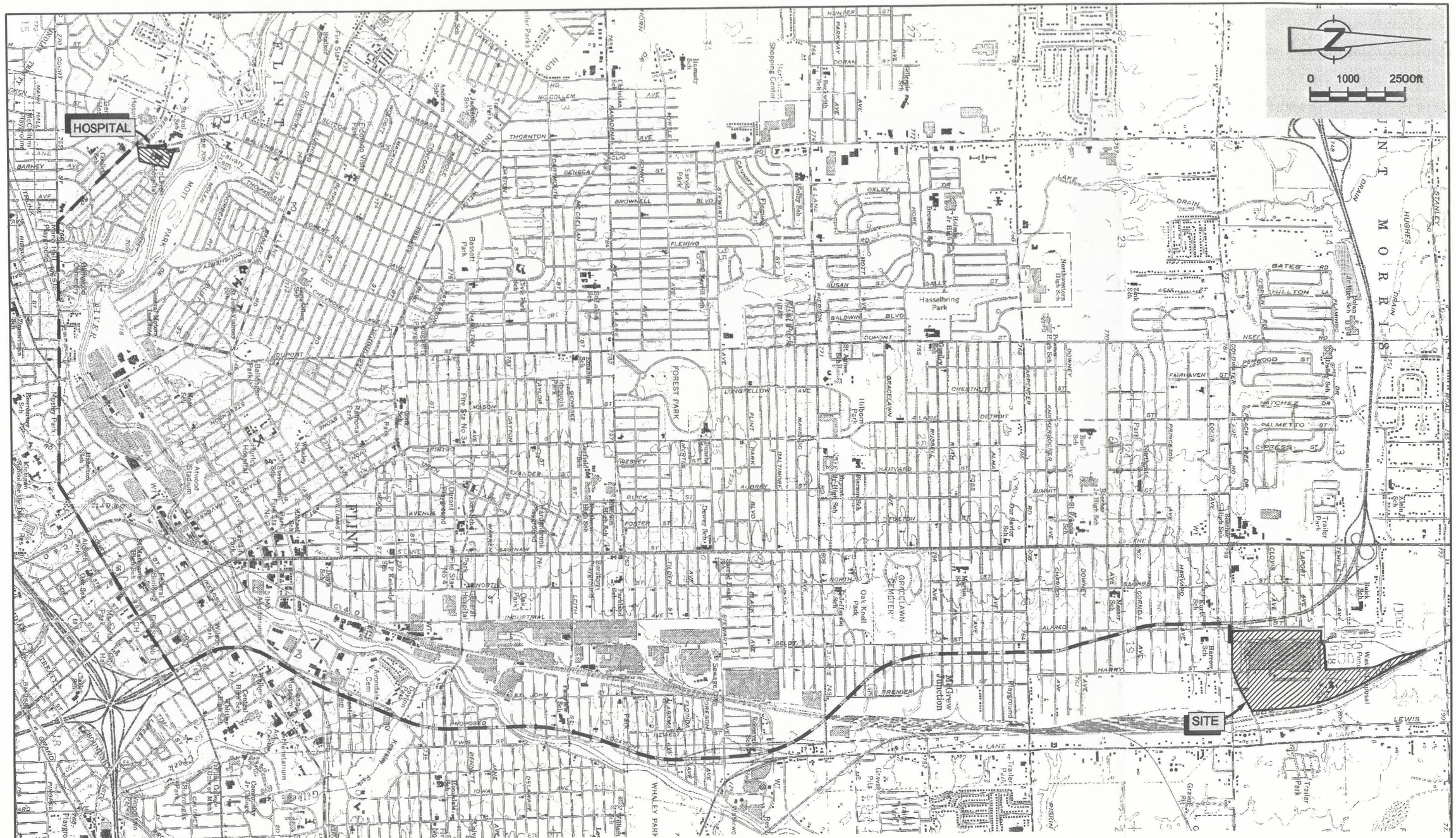
SOURCE:

USGS FLINT NORTH QUADRANGLE
MICHIGAN-GENESEE CO.
MAPS, PHOTOREVISED 1975.

figure E.2.1
FACILITY LOCATION
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA001 MAY 15/2000


HOSPITAL
SITE
N
0 1000 2500ft
FLINT
GENESEE TOWNSHIP
MORRIS
Hasselbring Park
Bassett Park
Atwood Stadium
Oak Park
Oak Knoll Park
Hilborn Park
Forest Park
Ballenger Park
Ramona Park
Whaley Park
Kearsley Park
Mott Park
Sarvis Park
Fleming Park
Grand Traverse Park
Trailer Park
Northwestern High Sch
Central High Sch
Bentley High Sch
Brownell Sch
Holmes Jr High Sch
Durant Sch
Dewey Sch
Whittier Sch
Washington Sch
Lincoln Sch
Martin Sch
Harvey Sch
Bunche Sch
Potter Sch
Doyle Sch
Pierce Sch
Jefferson Sch
Hamady Sch
Beecher Sch
Northern High Sch
Our Savior Sch
Grace Lutheran Sch
Flint Hospital
Hurley Hospital
Genesee Memorial Hospital
Calvary Cem
Glenwood Cem
Aresthale Cem
Oak Grove Cem
Shopping Center
Mass Transit Facility
Fire Sta No 3
Fire Sta No 4
Fire Sta No 5
Fire Sta No 6
Fire Sta No 7
Fire Sta No 8
Fire Sta No 9
Fire Sta No 10
Fire Sta No 11
Fire Sta No 12
Fire Sta No

ATTACHMENT E-1

TRAINING ACKNOWLEDGEMENT FORM

TRAINING ACKNOWLEDGEMENT FORM

Please Print:

NAME: _____

ADDRESS: _____

SOCIAL SECURITY NUMBER: _____

EMPLOYER: _____

JOB SITE: _____

I have attended and understood the mandatory Site-specific initiation session for the above-referenced job site. This program referenced the following topics:

i) known potential hazards on Site;

ii) level of personal protection equipment required;

iii) emergency procedures for the Site; and

iv) have received explanation of the basics of the Health and Safety Plan.

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

APPENDIX F

COMMUNITY RELATIONS PLAN

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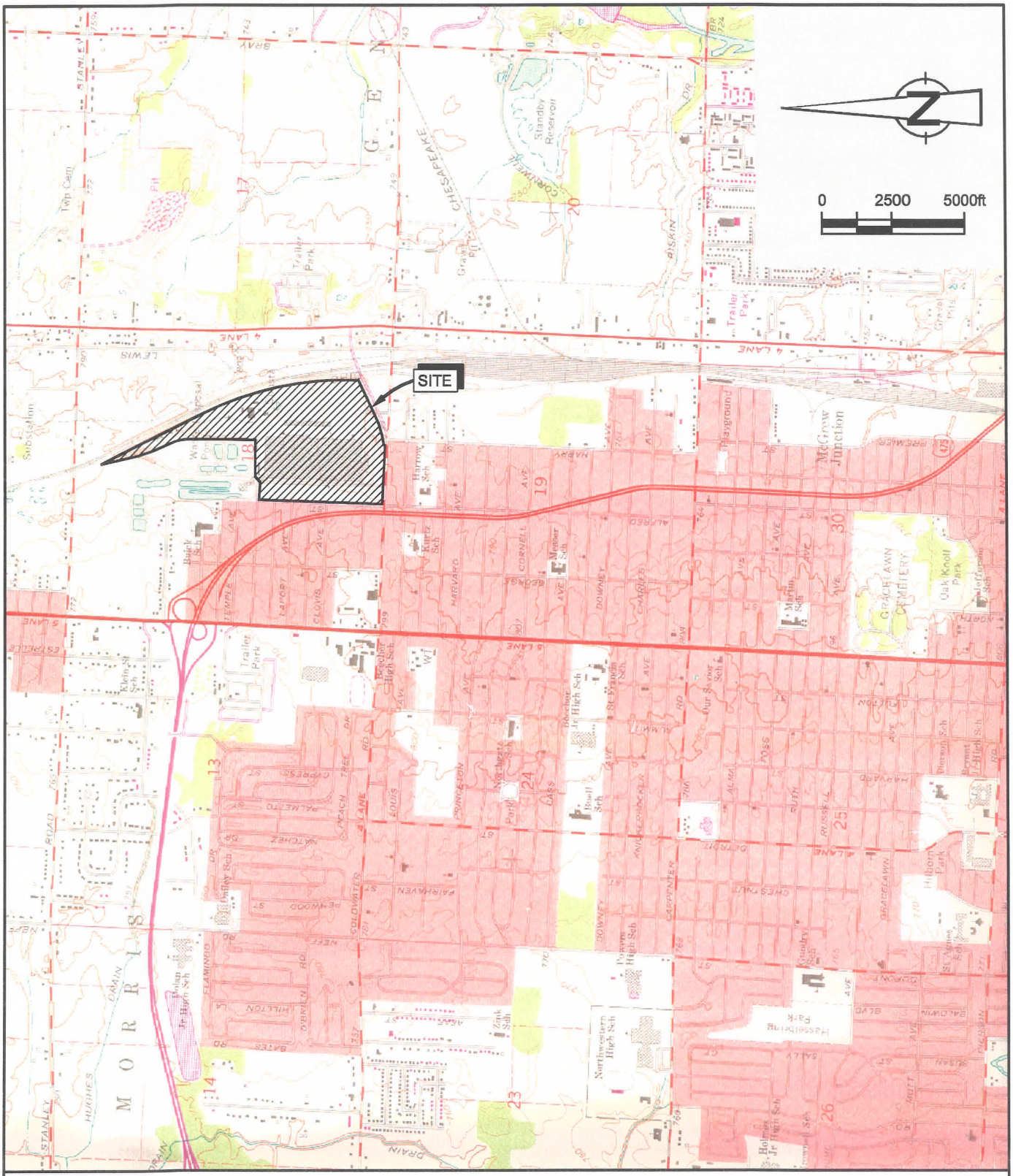
1.0 INTRODUCTION

This Community Relations Plan (CRP) is submitted as an appendix to and forms part of the RCRA Facility Investigation (RFI) Work Plan prepared by Remediation and Liability Management Company, Inc. (REALM) for the Former Peregrine (US) Inc. Coldwater Road Facility (Facility).

The Facility is located at G1245 East Coldwater Road, Genesee Township, Michigan as presented on Figure F.1.1. The plant was used as a plating facility and a stamping plant that produced interior components for cars and light trucks, principally door hinges and window regulators. Operations at the Facility began in 1953. In 1996 the plant and its operations were sold to Peregrine, Inc. Operations at the Site ceased in 1998 and REALM has reacquired the property and the plant and is in the process of decommissioning and demolishing the plant buildings.

The CRP is intended to identify the mechanisms which will be utilized for the dissemination of information to the public regarding RFI/CMS project activities.



SOURCE:

USGS FLINT NORTH QUADRANGLE
MICHIGAN-GENESEE CO.
MAPS, PHOTOREVISED 1975.

figure F.1.1
FACILITY LOCATION
FORMER PEREGRINE (US), INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

12636-40(011)GN-WA027 MAY 15/2000

2.0 FACILITY HISTORY AND DESCRIPTION

2.1 FACILITY HISTORY

A detailed description of the Facility history is presented in Section 2.2 of the RFI Work Plan.

2.2 CURRENT OPERATIONS

There are currently no manufacturing operations at this Facility. The Facility is undergoing a complete and systematic environmental decommissioning and demolition program that began during the summer of 1999 and is expected to be completed in 2001.

3.0 COMMUNITY RELATIONS PROGRAM ACTIVITIES

3.1 COMMUNITY RELATIONS GOALS

REALM will conduct community relations on an as required basis.

The goal of the CRP is as follows:

1. keep the public informed as the project progresses and as results are received; and
2. provide a mechanism for disseminating information on a routine as well as a non-routine basis in response to individual requests.

3.2 CRP IMPLEMENTATION

3.2.1 SCOPE

Implementation of the CRP will ensure a regular flow of information/data/results from REALM to the general public during the course of the RFI/CMS project (as required). An Information Repository will be created at the Genesee Township office, as required. The phone number for the Genesee Township office is 1-810-640-2000.

It should be noted, however, that significant public participation is not expected until the CMS and CMI phases of the overall project.

3.2.2 RFI COMPLETION

Upon approval by MDEQ-WMD of a CMS Final Report, the MDEQ-WMD will provide the public with an opportunity to review and comment on the proposed corrective measures, if any.

3.2.3 PROGRESS LETTERS

Quarterly progress reports will be prepared to provide prompt and accurate information regarding the status of the project to the MDEQ-WMD.

3.2.4 UNSCHEDULED COMMUNICATION

REALM will, as necessary, respond to comments or concerns of individual members of the public and/or local agencies in response to individual requests.

APPENDIX G

FIELD SAMPLING PLAN

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1.0 INTRODUCTION

This Field Sampling Plan (FSP) is submitted as an appendix to and forms part of the RCRA Facility Investigation (RFI) Work Plan prepared by Remediation and Liability Management Company, Inc. (REALM) for the Former Peregrine (US) Inc. Coldwater Road Facility (Facility).

The FSP describes procedures for the collection of soil and groundwater samples.

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 the QAPP (Appendix C), samples will be collected in order of decreasing analyte volatility (i.e., VOCs first, SVOCs second, which are followed by containers for the remaining analysis). Table G.2.1 summarizes sample collection and analysis including 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.
2. A new pair of disposable latex gloves will be used at each location to be sampled for chemical analyses. Additional glove changes 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, tyveks, etc. will be collected and containerized for proper disposal at RCRA Subtitle D waste.
5. Samples will be identified using labels. Samples will also be labeled noting the Facility, sample location, sample interval (if appropriate), analysis required, preservative added, date, time and sampler's initials. All sample preservation protocols will be followed in adherence with the QAPP. A hard cover bound field book will be maintained to record all samples and sampling events.
6. Containers for sample collection and preservation requirements will be determined as required by the analytical parameters. 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 Facility in sealed containers.
7. All collected sample shipments for chemical analysis will be iced in laboratory supplied coolers after collection and labeling. Any remaining space will be filled with packing to cushion the containers within the shipment coolers. Each cooler

TABLE G.2.1

PROPOSED RFI SUMMARY
FORMER PEREGRINE (US), INC.
COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

AOI Number	AOI Description	Selection Rationale	Previous Investigation	# Boreholes or Wells	Total # of Samples	Exceedance of Standards					Proposed # of Boreholes/Samples (1)	Analytical Parameters Comments (2)(3)
						Matrix	Location	Parameter	Concentration	Standard		
Bkgd	Background	Establish Background	No	0	0	--No sampling performed--					4/8	TAL Grain Size Hydraulic Conductivity (K)
AOI 1	Former Drum Storage Area	U.S. EPA SWMU 1	Yes	6 2	8 4	Soil Water	MW-14/PFB-6 MW-14	TCE TCE	2,210/1,600 µg/kg 21.3/6.0 µg/L	IDWP IDWC	None	Ongoing RCRA Closure Sampling (To Be Conducted)
AOI 2	Former Waste Pile Pad	U.S. EPA SWMU 2	Yes	1 1	1 2	Soil Water	 MW-13	 TCE	No exceedances 8.4 µg/L	 IDWC	3/6 Resample	VOC/SVOC/PCB/TAL VOC
AOI 3	Former USTs	U.S. EPA SWMU 3	Yes	1 1	1 1	Soil Water			No exceedances No exceedances		None	No sampling proposed (closed)
AOI 4	Former Satellite Accumulation Areas	U.S. EPA SWMU 4	No	0	0	- - No sampling performed - -					None	No sampling proposed U.S. EPA identified low potential for concern Addressed through facility - wide sampling
AOI 5	Former Hazardous Materials Storage Area	U.S. EPA SWMU 5	No	0	0	- - No sampling performed - -					None	No sampling proposed AOI 5 cleaned by Peregrine U.S. EPA identified low potential for concern
AOI 6	Former Wastewater Storage Tanks	U.S. EPA SWMU 6	No	0	0	- - No sampling performed - -					None	No sampling proposed Tanks removed by Peregrine U.S. EPA identified low potential for concern
AOI 7	Former Scrap Metal Management System	U.S. EPA SWMU 7	Yes	8 1	10 1	Soil Water	 PFW-10	 Barium	No exceedances 2,600 µg/kg	 IDWC	None	No sampling proposed (see AOI #19)
AOI 8	Former Scrap Metal Storage Area	U.S. EPA SWMU 8	Yes	5 1	5 0	Soil Water	PFB-7	Arsenic	32,000 µg/kg Well was dry	IDWP	5/10 [4]	VOC/SVOC/PCB/TAL Includes 2 boreholes at the southern end of Building 63
AOI 9	Former Plating Areas	U.S. EPA SWMU 9	Yes	12 1	15 1	Soil Water	K-32 K-35 K-35 K-35 F-30 	Lead Cadmium Chromium Lead Cyanide 	42,800 µg/kg 6,050 µg/kg 1,160,000 µg/kg 1,280,000 µg/kg 31,800 µg/kg No exceedances	IDWP IDWP IDWP IDWP/IDC IDWP 	30/60	VOC/TAL Modified Appendix IX (2 samples)

TABLE G.2.1

PROPOSED RFI SUMMARY
FORMER PEREGRINE (US), INC.
COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

AOI Number	AOI Description	Selection Rationale	Previous Investigation	# Boreholes or Wells	Total # of Samples	Exceedance of Standards					Proposed # of Boreholes/Samples (1)	Analytical Parameters Comments (2)(3)
						Matrix	Location	Parameter	Concentration	Standard		
AOI 10	Overhead Waste Pipes	U.S. EPA SWMU 10	Yes	--	5	Waste-TCLP			No exceedances		None	No sampling proposed
AOI 11	Former Coal Storage Area	U.S.EPA AOC	Yes	2 0	2 0	Soil Water			No exceedances No samples collected		5/10 [5]	SVOC/PCB/TAL
AOI 12	Former Caustic Tanks	U.S.EPA AOC	No (moderate potential)	0	0	-- No sampling performed --					2/4	pH/VOC/TAL
AOI 13	Former Headliner Adhesive Tanks	REALM AOI	Yes	1 0 --	2 0 1	Soil Water Waste-TCLP	 AST No. 6 AST No. 6	 Benzene Flammability	No exceedances No samples collected 79000 µg/L <78 F	 Haz Waste Disposal	2/4 None	VOC
AOI 14	Former Incinerator	REALM AOI	Yes	2 2	2 3	Soil Water			No exceedances No exceedances		2/4	SVOC/PCB/TAL
AOI 15	Other Former USTs	REALM AOI	Yes	4	5	Soil			No exceedances		None	No sampling proposed (closed)
AOI 16	Former Potential Tank Farm	REALM AOI	Yes	2	2	Soil			No exceedances		None	No sampling proposed No storage tanks ever installed
AOI 17	Substation Drainage Sump	REALM AOI	Yes	1 0	2 0	Soil Water			No exceedances No samples collected		2/4 [6]	SVOC/PCB
AOI 18	Former IERY	REALM AOI	Yes	8 2	10 4	Soil Water	 MW-3	 Lead	No exceedances 12 µg/L	 IDWC	4/8 [7] Resample	VOC/SVOC/PCB/TAL Lead only
AOI 19	Former Press Room	REALM AOI	Yes	2 0	2 0	Soil Water			No exceedances No samples collected		8/16	VOC/SVOC/PCB/TAL Modified Appendix IX (1 sample)
AOI 20	Former Tool Room	REALM AOI	Yes	0 0 --	0 0 1	Soil Water Waste-TCLP	 Air Wash Unit Air Wash Unit	 Chromium Lead	No samples collected No samples collected 8800 µg/L 6100 µg/L	 Haz Waste Disposal	4/8	VOC/SVOC/PCB/TAL Modified Appendix IX (1 sample)

TABLE G.2.1

PROPOSED RFI SUMMARY
FORMER PEREGRINE (US), INC.
COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

AOI Number	AOI Description	Selection Rationale	Previous Investigation	# Boreholes or Wells	Total # of Samples	Exceedance of Standards					Proposed # of Boreholes/Samples (1)	Analytical Parameters Comments (2)(3)
						Matrix	Location	Parameter	Concentration	Standard		
AOI 21	Powerhouse Fuel Oil Storage Tanks	REALM AOI	Yes	1 1	1 1	Soil Water			No exceedances No exceedances		2/4	VOC/SVOC/TAL
Other	Non-specific	Previous Sampling	Yes	3	4	Water Water Water	MW-4 MW-4 MW-10	TCE Lead Lead	5.3 µg/L 26 µg/L 14 µg/L	IDWC IDWC IDWC	Resample Resample Resample	VOCs Lead only Lead only
				2	3	Water	PFW-9	Lead	9.0 µg/L	IDWC	Resample	Lead only
TOTAL # BOREHOLES & SAMPLES (performed during previous investigations)				**73**	**99**						**TOTAL # BOREHOLES/SAMPLES** (to be performed during proposed RFI)	**73/146**

Notes:

(1) It is expected that a maximum of two soil samples will be collected from each borehole. The first sample will be collected at the 0 to 2 foot bgs horizon. The second sample will be collected at the interval exhibiting the greatest degree of staining or positive response on the PID. If no staining or PID response is encountered, the lower soil sample may not be collected. All boreholes will be installed to a depth of approximately 5 feet into the native clay till horizon. All boreholes will be sampled continuously to define stratigraphy and the potential presence of a continuous perched water table zone.

(2) pH - pH
VOC - Target Compound List Volatile Organic Compounds
SVOC - Target Compound List Semi-Volatile Organic Compounds
PCB - Target Compound List Polychlorinated Byphenols
TAL - Target Analyte List Inorganics (include both total and hexavalent chromium)

(3) A perched water table zone exists in isolated locations of the near surface at the Facility. Perched water table monitoring wells will be installed during a second phase of sampling if a significant groundwater aquifer is identified at each of the AOIs. A significant groundwater aquifer may be present if groundwater is identified to be continuous over the Facility and the formation is capable of producing a significant yield (eg. 5 feet of porous media, 2 feet saturated, continuous horizontally). The exact location and number of monitoring wells will be determined after the boreholes have been completed.

(4) The boreholes will be completed either through or immediately adjacent to the former scrap metal collection bins.

(5) The boreholes will be completed after the residual coal has been removed.

(6) The boreholes will be completed either through or immediately adjacent to the substation drainage sump.

(7) Borehole locations will be biased to low areas with broken/cracked concrete or asphalt.

will be sealed with a transportation custody seal containing the sampler's initials. The cooler will then be sealed with packing tape.

All samples will be delivered to the laboratory by commercial courier or CRA personnel, the day following sample collection.

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

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 by a pressurized water or steam wash to remove oil, grease, mud and other foreign matter. The drill rig will be inspected by the CRA engineer or geologist to ensure that all seals and gaskets are intact, and that no fluids are leaking. After the visual inspection has been completed, the engineer or geologist will determine if additional cleaning is required. If required, the drill rig will receive a thorough re-cleaning using a pressurized water or steam wash. The drill rig, augers, and stems will be decontaminated between boreholes, as required. The split spoons will be decontaminated between samples by washing thoroughly with non-phosphate detergent and potable water using a brush to remove particulate matter or surface film, if any.

Prior to the collection of any samples designated for chemical analyses, all sampling equipment and tools, except for disposable bailers or dedicated equipment, will be cleaned using the following cleaning protocols as follows:

i) wash thoroughly with non-phosphate detergent using a brush to remove particulate matter or surface film if any;

ii) tap water rinse and steam clean;

iii) thorough rinse with deionized distilled water;

iv) air dry to the extent practical; and

v) wrap in aluminum foil until required and during transport to the sampling site.

Electric water level tapes will be cleaned prior to use at each monitoring well location by rinsing with distilled/deionized water rinse.

Upon completion of drilling activities, the drill rig will be thoroughly cleaned by a pressurized water or steam wash to remove soils and other foreign matter prior to demobilization from the Facility.

Fluids used for cleaning will not be recycled.

2.3 WASTE HANDLING

Soil cuttings will be placed back into the boring from which they were generated. Non-hazardous debris will be disposed of with plant trash.

All development water and excess sampling waters will be either discharged to ground surface in the vicinity of the well from which they were generated or managed until compatibility with wastewater treatment has been determined. Wash waters may be collected for treatment at the Buick City wastewater treatment plant (WWTP) (if necessary).

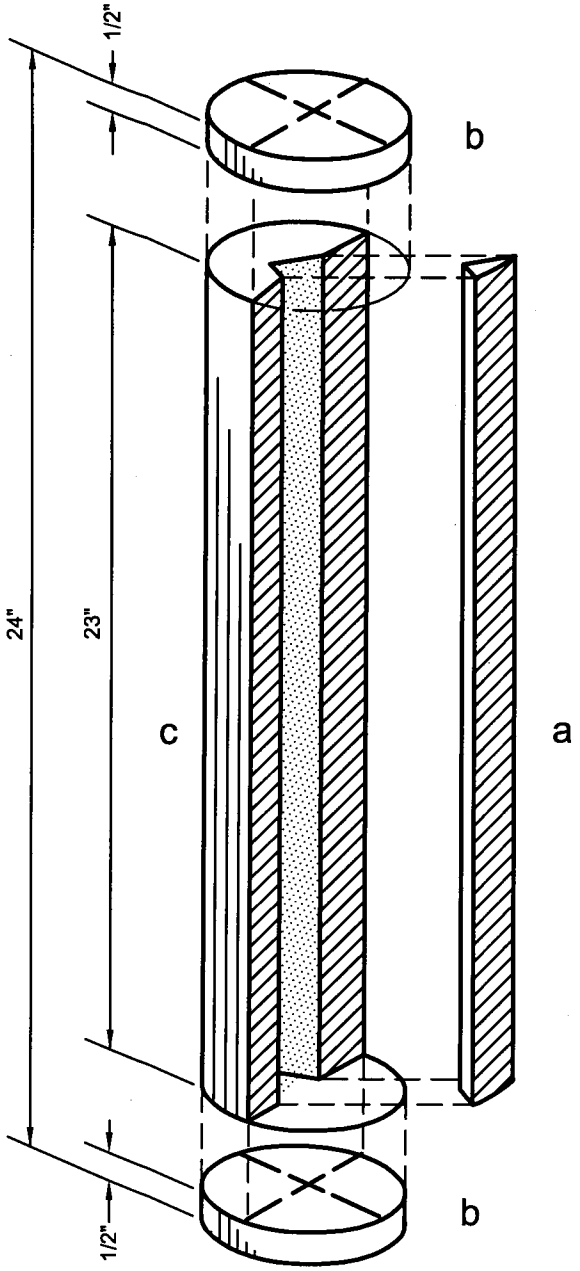
Additional protocols specific to each sampling method are presented in the following sections.

3.0 SOIL SAMPLING PROTOCOLS

Soil samples will be collected continuously during drilling to identify and classify soil materials. All soil samples will be collected using the Dual Tube Soil Sampling System (Geoprobe System). Soils will be described and classified according to the Unified Soil Classification System (USCS).

The soil samples selected for chemical analyses will be sampled in the following manner:

1. A new pair of disposable latex gloves will be used for each sample collected.
2. Prior to use, for each sample, all sampling equipment will be precleaned in accordance with the prescribed rinse sequence.
3. Soil samples will be collected in a cleaned thin-walled PETG tube.
4. Following collection of each soil sample, the tube will be opened.
5. The core will be examined for changes in stratigraphy. Screening for VOCs will be accomplished by cutting the core and running the end of the PID along the entire length of the core.
6. Sample depths chosen for analysis will have samples collected in the following manner:
    - 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 in 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 pin 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 the T-handle.



TYPICAL SOIL CORE

a= PORTION OF SAMPLE FOR CHEMICAL ANALYSES

b= PORTION OF SAMPLE DISCARDED

c= PORTION OF SAMPLE FOR GEOLOGIC RECORD

figure G.3.1

SOIL SAMPLE SELECTION DETAIL
RFI WORK PLAN
FORMER PEREGRINE (US) INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

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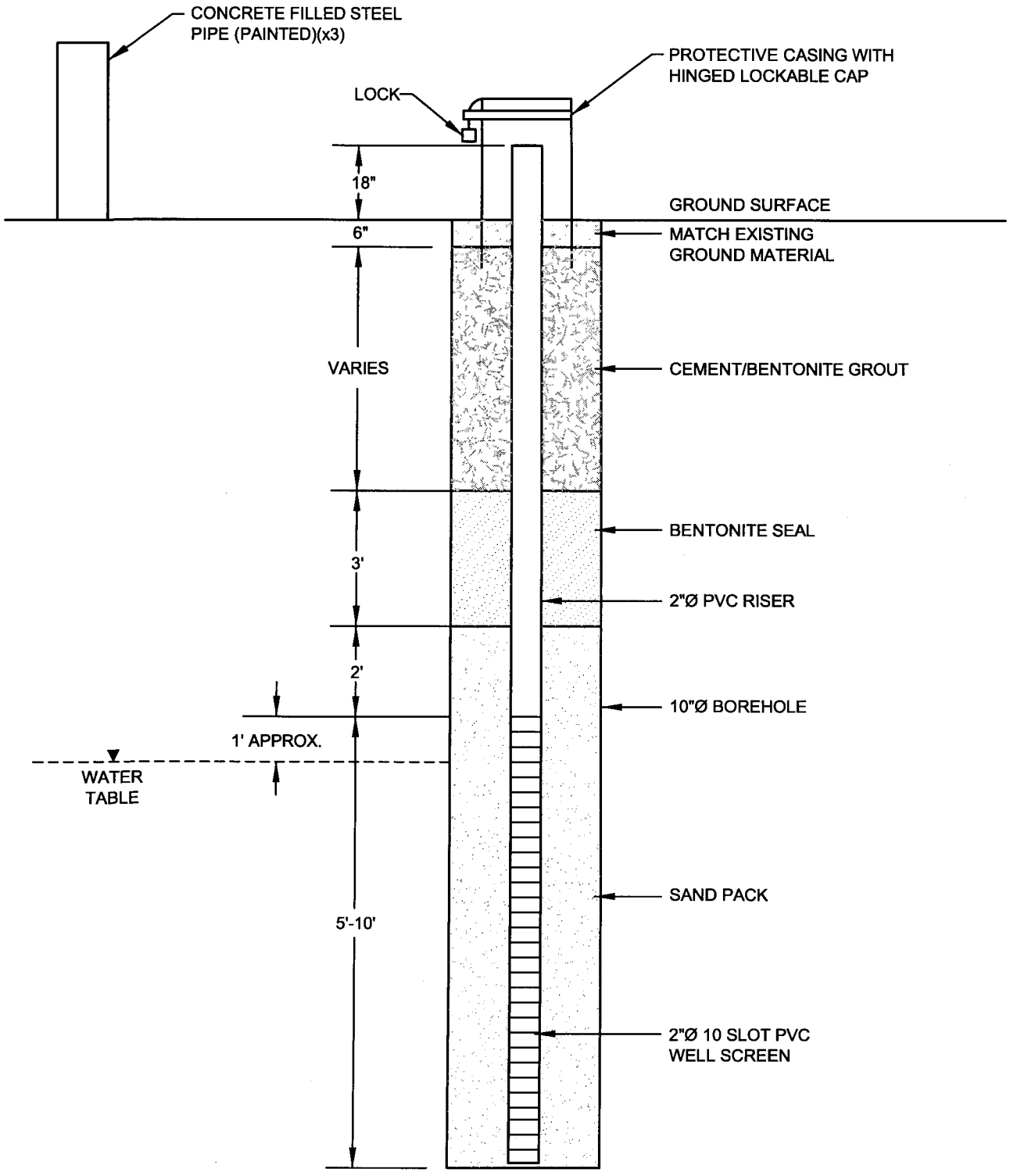

figure G.4.1

MONITORING WELL DETAIL
RFI WORK PLAN
FORMER PEREGRINE (US) INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

- Rotate the sampler rod counterclockwise until the wings rest firmly against the lower tabs to lock the sampler.
- 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.

• Samples for other chemical analysis will be as follows:

- Samples removed with a precleaned stainless steel cutting tool from the stratigraphic layer(s) and homogenized in a precleaned stainless steel bowl and then transferred into the appropriate sample bottles in a manner illustrated on Figure G.3.1. The remainder of the sample core may be stored in plain glass containers as a geologic record of the sample taken.

7. Visual and olfactory observations will be noted.

8. All soil samples collection will be described and classified according to the USCS.

9. The sample locations will be surveyed upon completion.

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

4.0 HYDROGEOLOGIC INVESTIGATION

4.1 MONITORING WELL INSTALLATION PROCEDURES (IF REQUIRED)

Either PID readings or olfactory evidence will determine the soil sample depth collected from each borehole. If significant groundwater is present within the borehole (see RFI Work Plan), a monitoring well may be installed and a groundwater sample collected.

Monitoring well installations will be completed in accordance with U.S. EPA and MDEQ technical guidance. Wells will be screened across the water table or at the base of the surficial water bearing zone. All cuttings will be containerized and disposed of in accordance with Section 2.3.

Monitoring Wells

The monitoring wells will be completed as follows:

1. A borehole will be advanced to the determined depth using a 4 1/4-inch I.D. hollow-stem auger (or Geoprobe equivalent).
2. A 5 to 10-foot long, 2-inch diameter PVC well screen (#10 slot) and PVC riser will be set into and centered into the borehole. The top of the well screen will be set 1 foot above the groundwater table. The typical seasonal variation of the water table is expected to be approximately 1 to 2 feet. Screen lengths may be modified by the CRA field representative dependant upon the borehole specific conditions encountered.
3. A sand pack will be placed to a depth of approximately 2 feet above the top of the well screen to ensure that bentonite pellets and/or grout do not permeate the screen.
4. A bentonite pellet plug 3 feet thick will be placed immediately above the sand pack. The bentonite pellets will be tamped to ensure that a good seal is obtained.
5. The annular space between the borehole wall and the 2-inch casing will be grouted to within 6 inches of the ground surface with a cement/bentonite grout mixture. The grout will be placed using a tremie pipe set near the top of the bentonite pellet plug.
6. The top 6 inches will be backfilled with material consistent with the surrounding surface material (e.g., asphalt, concrete, topsoil, etc.)

The well will have a minimum stickup of 18 inches above the existing grade or may be completed as flushmount to prevent damage in high traffic areas. The well will be fitted with a surface protective casing constructed of 4-inch diameter steel pipe, 5 feet long, fitted with a locking cap. The protective casing will be set into the grout used to grout the borehole. Knockdown protection consisting of three concrete filled steel posts will be installed if deemed necessary. The concrete posts and the protective casing will be painted orange. In high traffic areas of the Plant, flush-mounted road boxes may be utilized.

A typical overburden monitoring well installation detail is presented on Figure G.4.1. An alternate flushmount installation detail is presented on Figure G.4.2.

Care shall be taken to maintain plumbness and alignment of the well installation and to center it in the borehole. An open borehole will be maintained to ensure proper well installation and completion.

4.2 WELL DEVELOPMENT

All monitoring wells will be thoroughly developed prior to sampling in accordance with the following protocols:

1. Water levels in all wells will be measured to ±0.01 foot prior to development;
2. Initial development will be conducted using a low flow pump, bailers and/or surge blocks to remove suspended solids. Wells will be developed in this manner until development criteria are achieved or until a maximum of ten well volumes have been removed;
3. After each well volume is removed, a sample will be analyzed for pH, temperature, and conductivity. Development will continue until three consecutive and consistent readings of pH, conductivity, and temperature are obtained. Readings will be considered consistent if all three values are within 10 percent of the average value over the last three volumes and all pH values are within ±1 pH unit of the average value over the last three well volumes;
4. Well development records shall be maintained as each well is developed; and
5. Development and purge water generated at each monitoring well location shall be handled in accordance with Section 2.3.

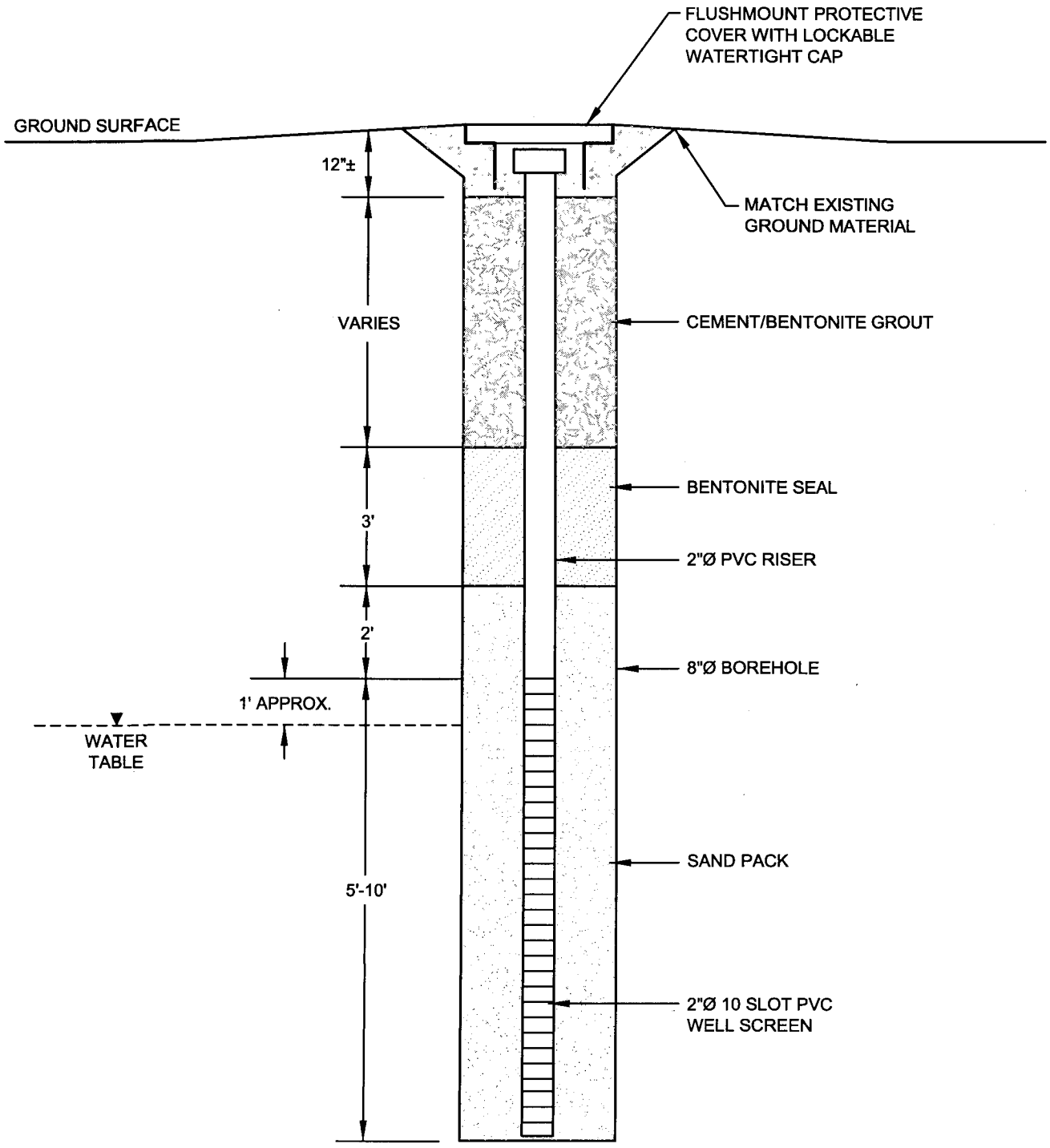

figure G.4.2

FLUSH MOUNT MONITORING WELL DETAIL
RFI WORK PLAN
FORMER PEREGRINE (US) INC. COLDWATER ROAD FACILITY
Genesee Township, Michigan

CRA

Sampling activities will not be commenced until one week after completion of the well development.

4.3 SURVEY AND 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 calculated by subtracting the measured depth to water from the TOC elevation. Water elevations will be measured to the nearest 0.01 foot. The measuring device will be precleaned prior to being used in each well as described previously.

5.0 GROUNDWATER QUALITY SAMPLING PROTOCOLS

One round of unfiltered and filtered groundwater samples will be collected from the locations indicated in Table G.1. Appropriate groundwater sampling procedures will be utilized if the samples are turbid (e.g., low flow purging).

All wells used for groundwater quality monitoring will be sampled according to the following protocols:

1. New disposable latex gloves will be used when sampling each well. Additional glove changes will be 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.
2. Prior to use, for each sample, all sampling equipment will be precleaned using the prescribed rinse sequence.
3. The sampler will measure and record the depth to water in each well to the nearest 0.01 foot using an electric tape. The electric tape will be cleaned prior to use in each well.
4. Prior to sampling, each well will be purged using a low flow pump. The monitoring wells will be purged by removing a minimum of three standing well volumes of groundwater where the volume of standing water is calculated as follows:

   $V = 0.041\ d^2\ h$

   where:

   V = volume of standing water in gallons

   d = diameter of the well in inches

   h = depth of water in feet

   Field measurements of pH, conductivity, and temperature of the purged water will be obtained and recorded following removal of each standing well volume and prior to sample collection. Calibration of field instruments, as specified in the QAPP, will be undertaken prior to use at each well location. Well purging will continue until three consecutive and consistent readings (± 10 percent) of pH, conductivity, and temperature are obtained. In the event that a well is pumped dry prior to achieving three well volumes, groundwater will be permitted to recover to a level sufficient for sample collection. Upon recovery,

the sample will be collected. All waste groundwater not collected for analysis will be disposed of as discussed in Section 2.3.

5. After purging the required volume of well water, water samples will be collected using a low flow pump. Filtered groundwater samples for metal analyses will be field filtered with a 0.45 micron filter. The water samples will be emptied directly into the appropriate sample containers, using techniques which will minimize sample agitation.

6.0 FIELD LOG

The field log book will be a bound document with consecutively numbered pages. The entries for each day commence on a new page which will be dated. All entries will be made only in indelible ink. Corrections will be made by marking through the error with a single line, so as to remain legible, and initialing this action followed by writing the correction. The field log books generated will be numbered consecutively and maintained by CRA.

The following information will be recorded in the field log book for each sample collected:

i) site location identification;

ii) unique sample identification number;

iii) date and time (in 2400 hour time format) of sample collection;

iv) weather conditions;

v) designation as to the type of sample (sediment or soil);

vi) designation as to the means of collection;

vii) name of sampler;

viii) analyses to be performed on sample; and

ix) any other relevant comments such as odor, staining, texture, filtering, preservation, etc.

7.0 SAMPLE SHIPMENT AND CONTAINERS

7.1 CHAIN-OF-CUSTODY FORMS

Chain-of-custody records will be used to track all samples from time of sampling to the arrival of samples at the laboratory.

Each shipping container being sent to the laboratory will contain a chain-of-custody form. The chain-of-custody form consists of four copies which are distributed to the sampler, to the shipper, to the contract laboratory, and to the office file of CRA. The sampler and shipper will maintain their copies while the other two copies are enclosed in a waterproof enclosure within the sample container. The laboratory, upon receiving the samples, will complete the remaining copies. The laboratory will maintain one copy for its records. The executed original will be returned to CRA with the data deliverables package.

7.2 SAMPLE CONTAINERS AND HANDLING

Required sample containers, sample preservation methods, maximum holding times, and filling instructions are provided in the QAPP.

All samples will be placed in appropriate sample containers, labeled and properly sealed. In addition, sample labels will include sample number, place of collection, date and time of collection, and analyses to be performed. Samples will be cushioned within the shipping coolers by the use of vermiculite and/or bubble pack. Samples will be kept cool by the use of plastic bags of ice or cooler packs, as required and each sample will have an individual sample tag.

Samples will be shipped by commercial courier on a daily basis to the project laboratory.

Two seals comprised of CRA's chain-of-custody tape will be placed around each shipping cooler prior to shipment to secure the lid and provide evidence that the samples have not been tampered with en route to the laboratory. Clear tape will be placed over the seals to ensure that they are not accidentally broken during shipment.

Upon receipt of the cooler at the laboratory, the cooler will be inspected by the designated sample custodian. The condition of the cooler and seal will be noted on the

chain-of-custody form by the sample custodian. The sample custodian will document the date and time of receipt of the cooler and sign the chain-of-custody forms.

The sample custodian then will check the contents of the cooler with those samples listed on the chain-of-custody form. If damage or discrepancies are noticed, they will be recorded in the remarks column of the chain-of-custody form, dated, and signed. They will be reported to the laboratory supervisor who will inform the laboratory manager and QA officer.

Sample disposal will be the responsibility of the laboratory. Upon disposal, the laboratory shall sign the next open "Relinquished by" box, and the word "Disposed" shall be written in the "Received by" box.

Geologic samples will be maintained at the Facility until completion of the RFI Report.

ATTACHMENT G-1

STANDARD PENETRATION TEST (ASTM D1586-84)

Designation: D 1586 – 84

Standard Method for Penetration Test and Split-Barrel Sampling of Soils[1]

This standard is issued under the fixed designation D 1586; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This method has been approved for use by agencies of the Department of Defense and for listing in the DOD Index of Specifications and Standards

1. Scope

1.1 This method describes the procedure, generally known as the Standard Penetration Test (SPT), for driving a split-barrel sampler to obtain a representative soil sample and a measure of the resistance of the soil to penetration of the sampler.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For a specific precautionary statement, see 5.4.1.

1.3 The values stated in inch-pound units are to be regarded as the standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 2487 Test Method for Classification of Soils for Engineering Purposes[2]

D 2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)[2]

D 4220 Practices for Preserving and Transporting Soil Samples[2]

3. Descriptions of Terms Specific to This Standard

3.1 *anvil*—that portion of the drive-weight assembly which the hammer strikes and through which the hammer energy passes into the drill rods.

3.2 *cathead*—the rotating drum or windlass in the rope-cathead lift system around which the operator wraps a rope to lift and drop the hammer by successively tightening and loosening the rope turns around the drum.

3.3 *drill rods*—rods used to transmit downward force and torque to the drill bit while drilling a borehole.

3.4 *drive-weight assembly*—a device consisting of the hammer, hammer fall guide, the anvil, and any hammer drop system.

3.5 *hammer*—that portion of the drive-weight assembly consisting of the 140 ± 2 lb (63.5 ± 1 kg) impact weight which is successively lifted and dropped to provide the energy that accomplishes the sampling and penetration.

3.6 *hammer drop system*—that portion of the drive-weight assembly by which the operator accomplishes the lifting and dropping of the hammer to produce the blow.

3.7 *hammer fall guide*—that part of the drive-weight assembly used to guide the fall of the hammer.

3.8 *N-value*—the blowcount representation of the penetration resistance of the soil. The *N*-value, reported in blows per foot, equals the sum of the number of blows required to drive the sampler over the depth interval of 6 to 18 in. (150 to 450 mm) (see 7.3).

3.9 ΔN—the number of blows obtained from each of the 6-in. (150-mm) intervals of sampler penetration (see 7.3).

3.10 *number of rope turns*—the total contact angle between the rope and the cathead at the beginning of the operator's rope slackening to drop the hammer, divided by 360° (see Fig. 1).

3.11 *sampling rods*—rods that connect the drive-weight assembly to the sampler. Drill rods are often used for this purpose.

3.12 *SPT*—abbreviation for Standard Penetration Test, a term by which engineers commonly refer to this method.

4. Significance and Use

4.1 This method provides a soil sample for identification purposes and for laboratory tests appropriate for soil obtained from a sampler that may produce large shear strain disturbance in the sample.

4.2 This method is used extensively in a great variety of geotechnical exploration projects. Many local correlations and widely published correlations which relate SPT blowcount, or *N*-value, and the engineering behavior of earthworks and foundations are available.

5. Apparatus

5.1 *Drilling Equipment*—Any drilling equipment that provides at the time of sampling a suitably clean open hole before insertion of the sampler and ensures that the penetration test is performed on undisturbed soil shall be acceptable. The following pieces of equipment have proven to be suitable for advancing a borehole in some subsurface conditions.

5.1.1 *Drag, Chopping, and Fishtail Bits*, less than 6.5 in. (162 mm) and greater than 2.2 in. (56 mm) in diameter may be used in conjuction with open-hole rotary drilling or casing-advancement drilling methods. To avoid disturbance of the underlying soil, bottom discharge bits are not permitted; only side discharge bits are permitted.

5.1.2 *Roller-Cone Bits*, less than 6.5 in. (162 mm) and greater than 2.2 in. (56 mm) in diameter may be used in

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[1] This method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.02 on Sampling and Related Field Testing for Soil Investigations.

Current edition approved Sept. 11, 1984. Published November 1984. Originally published as D 1586 – 58 T. Last previous edition D 1586 – 67 (1974).

[2] *Annual Book of ASTM Standards*, Vol 04.08.

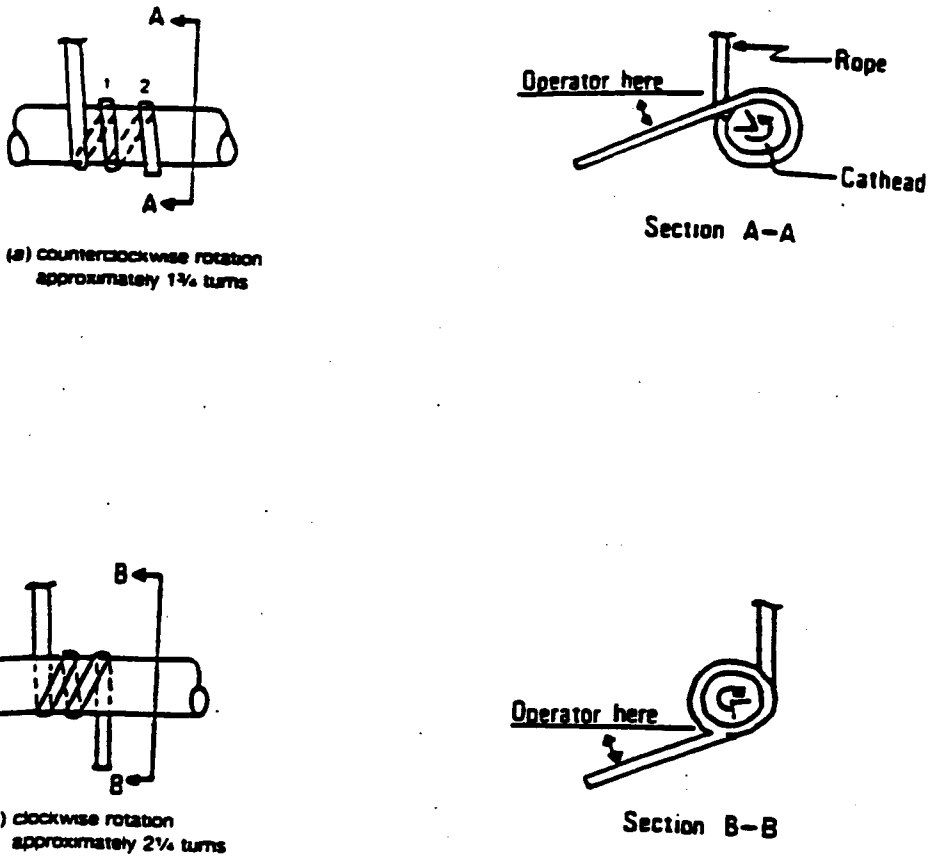


FIG. 1 Definitions of the Number of Rope Turns and the Angle for (a) Counterclockwise Rotation and (b) Clockwise Rotation of the Cathead

conjunction with open-hole rotary drilling or casing-advancement drilling methods if the drilling fluid discharge is deflected.

5.1.3 *Hollow-Stem Continuous Flight Augers*, with or without a center bit assembly, may be used to drill the boring. The inside diameter of the hollow-stem augers shall be less than 6.5 in. (162 mm) and greater than 2.2 in. (56 mm).

5.1.4 *Solid, Continuous Flight, Bucket and Hand Augers*, less than 6.5 in. (162 mm) and greater than 2.2 in. (56 mm) in diameter may be used if the soil on the side of the boring does not cave onto the sampler or sampling rods during sampling.

5.2 *Sampling Rods*—Flush-joint steel drill rods shall be used to connect the split-barrel sampler to the drive-weight assembly. The sampling rod shall have a stiffness (moment of inertia) equal to or greater than that of parallel wall "A" rod (a steel rod which has an outside diameter of 1⅝ in. (41.2 mm) and an inside diameter of 1⅛ in. (28.5 mm).

NOTE 1—Recent research and comparative testing indicates the type rod used, with stiffness ranging from "A" size rod to "N" size rod, will usually have a negligible effect on the *N*-values to depths of at least 100 ft (30 m).

5.3 *Split-Barrel Sampler*—The sampler shall be constructed with the dimensions indicated in Fig. 2. The driving shoe shall be of hardened steel and shall be replaced or repaired when it becomes dented or distorted. The use of liners to produce a constant inside diameter of 1⅜ in. (35 mm) is permitted, but shall be noted on the penetration record if used. The use of a sample retainer basket is permitted, and should also be noted on the penetration record if used.

NOTE 2—Both theory and available test data suggest that *N*-values may increase between 10 to 30 % when liners are used.

5.4 *Drive-Weight Assembly*:

5.4.1 *Hammer and Anvil*—The hammer shall weigh 140 ± 2 lb (63.5 ± 1 kg) and shall be a solid rigid metallic mass. The hammer shall strike the anvil and make steel on steel contact when it is dropped. A hammer fall guide permitting a free fall shall be used. Hammers used with the cathead and rope method shall have an unimpeded overlift capacity of at least 4 in. (100 mm). For safety reasons, the use of a hammer assembly with an internal anvil is encouraged.

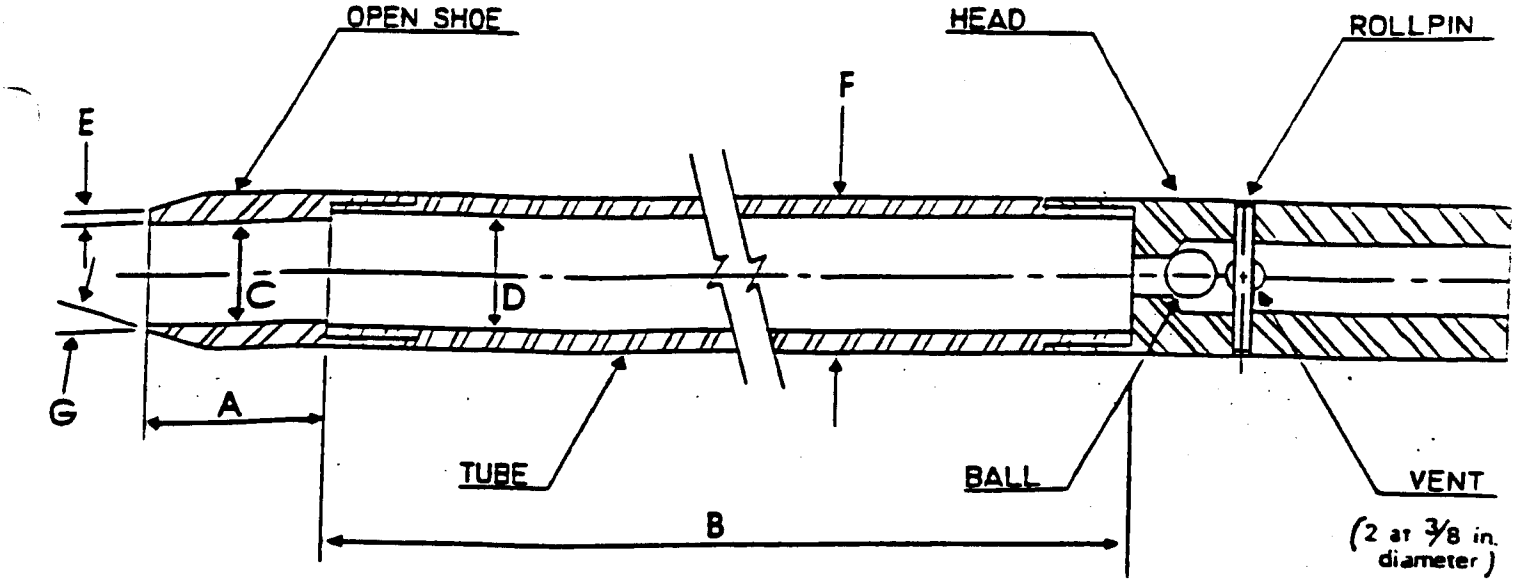
NOTE 3—It is suggested that the hammer fall guide be permanently marked to enable the operator or inspector to judge the hammer drop height.

5.4.2 *Hammer Drop System*—Rope-cathead, trip, semi-automatic, or automatic hammer drop systems may be used, providing the lifting apparatus will not cause penetration of the sampler while re-engaging and lifting the hammer.

5.5 *Accessory Equipment*—Accessories such as labels, sample containers, data sheets, and groundwater level measuring devices shall be provided in accordance with the requirements of the project and other ASTM standards.

6. Drilling Procedure

6.1 The boring shall be advanced incrementally to permit



A = 1.0 to 2.0 in. (25 to 50 mm)
B = 18.0 to 30.0 in. (0.457 to 0.762 m)
C = 1.375 ± 0.005 in. (34.93 ± 0.13 mm)
D = 1.50 ± 0.05 − 0.00 in. (38.1 ± 1.3 − 0.0 mm)
E = 0.10 ± 0.02 in. (2.54 ± 0.25 mm)
F = 2.00 ± 0.05 − 0.00 in. (50.8 ± 1.3 − 0.0 mm)
G = 16.0° to 23.0°

The 1½ in. (38 mm) inside diameter split barrel may be used with a 16-gage wall thickness split liner. The penetrating end of the drive shoe may be slightly rounded. Metal or plastic retainers may be used to retain soil samples.

FIG. 2 Split-Barrel Sampler

intermittent or continuous sampling. Test intervals and locations are normally stipulated by the project engineer or geologist. Typically, the intervals selected are 5 ft (1.5 mm) or less in homogeneous strata with test and sampling locations at every change of strata.

6.2 Any drilling procedure that provides a suitably clean and stable hole before insertion of the sampler and assures that the penetration test is performed on essentially undisturbed soil shall be acceptable. Each of the following procedures have proven to be acceptable for some subsurface conditions. The subsurface conditions anticipated should be considered when selecting the drilling method to be used.

6.2.1 Open-hole rotary drilling method.

6.2.2 Continuous flight hollow-stem auger method.

6.2.3 Wash boring method.

6.2.4 Continuous flight solid auger method.

6.3 Several drilling methods produce unacceptable borings. The process of jetting through an open tube sampler and then sampling when the desired depth is reached shall not be permitted. The continuous flight solid auger method shall not be used for advancing the boring below a water table or below the upper confining bed of a confined non-cohesive stratum that is under artesian pressure. Casing may not be advanced below the sampling elevation prior to sampling. Advancing a boring with bottom discharge bits is not permissible. It is not permissible to advance the boring for subsequent insertion of the sampler solely by means of previous sampling with the SPT sampler.

6.4 The drilling fluid level within the boring or hollow-stem augers shall be maintained at or above the in situ groundwater level at all times during drilling, removal of drill rods, and sampling.

7. Sampling and Testing Procedure

7.1 After the boring has been advanced to the desired sampling elevation and excessive cuttings have been removed, prepare for the test with the following sequence of operations.

7.1.1 Attach the split-barrel sampler to the sampling rods and lower into the borehole. Do not allow the sampler to drop onto the soil to be sampled.

7.1.2 Position the hammer above and attach the anvil to the top of the sampling rods. This may be done before the sampling rods and sampler are lowered into the borehole.

7.1.3 Rest the dead weight of the sampler, rods, anvil, and drive weight on the bottom of the boring and apply a seating blow. If excessive cuttings are encountered at the bottom of the boring, remove the sampler and sampling rods from the boring and remove the cuttings.

7.1.4 Mark the drill rods in three successive 6-in. (0.15-m) increments so that the advance of the sampler under the impact of the hammer can be easily observed for each 6-in. (0.15-m) increment.

7.2 Drive the sampler with blows from the 140-lb (63.5-kg) hammer and count the number of blows applied in each 6-in. (0.15-m) increment until one of the following occurs:

7.2.1 A total of 50 blows have been applied during any one of the three 6-in. (0.15-m) increments described in 7.1.4.

7.2.2 A total of 100 blows have been applied.

7.2.3 There is no observed advance of the sampler during the application of 10 successive blows of the hammer.

7.2.4 The sampler is advanced the complete 18 in. (0.45 m) without the limiting blow counts occurring as described in 7.2.1, 7.2.2, or 7.2.3.

7.3 Record the number of blows required to effect each 6 in. (0.15 m) of penetration or fraction thereof. The first 6 in. is considered to be a seating drive. The sum of the number of blows required for the second and third 6 in. of penetration is termed the "standard penetration resistance," or the "N-value." If the sampler is driven less than 18 in. (0.45 m), as permitted in 7.2.1, 7.2.2, or 7.2.3, the number of blows per each complete 6-in. (0.15-m) increment and per each partial increment shall be recorded on the boring log. For partial increments, the depth of penetration shall be reported to the nearest 1 in. (25 mm), in addition to the number of blows. If the sampler advances below the bottom of the boring under the static weight of the drill rods or the weight of the drill rods plus the static weight of the hammer, this information should be noted on the boring log.

7.4 The raising and dropping of the 140-lb (63.5-kg) hammer shall be accomplished using either of the following two methods:

7.4.1 By using a trip, automatic, or semi-automatic hammer drop system which lifts the 140-lb (63.5-kg) hammer and allows it to drop 30 ± 1.0 in. (0.76 m ± 25 mm) unimpeded.

7.4.2 By using a cathead to pull a rope attached to the hammer. When the cathead and rope method is used the system and operation shall conform to the following:

7.4.2.1 The cathead shall be essentially free of rust, oil, or grease and have a diameter in the range of 6 to 10 in. (150 to 250 mm).

7.4.2.2 The cathead should be operated at a minimum speed of rotation of 100 RPM, or the approximate speed of rotation shall be reported on the boring log.

7.4.2.3 No more than 2¼ rope turns on the cathead may be used during the performance of the penetration test, as shown in Fig. 1.

NOTE 4—The operator should generally use either 1¾ or 2¼ rope turns, depending upon whether or not the rope comes off the top (1¾ turns) or the bottom (2¼ turns) of the cathead. It is generally known and accepted that 2¾ or more rope turns considerably impedes the fall of the hammer and should not be used to perform the test. The cathead rope should be maintained in a relatively dry, clean, and unfrayed condition.

7.4.2.4 For each hammer blow, a 30-in. (0.76-m) lift and drop shall be employed by the operator. The operation of pulling and throwing the rope shall be performed rhythmically without holding the rope at the top of the stroke.

7.5 Bring the sampler to the surface and open. Record the percent recovery or the length of sample recovered. Describe the soil samples recovered as to composition, color, stratification, and condition, then place one or more representative portions of the sample into sealable moisture-proof containers (jars) without ramming or distorting any apparent stratification. Seal each container to prevent evaporation of soil moisture. Affix labels to the containers bearing job designation, boring number, sample depth, and the blow count per 6-in. (0.15-m) increment. Protect the sample against extreme temperature changes. If there is a soil change within the sampler, make a jar for each stratum and note its location in the sampler barrel.

8. Report

8.1 Drilling information shall be recorded in the field and shall include the following:

8.1.1 Name and location of job,

8.1.2 Names of crew,

8.1.3 Type and make of drilling machine,

8.1.4 Weather conditions,

8.1.5 Date and time of start and finish of boring,

8.1.6 Boring number and location (station and coordinates, if available and applicable),

8.1.7 Surface elevation, if available,

8.1.8 Method of advancing and cleaning the boring,

8.1.9 Method of keeping boring open,

8.1.10 Depth of water surface and drilling depth at the time of a noted loss of drilling fluid, and time and date when reading or notation was made,

8.1.11 Location of strata changes,

8.1.12 Size of casing, depth of cased portion of boring,

8.1.13 Equipment and method of driving sampler,

8.1.14 Type sampler and length and inside diameter of barrel (note use of liners),

8.1.15 Size, type, and section length of the sampling rods, and

8.1.16 Remarks.

8.2 Data obtained for each sample shall be recorded in the field and shall include the following:

8.2.1 Sample depth and, if utilized, the sample number,

8.2.2 Description of soil,

8.2.3 Strata changes within sample,

8.2.4 Sampler penetration and recovery lengths, and

8.2.5 Number of blows per 6-in. (0.15-m) or partial increment.

9. Precision and Bias

9.1 Variations in *N*-values of 100 % or more have been observed when using different standard penetration test apparatus and drillers for adjacent borings in the same soil formation. Current opinion, based on field experience, indicates that when using the same apparatus and driller, *N*-values in the same soil can be reproduced with a coefficient of variation of about 10 %.

9.2 The use of faulty equipment, such as an extremely massive or damaged anvil, a rusty cathead, a low speed cathead, an old, oily rope, or massive or poorly lubricated rope sheaves can significantly contribute to differences in *N*-values obtained between operator-drill rig systems.

9.3 The variability in *N*-values produced by different drill rigs and operators may be reduced by measuring that part of the hammer energy delivered into the drill rods from the sampler and adjusting *N* on the basis of comparative energies. A method for energy measurement and *N*-value adjustment is currently under development.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

APPENDIX H

HISTORICAL ANALYTICAL RESULTS

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #					
							MW-14 (3-5) 11/6/96 AOI # 1	PFB-25 (6.3-7.8) 4/24/97 AOI # 1	PFB-25 (8-10) 4/24/97 AOI # 1	PFB-26 (10-12) 4/25/97 AOI # 1	PFB-26 (8-10) 4/25/97 AOI # 1	PFW-6 (3-5) 3/18/97 AOI # 1
General Chemistry												
Cyanide (total) (ug/Kg)	-	4000	250000	-	-	250000	NA	NA	NA	NA	NA	NA
Total Solids (%)	-	-	-	-	-	-	NA	88.87	88.47	91.05	97.77	87.17
Diesel Fuel (mg/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
Petroleum hydrocarbons (ug/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
Metals (ug/Kg)												
Arsenic	5800	23000	100000	-	-	910000	NA	NA	NA	NA	NA	6000
Barium	75000	1300000	320000000	-	-	150000000	NA	NA	NA	NA	NA	49000
Cadmium	1200	6000	4500000	-	-	2200000	NA	NA	NA	NA	NA	700
Chromium	18000	30000	22000000	-	-	-	NA	NA	NA	NA	NA	15000
Copper	32000	160000000	170000000	-	-	59000000	NA	NA	NA	NA	NA	12000
Lead	21000	1000	900000	-	-	44000000	NA	NA	NA	NA	NA	16000
Mercury	130	1700	1400000	-	-	-	NA	NA	NA	NA	NA	ND (100)
Selenium	410	4000	23000000	-	-	59000000	NA	NA	NA	NA	NA	ND (500)
Silver	1000	13000	21000000	-	-	2900000	NA	NA	NA	NA	NA	ND (500)
Zinc	47000	5000000	1000000000	-	-	-	NA	NA	NA	NA	NA	40000
PCBs (ug/Kg)												
Aroclor-1016	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1221	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1232	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1242	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1248	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1254	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1260	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Total PCBs	-	-	9900	16000000	820000	6500000	NA	NA	NA	NA	NA	ND (330)
Semi - Volatiles (ug/Kg)												
1,2,4-Trichlorobenzene	-	4200	1100000	1100000	34000000	11000000000	NA	NA	NA	NA	NA	NA
2,4,6-Trichlorophenol	-	45000	14000000	-	-	1300000000	NA	NA	NA	NA	NA	NA
2,4-Dichlorophenol	-	7700	10000000	-	-	2300000000	NA	NA	NA	NA	NA	NA
2,4-Dimethylphenol	-	20000	230000000	-	-	2100000000	NA	NA	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
2,4-Dinitrotoluene	-	15000	220000	-	-	20000000	NA	NA	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
2-Chloronaphthalene	-	1800000	190000000	-	-	-	NA	NA	NA	NA	NA	NA
2-Chlorophenol	-	2600	4600000	-	-	-	NA	NA	NA	NA	NA	NA
2-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
2-Nitrophenol	-	1200	13000000	-	-	-	NA	NA	NA	NA	NA	NA
3,3-Dichlorobenzidine	-	2000	55000	-	-	8200000	NA	NA	NA	NA	NA	NA
3-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	-	1700	1600000	-	-	-	NA	NA	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
4-Chloro-3-methylphenol	-	16000	15000000	-	-	-	NA	NA	NA	NA	NA	NA
4-Chloroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
4-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA

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4-Nitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
Acenaphthene	-	870000	810000000	350000000	97000000	6200000000	NA	NA	NA	NA	NA	ND (330)
Acenaphthylene	-	8500	16000000	3000000	2700000	1000000000	NA	NA	NA	NA	NA	ND (330)
Acetonitrile	-	8000	14000000	22000000	11000000	10000000000	NA	NA	NA	NA	NA	NA
Aniline	-	12000	4500000	-	-	29000000	NA	NA	NA	NA	NA	NA
Anthracene	-	41000	1000000000	1000000000	1600000000	29000000000	NA	NA	NA	NA	NA	ND (330)
Benzidine	-	1000	1000	-	-	59,000	NA	NA	NA	NA	NA	NA
Benzo(a)anthracene	-	-	210000	-	-	-	NA	NA	NA	NA	NA	ND (330)
Benzo(a)pyrene	-	-	21000	-	-	1900000	NA	NA	NA	NA	NA	ND (330)
Benzo(b)fluoranthene	-	-	210000	-	-	-	NA	NA	NA	NA	NA	ND (330)
Benzo(g,h,i)perylene	-	-	16000000	-	-	350000000	NA	NA	NA	NA	NA	ND (330)
Benzo(k)fluoranthene	-	-	2100000	-	-	-	NA	NA	NA	NA	NA	NA
bis(2-Chloroethoxy)methane	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
bis(2-Chloroethyl)ether	-	330	23000	44000	13000	12000000	NA	NA	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	-	-	10000000	-	-	8900000000	NA	NA	NA	NA	NA	NA
Butyl benzylphthalate	-	310000	310000	-	-	21000000000	NA	NA	NA	NA	NA	ND (330)
Chrysene	-	-	21000000	-	-	-	NA	NA	NA	NA	NA	ND (330)
Dibenzo(a,h)anthracene	-		21000	-	-	-	NA	NA	NA	NA	NA	NA
Dibenzofuran	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
Diethyl phthalate	-	320000	740000	-	-	1500000000	NA	NA	NA	NA	NA	NA
Dimethyl phthalate	-	790000	790000	-	-	1500000000	NA	NA	NA	NA	NA	NA
Di-n-butylphthalate	-	760000	760000	-	-	1500000000	NA	NA	NA	NA	NA	NA
Di-n-octyl phthalate	-	140000000	81000000	-	-	-	NA	NA	NA	NA	NA	NA
Fluoranthene	-	720000	540000000	1000000000	880000000	4100000000	NA	NA	NA	NA	NA	ND (330)
Fluorene	-	890000	540000000	1000000000	150000000	4100000000	NA	NA	NA	NA	NA	ND (330)
Hexachlorobenzene	-	1800	94000	220000	56000	85000000	NA	NA	NA	NA	NA	NA
Hexachlorobutadiene	-	77000	350000	350000	460000	180000000	NA	NA	NA	NA	NA	NA
Hexachlorocyclopentadiene	-	36000	81000	-	-	-	NA	NA	NA	NA	NA	NA
Hexachloroethane	-	69000	1800000	370000	1400000	100000000	NA	NA	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	-	-	210000	-	-	-	NA	NA	NA	NA	NA	ND (330)
Isophorone	-	74000	2400000	-	-	8200000000	NA	NA	NA	NA	NA	NA
Naphthalene	-	50000	160000000	77000000	59000000	15000000000	NA	NA	NA	NA	NA	ND (330)
Nitrobenzene	-	330	340000	490000	4600000	1500000000	NA	NA	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	-	330	3500	-	-	2000000	NA	NA	NA	NA	NA	NA
N-Nitrosodiphenylamine	-	14000	5100000	-	-	-	NA	NA	NA	NA	NA	NA
Octachlorocyclopentene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
Pentachlorophenol	-	3200	63000	-	-	130000000	NA	NA	NA	NA	NA	ND (330)
Phenanthrene	-	34000	16000000	28000000	150000	59000000	NA	NA	NA	NA	NA	NA
Phenol	-	260000	12000000	-	-	18000000000	NA	NA	NA	NA	NA	ND (330)
Pyrene	-	470000	340000000	1000000000	770000000	2900000000	NA	NA	NA	NA	NA	ND (330)
Volatiles (ug/Kg)												
1,1,1-Trichloroethane	-	4000	460000	460000	4500000	29000000000	ND	ND (10)	ND (10)	ND (10)	ND (10)	ND (24)
1,1,2,2-Tetrachloroethane	-	340	120000	23000	34000	68000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (29)
1,1,2-Trichloroethane	-	100	440000	24000	57000	250000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (6.3)
1,1-Dichloroethane	-	50000	790000	790000	36000000	240000000000	1.7	ND (10)	ND (10)	ND (10)	ND (10)	ND (14)
1,1-Dichloroethene	-	140	580000	330	3700	78000000	ND	ND (10)	ND (10)	ND (10)	ND (10)	ND (13)

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels (2)	Industrial Drinking Water Protection Criteria (2)	Industrial Direct Contact Criteria (2)	Soil Volatilization to Indoor Air Inhalation Criteria (2)	Volatile Soil Inhalation Criteria (2)	Particulate Soil Inhalation Criteria (2)	Sample Location (depth ft. bgs) / Sample Date / AOI #					
							MW-14 (3-5) 11/6/96 AOI # 1	PFB-25 (6.3-7.8) 4/24/97 AOI # 1	PFB-25 (8-10) 4/24/97 AOI # 1	PFB-26 (10-12) 4/25/97 AOI # 1	PFB-26 (8-10) 4/25/97 AOI # 1	PFW-6 (3-5) 3/18/97 AOI # 1
1,2-Dibromoethane (EDB)	-	10	290	3600	5800	18000000	NA	NA	NA	NA	NA	NA
1,2-Dichlorobenzene	-	13000	210000	210000	46000000	44000000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (12)
1,2-Dichloroethane	-	100	270000	11000	21000	150000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (12)
1,2-Dichloropropane	-	100	360000	7400	30000	120000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)
1,3-Dichlorobenzene	-	18000	200000	-	-	-	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (21)
1,4-Dichlorobenzene	-	1700	1000000	100000	260000	570000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (19)
2-Butanone	-	760000	27000000	27000000	35000000	29000000000	NA	NA	NA	NA	NA	NA
2-Chloroethyl vinyl ether	-	-	-	-	-	-	NA	ND (100)	ND (100)	ND (100)	ND (100)	ND (25)
2-Hexanone	-	58000	2500000	1800000		1200000000	NA	NA	NA	NA	NA	NA
4-Methyl-2-pentanone	-	100000	2700000	2700000	53000000	60000000000	NA	NA	NA	NA	NA	NA
Acetone	-	42000	74000000	110000000	160000000	170000000000	NA	NA	NA	NA	NA	NA
Benzene	-	100	400000	8400	45000	470000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)
Bromodichloromethane	-	2000	400000	6400	31000	110000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (17)
Bromoform	-	2000	870000	770000	3100000	3600000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (18)
Bromomethane	-	580	1000000	1600	13000	150000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (25)
Carbon disulfide	-	46000	280000	140000	1600000	21000000000	NA	NA	NA	NA	NA	NA
Carbon tetrachloride	-	100	190000	990	12000	170000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (12)
Chlorobenzene	-	2000	260000	220000	920000	2100000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (30)
Chloroethane	-	18000	970000	970000	36000000	290000000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (25)
Chloroform	-	2000	1500000	38000	150000	1600000000	ND	ND (10)	ND (10)	ND (10)	ND (10)	ND (7.8)
Chloromethane	-	5400	1100000	12000	140000	6100000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (25)
cis-1,2-Dichloroethene	-	1400	640000	640000	47000000	230000000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	110
cis-1,3-Dichloropropene	-	-	-	-	-	-	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (9.1)
Dibromochloromethane	-	2000	300000	21000	80000	160000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (25)
Dichlorodifluoromethane (CFC-12)	-	270000	1000000	1000000	63000000	1500000000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (25)
Ethylbenzene	-	1500	140000	140000	11000000	29000000000	ND	ND (10)	ND (10)	ND (10)	ND (10)	150
Ethylene oxide	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
m&p-Xylene	-	-	-	-	-	-	NA	ND (30)	ND (30)	ND (30)	ND (30)	ND (30)
Methyl Tert Butyl Ether	-	800	6000000	6000000	31000000	88000000000	NA	NA	NA	NA	NA	NA
Methylene chloride	-	100	2300000	240000	700000	8300000000	11.4	ND (10)	ND (10)	ND (10)	ND (10)	ND (19)
o-Xylene	-	-	-	-	-	-	NA	ND (30)	ND (30)	ND (30)	ND (30)	ND (30)
Styrene	-	2700	520000	520000	3200000	6600000000	NA	NA	NA	NA	NA	NA
Tetrachloroethene	-	100	88000	60000	600000	6800000000	1.3	ND (10)	ND (10)	ND (10)	ND (10)	ND (17)
Toluene	-	16000	250000	250000	3300000	12000000000	1	ND (10)	ND (10)	ND (10)	ND (10)	ND (9)
trans-1,2-Dichloroethene	-	2000	1400000	1400000	37000000	230000000000	4.7	ND (10)	ND (10)	ND (10)	ND (10)	5.2
trans-1,3-Dichloropropene	-	-	-	-	-	-	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (6.3)
Trichloroethene	-	100	500000	37000	260000	2300000000	2210	ND (10)	ND (10)	ND (10)	ND (10)	1600
Trichlorofluoromethane (CFC-11)	-	150000	560000	560000	110000000	1700000000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (13)
Vinyl chloride	-	40	11000	150	1500	47000000	NA	ND (10)	ND (10)	ND (10)	ND (10)	ND (25)
Xylene (total)	-	5600	150000	150000	54000000	130000000000	ND	NA	NA	NA	NA	NA

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #							
							SB-14 (1-3) 11/6/96 AOI # 1	SB-15 (1-3) 11/6/96 AOI # 1	MW-13 (4-6) 11/6/96 AOI # 2	PFW-11 (0.5-1.5) 3/20/97 AOI # 3	M-1 (6-8) 11/6/96 AOI # 7	M-1 (8-10) 11/6/96 AOI # 7	M-1 (8-10) 11/6/96 AOI # 7	P-28 11/6/96 AOI # 7
General Chemistry														
Cyanide (total) (ug/Kg)	-	4000	250000	-	-	250000	NA	NA	NA	NA	NA	NA	NA	NA
Total Solids (%)	-	-	-	-	-	-	NA	NA	NA	86.44	NA	NA	NA	NA
Diesel Fuel (mg/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
Petroleum hydrocarbons (ug/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA	505000
Metals (ug/Kg)														
Arsenic	5800	23000	100000	-	-	910000	NA	NA	NA	10000	6810	6800	4300	NA
Barium	75000	1300000	320000000	-	-	150000000	NA	NA	NA	74000	72600	100000	43400	NA
Cadmium	1200	6000	4500000	-	-	2200000	NA	NA	NA	810	ND	170	ND	NA
Chromium	18000	30000	22000000	-	-	-	NA	NA	NA	20000	13700	9300	12800	NA
Copper	32000	160000000	170000000	-	-	59000000	NA	NA	NA	16000	ND	21000	ND	NA
Lead	21000	1000	900000	-	-	44000000	NA	NA	NA	18000	8070	11000	8980	NA
Mercury	130	1700	1400000	-	-	-	NA	NA	NA	ND (100)	ND	ND (100)	ND	NA
Selenium	410	4000	23000000	-	-	59000000	NA	NA	NA	ND (500)	ND	540	ND	NA
Silver	1000	13000	21000000	-	-	2900000	NA	NA	NA	900	ND	ND (500)	ND	NA
Zinc	47000	5000000	1000000000	-	-	-	NA	NA	NA	52000	ND	41000	ND	NA
PCBs (ug/Kg)														
Aroclor-1016	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
Aroclor-1221	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
Aroclor-1232	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
Aroclor-1242	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
Aroclor-1248	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
Aroclor-1254	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA	ND
Aroclor-1260	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA	ND
Total PCBs	-	-	9900	16000000	820000	6500000	NA	NA	NA	NA	NA	NA	NA	ND
Semi - Volatiles (ug/Kg)														
1,2,4-Trichlorobenzene	-	4200	1100000	1100000	34000000	11000000000	NA	NA	NA	NA	NA	NA	NA	NA
2,4,6-Trichlorophenol	-	45000	14000000	-	-	1300000000	NA	NA	NA	NA	NA	NA	NA	NA
2,4-Dichlorophenol	-	7700	10000000	-	-	2300000000	NA	NA	NA	NA	NA	NA	NA	NA
2,4-Dimethylphenol	-	20000	230000000	-	-	2100000000	NA	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrotoluene	-	15000	220000	-	-	20000000	NA	NA	NA	NA	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
2-Chloronaphthalene	-	1800000	190000000	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
2-Chlorophenol	-	2600	4600000	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
2-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
2-Nitrophenol	-	1200	13000000	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
3,3-Dichlorobenzidine	-	2000	55000	-	-	8200000	NA	NA	NA	NA	NA	NA	NA	NA
3-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	-	1700	1600000	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
4-Chloro-3-methylphenol	-	16000	15000000	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
4-Chloroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
4-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #							
							SB-14 (1-3) 11/6/96 AOI # 1	SB-15 (1-3) 11/6/96 AOI # 1	MW-13 (4-6) 11/6/96 AOI # 2	PFW-11 (0.5-1.5) 3/20/97 AOI # 3	M-1 (6-8) 11/6/96 AOI # 7	M-1 (8-10) 11/6/96 AOI # 7	M-1 (8-10) 11/6/96 AOI # 7	P-28 11/6/96 AOI # 7
4-Nitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
Acenaphthene	-	870000	810000000	350000000	97000000	6200000000	NA	NA	NA	NA	NA	NA	NA	NA
Acenaphthylene	-	8500	16000000	3000000	2700000	1000000000	NA	NA	NA	NA	NA	NA	NA	NA
Acetonitrile	-	8000	14000000	22000000	11000000	10000000000	NA	NA	NA	NA	NA	NA	NA	NA
Aniline	-	12000	4500000	-	-	29000000	NA	NA	NA	NA	NA	NA	NA	NA
Anthracene	-	41000	1000000000	1000000000	1600000000	29000000000	NA	NA	NA	NA	NA	NA	NA	NA
Benzidine	-	1000	1000	-	-	59,000	NA	NA	NA	NA	NA	NA	NA	NA
Benzo(a)anthracene	-	-	210000	-	-	-	NA	NA	NA	NA	NA	NA	NA	ND
Benzo(a)pyrene	-	-	21000	-	-	1900000	NA	NA	NA	NA	NA	NA	NA	ND
Benzo(b)fluoranthene	-	-	210000	-	-	-	NA	NA	NA	NA	NA	NA	NA	ND
Benzo(g,h,i)perylene	-	-	16000000	-	-	350000000	NA	NA	NA	NA	NA	NA	NA	NA
Benzo(k)fluoranthene	-	-	2100000	-	-	-	NA	NA	NA	NA	NA	NA	NA	ND
bis(2-Chloroethoxy)methane	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroethyl)ether	-	330	23000	44000	13000	12000000	NA	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	-	-	10000000	-	-	8900000000	NA	NA	NA	NA	NA	NA	NA	NA
Butyl benzylphthalate	-	310000	310000	-	-	21000000000	NA	NA	NA	NA	NA	NA	NA	NA
Chrysene	-	-	21000000	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
Dibenzo(a,h)anthracene	-		21000	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
Dibenzofuran	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
Diethyl phthalate	-	320000	740000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA	NA
Dimethyl phthalate	-	790000	790000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA	NA
Di-n-butylphthalate	-	760000	760000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA	NA
Di-n-octyl phthalate	-	140000000	81000000	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
Fluoranthene	-	720000	540000000	1000000000	880000000	4100000000	NA	NA	NA	NA	NA	NA	NA	NA
Fluorene	-	890000	540000000	1000000000	150000000	4100000000	NA	NA	NA	NA	NA	NA	NA	NA
Hexachlorobenzene	-	1800	94000	220000	56000	85000000	NA	NA	NA	NA	NA	NA	NA	NA
Hexachlorobutadiene	-	77000	350000	350000	460000	180000000	NA	NA	NA	NA	NA	NA	NA	NA
Hexachlorocyclopentadiene	-	36000	81000	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
Hexachloroethane	-	69000	1800000	370000	1400000	100000000	NA	NA	NA	NA	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	-	-	210000	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
Isophorone	-	74000	2400000	-	-	8200000000	NA	NA	NA	NA	NA	NA	NA	NA
Naphthalene	-	50000	160000000	77000000	59000000	15000000000	NA	NA	NA	NA	NA	NA	NA	NA
Nitrobenzene	-	330	340000	490000	4600000	1500000000	NA	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	-	330	3500	-	-	2000000	NA	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodiphenylamine	-	14000	5100000	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
Octachlorocyclopentene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
Pentachlorophenol	-	3200	63000	-	-	130000000	NA	NA	NA	NA	NA	NA	NA	NA
Phenanthrene	-	34000	16000000	28000000	150000	59000000	NA	NA	NA	NA	NA	NA	NA	NA
Phenol	-	260000	12000000	-	-	18000000000	NA	NA	NA	NA	NA	NA	NA	NA
Pyrene	-	470000	340000000	1000000000	770000000	2900000000	NA	NA	NA	NA	NA	NA	NA	NA
Volatiles (ug/Kg)														
1,1,1-Trichloroethane	-	4000	460000	460000	4500000	29000000000	ND	ND	10.4	ND (10)	ND	ND (10)	ND	NA
1,1,2,2-Tetrachloroethane	-	340	120000	23000	34000	68000000	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
1,1,2-Trichloroethane	-	100	440000	24000	57000	250000000	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
1,1-Dichloroethane	-	50000	790000	790000	36000000	240000000000	ND	ND	33.8	ND (10)	ND	ND (10)	ND	NA
1,1-Dichloroethene	-	140	580000	330	3700	78000000	ND	ND	ND	ND (10)	ND	ND (10)	ND	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels (2)	Industrial Drinking Water Protection Criteria (2)	Industrial Direct Contact Criteria (2)	Soil Volatilization to Indoor Air Inhalation Criteria (2)	Volatile Soil Inhalation Criteria (2)	Particulate Soil Inhalation Criteria (2)	Sample Location (depth ft. bgs) / Sample Date / AOI #							
							SB-14 (1-3) 11/6/96 AOI # 1	SB-15 (1-3) 11/6/96 AOI # 1	MW-13 (4-6) 11/6/96 AOI # 2	PFW-11 (0.5-1.5) 3/20/97 AOI # 3	M-1 (6-8) 11/6/96 AOI # 7	M-1 (8-10) 11/6/96 AOI # 7	M-1 (8-10) 11/6/96 AOI # 7	P-28 11/6/96 AOI # 7
1,2-Dibromoethane (EDB)	-	10	290	3600	5800	18000000	NA	NA	NA	NA	NA	NA	NA	NA
1,2-Dichlorobenzene	-	13000	210000	210000	46000000	44000000000	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
1,2-Dichloroethane	-	100	270000	11000	21000	150000000	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
1,2-Dichloropropane	-	100	360000	7400	30000	120000000	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
1,3-Dichlorobenzene	-	18000	200000	-	-	-	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
1,4-Dichlorobenzene	-	1700	1000000	100000	260000	570000000	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
2-Butanone	-	760000	27000000	27000000	35000000	29000000000	NA	NA	NA	NA	NA	ND (100)	NA	NA
2-Chloroethyl vinyl ether	-	-	-	-	-	-	NA	NA	NA	ND (100)	NA	NA	NA	NA
2-Hexanone	-	58000	2500000	1800000		1200000000	NA	NA	NA	NA	NA	ND (100)	NA	NA
4-Methyl-2-pentanone	-	100000	2700000	2700000	53000000	60000000000	NA	NA	NA	NA	NA	ND (100)	NA	NA
Acetone	-	42000	74000000	110000000	160000000	170000000000	NA	NA	NA	NA	NA	ND (100)	NA	NA
Benzene	-	100	400000	8400	45000	470000000	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
Bromodichloromethane	-	2000	400000	6400	31000.	110000000	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
Bromoform	-	2000	870000	770000	3100000	3600000000	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
Bromomethane	-	580	1000000	1600	13000	150000000	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
Carbon disulfide	-	46000	280000	140000	1600000	21000000000	NA	NA	NA	NA	NA	ND (100)	NA	NA
Carbon tetrachloride	-	100	190000	990	12000	170000000	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
Chlorobenzene	-	2000	260000	220000	920000	2100000000	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
Chloroethane	-	18000	970000	970000	36000000	290000000000	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
Chloroform	-	2000	1500000	38000	150000	1600000000	ND	ND	ND	ND (10)	ND	ND (10)	ND	NA
Chloromethane	-	5400	1100000	12000	140000	6100000000	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
cis-1,2-Dichloroethene	-	1400	640000	640000	47000000	230000000000	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
cis-1,3-Dichloropropene	-	-	-	-	-	-	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
Dibromochloromethane	-	2000	300000	21000	80000	160000000	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
Dichlorodifluoromethane (CFC-12)	-	270000	1000000	1000000	63000000	1500000000000	NA	NA	NA	ND (10)	NA	NA	NA	NA
Ethylbenzene	-	1500	140000	140000	11000000	29000000000	ND	ND	ND	ND (10)	1.3	ND (10)	ND	NA
Ethylene oxide	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA
m&p-Xylene	-	-	-	-	-	-	NA	NA	NA	ND (30)	NA	NA	NA	NA
Methyl Tert Butyl Ether	-	800	6000000	6000000	31000000	88000000000	NA	NA	NA	NA	NA	NA	NA	NA
Methylene chloride	-	100	2300000	240000	700000	8300000000	ND	ND	66.7	ND (10)	19.3	ND (10)	ND	NA
o-Xylene	-	-	-	-	-	-	NA	NA	NA	ND (30)	NA	NA	NA	NA
Styrene	-	2700	520000	520000	3200000	6600000000	NA	NA	NA	NA	NA	ND (10)	NA	NA
Tetrachloroethene	-	100	88000	60000	600000	6800000000	ND	1.6	ND	ND (10)	ND	ND (10)	ND	NA
Toluene	-	16000	250000	250000	3300000	12000000000	2.7	1.1	ND	ND (10)	ND	ND (10)	ND	NA
trans-1,2-Dichloroethene	-	2000	1400000	1400000	37000000	230000000000	ND	ND	ND	ND (10)	ND	ND (10)	ND	NA
trans-1,3-Dichloropropene	-	-	-	-	-	-	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
Trichloroethene	-	100	500000	37000	260000	2300000000	ND	86.3	ND	ND (10)	ND	ND (10)	ND	NA
Trichlorofluoromethane (CFC-11)	-	150000	560000	560000	110000000	1700000000000	NA	NA	NA	ND (10)	NA	NA	NA	NA
Vinyl chloride	-	40	11000	150	1500	47000000	NA	NA	NA	ND (10)	NA	ND (10)	NA	NA
Xylene (total)	-	5600	150000	150000	54000000	130000000000	ND	1	ND	NA	14	ND (30)	ND	NA

Notes:
PCBs - Polychlorinated Biphenyls.
TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.
ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.
NA - Parameter not analyzed.
(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							PFB-13 (2.9-3.5) 3/24/97 AOI # 7	PFB-14 (4.7-5.4) 3/25/97 AOI # 7	PFW-10 (2.1-2.7) 3/20/97 AOI # 7	Q-32 11/6/96 AOI # 7	Q-33 11/6/96 AOI # 7	Q-34 11/6/96 AOI # 7	BLD 63-24 11/6/96 AOI # 8
General Chemistry													
Cyanide (total) (ug/Kg)	-	4000	250000	-	-	250000	NA	NA	NA	NA	NA	NA	NA
Total Solids (%)	-	-	-	-	-	-	81.5	87.46	84.84	NA	NA	NA	NA
Diesel Fuel (mg/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Petroleum hydrocarbons (ug/Kg)	-	-	-	-	-	-	NA	NA	NA	576000	480000	NA	471000
Metals (ug/Kg)													
Arsenic	5800	23000	100000	-	-	910000	10000	10000	4400	NA	NA	NA	5150
Barium	75000	1300000	320000000	-	-	150000000	110000	55000	37000	NA	NA	NA	47300
Cadmium	1200	6000	4500000	-	-	2200000	670	860	ND (50)	NA	NA	NA	ND
Chromium	18000	30000	22000000	-	-	-	24000	17000	11000	NA	NA	NA	10800
Copper	32000	160000000	170000000	-	-	59000000	14000	17000	6300	NA	NA	NA	ND
Lead	21000	1000	900000	-	-	44000000	17000	18000	11000	NA	NA	NA	5480
Mercury	130	1700	1400000	-	-	-	ND (100)	ND (100)	ND (100)	NA	NA	NA	ND
Selenium	410	4000	23000000	-	-	59000000	ND (500)	ND (500)	ND (500)	NA	NA	NA	ND
Silver	1000	13000	21000000	-	-	2900000	ND (500)	1000	ND (500)	NA	NA	NA	ND
Zinc	47000	5000000	1000000000	-	-	-	58000	52000	31000	NA	NA	NA	ND
PCBs (ug/Kg)													
Aroclor-1016	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1221	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1232	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1242	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1248	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1254	-	-	9900	-	-	-	NA	NA	NA	ND	ND	279	ND
Aroclor-1260	-	-	9900	-	-	-	NA	NA	NA	ND	ND	ND	ND
Total PCBs	-	-	9900	16000000	820000	6500000	ND (330)	ND (330)	ND (330)	ND	ND	279	ND
Semi - Volatiles (ug/Kg)													
1,2,4-Trichlorobenzene	-	4200	1100000	1100000	34000000	11000000000	NA	NA	NA	NA	NA	NA	NA
2,4,6-Trichlorophenol	-	45000	14000000	-	-	1300000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dichlorophenol	-	7700	10000000	-	-	2300000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dimethylphenol	-	20000	230000000	-	-	2100000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrotoluene	-	15000	220000	-	-	20000000	NA	NA	NA	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Chloronaphthalene	-	1800000	190000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Chlorophenol	-	2600	4600000	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Nitrophenol	-	1200	13000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
3,3-Dichlorobenzidine	-	2000	55000	-	-	8200000	NA	NA	NA	NA	NA	NA	NA
3-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	-	1700	1600000	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chloro-3-methylphenol	-	16000	15000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chloroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							PFB-13 (2.9-3.5) 3/24/97 AOI # 7	PFB-14 (4.7-5.4) 3/25/97 AOI # 7	PFW-10 (2.1-2.7) 3/20/97 AOI # 7	Q-32 11/6/96 AOI # 7	Q-33 11/6/96 AOI # 7	Q-34 11/6/96 AOI # 7	BLD 63-24 11/6/96 AOI # 8
4-Nitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Acenaphthene	-	870000	810000000	350000000	97000000	6200000000	ND (330)	ND (330)	ND (330)	NA	NA	NA	NA
Acenaphthylene	-	8500	16000000	3000000	2700000	1000000000	ND (330)	ND (330)	ND (330)	NA	NA	NA	NA
Acetonitrile	-	8000	14000000	22000000	11000000	10000000000	NA	NA	NA	NA	NA	NA	NA
Aniline	-	12000	4500000	-	-	29000000	NA	NA	NA	NA	NA	NA	NA
Anthracene	-	41000	1000000000	1000000000	160000000	29000000000	ND (330)	ND (330)	ND (330)	NA	NA	NA	NA
Benzidine	-	1000	1000	-	-	59,000	NA	NA	NA	NA	NA	NA	NA
Benzo(a)anthracene	-	-	210000	-	-	-	ND (330)	400	ND (330)	ND	ND	32.6	NA
Benzo(a)pyrene	-	-	21000	-	-	1900000	ND (330)	ND (330)	ND (330)	ND	ND	26.1	NA
Benzo(b)fluoranthene	-	-	210000	-	-	-	ND (330)	450	ND (330)	ND	ND	40.6	NA
Benzo(g,h,i)perylene	-	-	16000000	-	-	350000000	ND (330)	ND (330)	ND (330)	NA	NA	NA	NA
Benzo(k)fluoranthene	-	-	2100000	-	-	-	ND (330)	ND (330)	ND (330)	ND	ND	13.1	NA
bis(2-Chloroethoxy)methane	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroethyl)ether	-	330	23000	44000	13000	12000000	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	-	-	10000000	-	-	8900000000	NA	NA	NA	NA	NA	NA	NA
Butyl benzylphthalate	-	310000	310000	-	-	21000000000	NA	NA	NA	NA	NA	NA	NA
Chrysene	-	-	21000000	-	-	-	ND (330)	460	ND (330)	NA	NA	NA	NA
Dibenzo(a,h)anthracene	-		21000	-	-	-	ND (330)	ND (330)	ND (330)	NA	NA	NA	NA
Dibenzofuran	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Diethyl phthalate	-	320000	740000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Dimethyl phthalate	-	790000	790000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Di-n-butylphthalate	-	760000	760000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Di-n-octyl phthalate	-	140000000	81000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Fluoranthene	-	720000	540000000	1000000000	880000000	4100000000	ND (330)	1300	ND (330)	NA	NA	NA	NA
Fluorene	-	890000	540000000	1000000000	150000000	4100000000	ND (330)	ND (330)	ND (330)	NA	NA	NA	NA
Hexachlorobenzene	-	1800	94000	220000	56000	85000000	NA	NA	NA	NA	NA	NA	NA
Hexachlorobutadiene	-	77000	350000	350000	460000	180000000	NA	NA	NA	NA	NA	NA	NA
Hexachlorocyclopentadiene	-	36000	81000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Hexachloroethane	-	69000	1800000	370000	1400000	100000000	NA	NA	NA	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	-	-	210000	-	-	-	ND (330)	ND (330)	ND (330)	NA	NA	NA	NA
Isophorone	-	74000	2400000	-	-	8200000000	NA	NA	NA	NA	NA	NA	NA
Naphthalene	-	50000	160000000	77000000	59000000	15000000000	ND (330)	ND (330)	ND (330)	NA	NA	NA	NA
Nitrobenzene	-	330	340000	490000	4600000	1500000000	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	-	330	3500	-	-	2000000	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodiphenylamine	-	14000	5100000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Octachlorocyclopentene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Pentachlorophenol	-	3200	63000	-	-	130000000	NA	NA	NA	NA	NA	NA	NA
Phenanthrene	-	34000	16000000	28000000	150000	59000000	ND (330)	1300	ND (330)	NA	NA	NA	NA
Phenol	-	260000	12000000	-	-	18000000000	NA	NA	NA	NA	NA	NA	NA
Pyrene	-	470000	340000000	1000000000	770000000	2900000000	ND (330)	1400	ND (330)	NA	NA	NA	NA
Volatiles (ug/Kg)													
1,1,1-Trichloroethane	-	4000	460000	460000	4500000	29000000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	ND
1,1,2,2-Tetrachloroethane	-	340	120000	23000	34000	68000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
1,1,2-Trichloroethane	-	100	440000	24000	57000	250000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
1,1-Dichloroethane	-	50000	790000	790000	36000000	240000000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	ND
1,1-Dichloroethene	-	140	580000	330	3700	78000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	ND

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels (2)	Industrial Drinking Water Protection Criteria (2)	Industrial Direct Contact Criteria (2)	Soil Volatilization to Indoor Air Inhalation Criteria (2)	Volatile Soil Inhalation Criteria (2)	Particulate Soil Inhalation Criteria (2)	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							PFB-13 (2.9-3.5) 3/24/97 AOI # 7	PFB-14 (4.7-5.4) 3/25/97 AOI # 7	PFW-10 (2.1-2.7) 3/20/97 AOI # 7	Q-32 11/6/96 AOI # 7	Q-33 11/6/96 AOI # 7	Q-34 11/6/96 AOI # 7	BLD 63-24 11/6/96 AOI # 8
1,2-Dibromoethane (EDB)	-	10	290	3600	5800	18000000	NA	NA	NA	NA	NA	NA	NA
1,2-Dichlorobenzene	-	13000	210000	210000	46000000	44000000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
1,2-Dichloroethane	-	100	270000	11000	21000	150000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
1,2-Dichloropropane	-	100	360000	7400	30000	120000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
1,3-Dichlorobenzene	-	18000	200000	-	-	-	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
1,4-Dichlorobenzene	-	1700	1000000	100000	260000	570000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
2-Butanone	-	760000	27000000	27000000	35000000	29000000000	NA	NA	NA	NA	NA	NA	NA
2-Chloroethyl vinyl ether	-	-	-	-	-	-	ND (100)	ND (100)	ND (100)	NA	NA	NA	NA
2-Hexanone	-	58000	2500000	1800000		1200000000	NA	NA	NA	NA	NA	NA	NA
4-Methyl-2-pentanone	-	100000	2700000	2700000	53000000	60000000000	NA	NA	NA	NA	NA	NA	NA
Acetone	-	42000	74000000	110000000	160000000	170000000000	NA	NA	NA	NA	NA	NA	NA
Benzene	-	100	400000	8400	45000	470000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
Bromodichloromethane	-	2000	400000	6400	31000	110000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
Bromoform	-	2000	870000	770000	3100000	3600000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
Bromomethane	-	580	1000000	1600	13000	150000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
Carbon disulfide	-	46000	280000	140000	1600000	21000000000	NA	NA	NA	NA	NA	NA	NA
Carbon tetrachloride	-	100	190000	990	12000	170000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
Chlorobenzene	-	2000	260000	220000	920000	2100000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
Chloroethane	-	18000	970000	970000	36000000	290000000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
Chloroform	-	2000	1500000	38000	150000	1600000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	ND
Chloromethane	-	5400	1100000	12000	140000	6100000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
cis-1,2-Dichloroethene	-	1400	640000	640000	47000000	230000000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
cis-1,3-Dichloropropene	-	-	-	-	-	-	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
Dibromochloromethane	-	2000	300000	21000	80000	160000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
Dichlorodifluoromethane (CFC-12)	-	270000	1000000	1000000	63000000	1500000000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
Ethylbenzene	-	1500	140000	140000	11000000	29000000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	ND
Ethylene oxide	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
m&p-Xylene	-	-	-	-	-	-	ND (30)	ND (30)	ND (30)	NA	NA	NA	NA
Methyl Tert Butyl Ether	-	800	6000000	6000000	31000000	88000000000	NA	NA	NA	NA	NA	NA	NA
Methylene chloride	-	100	2300000	240000	700000	8300000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	ND
o-Xylene	-	-	-	-	-	-	ND (30)	ND (30)	ND (30)	NA	NA	NA	NA
Styrene	-	2700	520000	520000	3200000	6600000000	NA	NA	NA	NA	NA	NA	NA
Tetrachloroethene	-	100	88000	60000	600000	6800000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	ND
Toluene	-	16000	250000	250000	3300000	12000000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	ND
trans-1,2-Dichloroethene	-	2000	1400000	1400000	37000000	230000000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	ND
trans-1,3-Dichloropropene	-	-	-	-	-	-	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
Trichloroethene	-	100	500000	37000	260000	2300000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	ND
Trichlorofluoromethane (CFC-11)	-	150000	560000	560000	110000000	1700000000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
Vinyl chloride	-	40	11000	150	1500	47000000	ND (10)	ND (10)	ND (10)	NA	NA	NA	NA
Xylene (total)	-	5600	150000	150000	54000000	130000000000	NA	NA	NA	NA	NA	NA	ND

Notes:
PCBs - Polychlorinated Biphenyls.
TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.
ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.
NA - Parameter not analyzed.
(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							BLD 63-25 11/6/96 AOI # 8	PFB-15 (11-11.5) 3/24/97 AOI # 8	PFB-7 (8-10) 3/18/97 AOI # 8	PFW-3 (0.8-1.5) 3/17/97 AOI # 8	F-30 (0-2) 11/6/96 AOI # 9	F-34 11/14/96 AOI # 9	F-34 11/14/96 AOI # 9
General Chemistry													
Cyanide (total) (ug/Kg)	-	4000	250000	-	-	250000	NA	NA	NA	NA	31800	NA	ND (200)
Total Solids (%)	-	-	-	-	-	-	NA	87.24	87.7	88.15	NA	88.8	NA
Diesel Fuel (mg/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Petroleum hydrocarbons (ug/Kg)	-	-	-	-	-	-	945000	NA	NA	NA	ND	NA	NA
Metals (ug/Kg)													
Arsenic	5800	23000	100000	-	-	910000	5280	9000	32000	10000	3250	4200	NA
Barium	75000	1300000	320000000	-	-	150000000	35600	75000	72000	61000	9420	36000	NA
Cadmium	1200	6000	4500000	-	-	2200000	ND	820	620	640	ND	110	NA
Chromium	18000	30000	22000000	-	-	-	9300	20000	14000	17000	5470	11000	NA
Copper	32000	160000000	170000000	-	-	59000000	ND	15000	11000	18000	ND	13000	NA
Lead	21000	1000	900000	-	-	44000000	4500	18000	15000	13000	ND	7200	NA
Mercury	130	1700	1400000	-	-	-	ND	ND (100)	ND (100)	ND (100)	ND	NA	ND (100)
Selenium	410	4000	23000000	-	-	59000000	ND	ND (500)	ND (500)	ND (500)	ND	NA	520
Silver	1000	13000	21000000	-	-	2900000	ND	2800	500	ND (500)	ND	NA	ND (500)
Zinc	47000	5000000	1000000000	-	-	-	ND	43000	37000	150000	ND	40000	NA
PCBs (ug/Kg)													
Aroclor-1016	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1221	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1232	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1242	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1248	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1254	-	-	9900	-	-	-	ND	NA	NA	NA	NA	NA	NA
Aroclor-1260	-	-	9900	-	-	-	ND	NA	NA	NA	NA	NA	NA
Total PCBs	-	-	9900	16000000	820000	6500000	ND	ND (330)	ND (330)	NA	NA	NA	NA
Semi - Volatiles (ug/Kg)													
1,2,4-Trichlorobenzene	-	4200	1100000	1100000	34000000	11000000000	NA	NA	NA	NA	NA	NA	NA
2,4,6-Trichlorophenol	-	45000	14000000	-	-	1300000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dichlorophenol	-	7700	10000000	-	-	2300000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dimethylphenol	-	20000	230000000	-	-	2100000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrotoluene	-	15000	220000	-	-	20000000	NA	NA	NA	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Chloronaphthalene	-	1800000	190000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Chlorophenol	-	2600	4600000	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Nitrophenol	-	1200	13000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
3,3-Dichlorobenzidine	-	2000	55000	-	-	8200000	NA	NA	NA	NA	NA	NA	NA
3-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	-	1700	1600000	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chloro-3-methylphenol	-	16000	15000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chloroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							BLD 63-25 11/6/96 AOI # 8	PFB-15 (11-11.5) 3/24/97 AOI # 8	PFB-7 (8-10) 3/18/97 AOI # 8	PFW-3 (0.8-1.5) 3/17/97 AOI # 8	F-30 (0-2) 11/6/96 AOI # 9	F-34 11/14/96 AOI # 9	F-34 11/14/96 AOI # 9
4-Nitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Acenaphthene	-	870000	810000000	350000000	97000000	6200000000	NA	ND (330)	ND (330)	NA	NA	NA	NA
Acenaphthylene	-	8500	16000000	3000000	2700000	1000000000	NA	ND (330)	ND (330)	NA	NA	NA	NA
Acetonitrile	-	8000	14000000	22000000	11000000	10000000000	NA	NA	NA	NA	NA	NA	NA
Aniline	-	12000	4500000	-	-	29000000	NA	NA	NA	NA	NA	NA	NA
Anthracene	-	41000	1000000000	1000000000	1600000000	29000000000	NA	ND (330)	ND (330)	NA	NA	NA	NA
Benzidine	-	1000	1000	-	-	59,000	NA	NA	NA	NA	NA	NA	NA
Benzo(a)anthracene	-	-	210000	-	-	-	NA	ND (330)	ND (330)	NA	NA	NA	NA
Benzo(a)pyrene	-	-	21000	-	-	1900000	NA	ND (330)	ND (330)	NA	NA	NA	NA
Benzo(b)fluoranthene	-	-	210000	-	-	-	NA	ND (330)	ND (330)	NA	NA	NA	NA
Benzo(g,h,i)perylene	-	-	16000000	-	-	350000000	NA	ND (330)	ND (330)	NA	NA	NA	NA
Benzo(k)fluoranthene	-	-	2100000	-	-	-	NA	ND (330)	ND (330)	NA	NA	NA	NA
bis(2-Chloroethoxy)methane	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroethyl)ether	-	330	23000	44000	13000	12000000	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	-	-	10000000	-	-	8900000000	NA	NA	NA	NA	NA	NA	NA
Butyl benzylphthalate	-	310000	310000	-	-	21000000000	NA	NA	NA	NA	NA	NA	NA
Chrysene	-	-	21000000	-	-	-	NA	ND (330)	ND (330)	NA	NA	NA	NA
Dibenzo(a,h)anthracene	-		21000	-	-	-	NA	ND (330)	ND (330)	NA	NA	NA	NA
Dibenzofuran	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Diethyl phthalate	-	320000	740000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Dimethyl phthalate	-	790000	790000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Di-n-butylphthalate	-	760000	760000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Di-n-octyl phthalate	-	140000000	81000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Fluoranthene	-	720000	540000000	1000000000	880000000	4100000000	NA	ND (330)	ND (330)	NA	NA	NA	NA
Fluorene	-	890000	540000000	1000000000	150000000	4100000000	NA	ND (330)	ND (330)	NA	NA	NA	NA
Hexachlorobenzene	-	1800	94000	220000	56000	85000000	NA	NA	NA	NA	NA	NA	NA
Hexachlorobutadiene	-	77000	350000	350000	460000	180000000	NA	NA	NA	NA	NA	NA	NA
Hexachlorocyclopentadiene	-	36000	81000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Hexachloroethane	-	69000	1800000	370000	1400000	100000000	NA	NA	NA	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	-	-	210000	-	-	-	NA	ND (330)	ND (330)	NA	NA	NA	NA
Isophorone	-	74000	2400000	-	-	8200000000	NA	NA	NA	NA	NA	NA	NA
Naphthalene	-	50000	160000000	77000000	59000000	15000000000	NA	ND (330)	ND (330)	NA	NA	NA	NA
Nitrobenzene	-	330	340000	490000	4600000	1500000000	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	-	330	3500	-	-	2000000	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodiphenylamine	-	14000	5100000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Octachlorocyclopentene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Pentachlorophenol	-	3200	63000	-	-	130000000	NA	NA	NA	NA	NA	NA	NA
Phenanthrene	-	34000	16000000	28000000	150000	59000000	NA	ND (330)	ND (330)	NA	NA	NA	NA
Phenol	-	260000	12000000	-	-	18000000000	NA	NA	NA	NA	NA	NA	NA
Pyrene	-	470000	340000000	1000000000	770000000	2900000000	NA	ND (330)	ND (330)	NA	NA	NA	NA
Volatiles (ug/Kg)													
1,1,1-Trichloroethane	-	4000	460000	460000	4500000	29000000000	ND	ND (10)	ND (10)	ND (10)	NA	NA	NA
1,1,2,2-Tetrachloroethane	-	340	120000	23000	34000	68000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
1,1,2-Trichloroethane	-	100	440000	24000	57000	250000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
1,1-Dichloroethane	-	50000	790000	790000	36000000	240000000000	ND	ND (10)	ND (10)	ND (10)	NA	NA	NA
1,1-Dichloroethene	-	140	580000	330	3700	78000000	ND	ND (10)	ND (10)	ND (10)	NA	NA	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels (2)	Industrial Drinking Water Protection Criteria (2)	Industrial Direct Contact Criteria (2)	Soil Volatilization to Indoor Air Inhalation Criteria (2)	Volatile Soil Inhalation Criteria (2)	Particulate Soil Inhalation Criteria (2)	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							BLD 63-25 11/6/96 AOI # 8	PFB-15 (11-11.5) 3/24/97 AOI # 8	PFB-7 (8-10) 3/18/97 AOI # 8	PFW-3 (0.8-1.5) 3/17/97 AOI # 8	F-30 (0-2) 11/6/96 AOI # 9	F-34 11/14/96 AOI # 9	F-34 11/14/96 AOI # 9
1,2-Dibromoethane (EDB)	-	10	290	3600	5800	18000000	NA	NA	NA	NA	NA	NA	NA
1,2-Dichlorobenzene	-	13000	210000	210000	46000000	44000000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
1,2-Dichloroethane	-	100	270000	11000	21000	150000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
1,2-Dichloropropane	-	100	360000	7400	30000	120000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
1,3-Dichlorobenzene	-	18000	200000	-	-	-	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
1,4-Dichlorobenzene	-	1700	1000000	100000	260000	570000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
2-Butanone	-	760000	27000000	27000000	35000000	29000000000	NA	NA	NA	NA	NA	NA	NA
2-Chloroethyl vinyl ether	-	-	-	-	-	-	NA	ND (100)	ND (100)	ND (100)	NA	NA	NA
2-Hexanone	-	58000	2500000	1800000		1200000000	NA	NA	NA	NA	NA	NA	NA
4-Methyl-2-pentanone	-	100000	2700000	2700000	53000000	60000000000	NA	NA	NA	NA	NA	NA	NA
Acetone	-	42000	74000000	110000000	160000000	170000000000	NA	NA	NA	NA	NA	NA	NA
Benzene	-	100	400000	8400	45000	470000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
Bromodichloromethane	-	2000	400000	6400	31000	110000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
Bromoform	-	2000	870000	770000	3100000	3600000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
Bromomethane	-	580	1000000	1600	13000	150000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
Carbon disulfide	-	46000	280000	140000	1600000	21000000000	NA	NA	NA	NA	NA	NA	NA
Carbon tetrachloride	-	100	190000	990	12000	170000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
Chlorobenzene	-	2000	260000	220000	920000	2100000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
Chloroethane	-	18000	970000	970000	36000000	290000000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
Chloroform	-	2000	1500000	38000	150000	1600000000	ND	ND (10)	ND (10)	ND (10)	NA	NA	NA
Chloromethane	-	5400	1100000	12000	140000	6100000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
cis-1,2-Dichloroethene	-	1400	640000	640000	47000000	230000000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
cis-1,3-Dichloropropene	-	-	-	-	-	-	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
Dibromochloromethane	-	2000	300000	21000	80000	160000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
Dichlorodifluoromethane (CFC-12)	-	270000	1000000	1000000	63000000	1500000000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
Ethylbenzene	-	1500	140000	140000	11000000	29000000000	ND	ND (10)	ND (10)	ND (10)	NA	NA	NA
Ethylene oxide	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
m&p-Xylene	-	-	-	-	-	-	NA	ND (30)	ND (30)	ND (30)	NA	NA	NA
Methyl Tert Butyl Ether	-	800	6000000	6000000	31000000	88000000000	NA	NA	NA	NA	NA	NA	NA
Methylene chloride	-	100	2300000	240000	700000	8300000000	ND	ND (10)	ND (10)	ND (10)	NA	NA	NA
o-Xylene	-	-	-	-	-	-	NA	ND (30)	ND (30)	ND (30)	NA	NA	NA
Styrene	-	2700	520000	520000	3200000	6600000000	NA	NA	NA	NA	NA	NA	NA
Tetrachloroethene	-	100	88000	60000	600000	6800000000	ND	ND (10)	ND (10)	ND (10)	NA	NA	NA
Toluene	-	16000	250000	250000	3300000	12000000000	ND	ND (10)	ND (10)	ND (10)	NA	NA	NA
trans-1,2-Dichloroethene	-	2000	1400000	1400000	37000000	230000000000	ND	ND (10)	ND (10)	ND (10)	NA	NA	NA
trans-1,3-Dichloropropene	-	-	-	-	-	-	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
Trichloroethene	-	100	500000	37000	260000	2300000000	ND	ND (10)	ND (10)	ND (10)	NA	NA	NA
Trichlorofluoromethane (CFC-11)	-	150000	560000	560000	110000000	1700000000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
Vinyl chloride	-	40	11000	150	1500	47000000	NA	ND (10)	ND (10)	ND (10)	NA	NA	NA
Xylene (total)	-	5600	150000	150000	54000000	130000000000	ND	NA	NA	NA	NA	NA	NA

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI # F-34 11/14/96 AOI # 9	K-2 11/6/96 AOI # 9	K-32 11/6/96 AOI # 9	K-33 11/6/96 AOI # 9	K-35 11/6/96 AOI # 9	PFB-16 (3.3-3.9) 3/25/97 AOI # 9	PFB-17 (5.2-6.0) 3/25/97 AOI # 9
General Chemistry													
Cyanide (total) (ug/Kg)	-	4000	250000	-	-	250000	ND	ND	ND	NA	NA	NA	NA
Total Solids (%)	-	-	-	-	-	-	NA	NA	NA	NA	NA	90.11	95.06
Diesel Fuel (mg/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Petroleum hydrocarbons (ug/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Metals (ug/Kg)													
Arsenic	5800	23000	100000	-	-	910000	3360	5190	2950	3060	10700	6000	6000
Barium	75000	1300000	320000000	-	-	150000000	33100	57700	10400	10300	325000	44000	36000
Cadmium	1200	6000	4500000	-	-	2200000	ND	ND	ND	ND	6050	810	860
Chromium	18000	30000	22000000	-	-	-	11200	12500	42800	9720	1160000	15000	10000
Copper	32000	160000000	170000000	-	-	59000000	ND	ND	ND	ND	ND	17000	11000
Lead	21000	1000	900000	-	-	44000000	4990	7000	5030	ND	1280000	16000	17000
Mercury	130	1700	1400000	-	-	-	ND	ND	ND	ND	90	ND (100)	ND (100)
Selenium	410	4000	23000000	-	-	59000000	ND	ND	ND	ND	649	ND (500)	ND (500)
Silver	1000	13000	21000000	-	-	2900000	ND	ND	ND	ND	2970	600	900
Zinc	47000	5000000	1000000000	-	-	-	ND	ND	ND	ND	ND	41000	49000
PCBs (ug/Kg)													
Aroclor-1016	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1221	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1232	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1242	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1248	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1254	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1260	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Total PCBs	-	-	9900	16000000	820000	6500000	NA	NA	NA	NA	NA	ND (330)	ND (330)
Semi - Volatiles (ug/Kg)													
1,2,4-Trichlorobenzene	-	4200	1100000	1100000	34000000	11000000000	NA	NA	NA	NA	NA	NA	NA
2,4,6-Trichlorophenol	-	45000	14000000	-	-	1300000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dichlorophenol	-	7700	10000000	-	-	2300000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dimethylphenol	-	20000	230000000	-	-	2100000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrotoluene	-	15000	220000	-	-	20000000	NA	NA	NA	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Chloronaphthalene	-	1800000	190000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Chlorophenol	-	2600	4600000	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Nitrophenol	-	1200	13000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
3,3-Dichlorobenzidine	-	2000	55000	-	-	8200000	NA	NA	NA	NA	NA	NA	NA
3-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	-	1700	1600000	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chloro-3-methylphenol	-	16000	15000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chloroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI # F-34 11/14/96 AOI # 9	K-2 11/6/96 AOI # 9	K-32 11/6/96 AOI # 9	K-33 11/6/96 AOI # 9	K-35 11/6/96 AOI # 9	PFB-16 (3.3-3.9) 3/25/97 AOI # 9	PFB-17 (5.2-6.0) 3/25/97 AOI # 9
4-Nitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Acenaphthene	-	870000	810000000	350000000	97000000	6200000000	NA	NA	NA	NA	NA	ND (330)	ND (330)
Acenaphthylene	-	8500	16000000	3000000	2700000	1000000000	NA	NA	NA	NA	NA	ND (330)	ND (330)
Acetonitrile	-	8000	14000000	22000000	11000000	10000000000	NA	NA	NA	NA	NA	NA	NA
Aniline	-	12000	4500000	-	-	29000000	NA	NA	NA	NA	NA	NA	NA
Anthracene	-	41000	1000000000	1000000000	1600000000	29000000000	NA	NA	NA	NA	NA	ND (330)	ND (330)
Benzidine	-	1000	1000	-	-	59,000	NA	NA	NA	NA	NA	NA	NA
Benzo(a)anthracene	-	-	210000	-	-	-	NA	NA	NA	NA	NA	ND (330)	ND (330)
Benzo(a)pyrene	-	-	21000	-	-	1900000	NA	NA	NA	NA	NA	ND (330)	ND (330)
Benzo(b)fluoranthene	-	-	210000	-	-	-	NA	NA	NA	NA	NA	ND (330)	ND (330)
Benzo(g,h,i)perylene	-	-	16000000	-	-	350000000	NA	NA	NA	NA	NA	ND (330)	ND (330)
Benzo(k)fluoranthene	-	-	2100000	-	-	-	NA	NA	NA	NA	NA	ND (330)	ND (330)
bis(2-Chloroethoxy)methane	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroethyl)ether	-	330	23000	44000	13000	12000000	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	-	-	10000000	-	-	8900000000	NA	NA	NA	NA	NA	NA	NA
Butyl benzylphthalate	-	310000	310000	-	-	21000000000	NA	NA	NA	NA	NA	NA	NA
Chrysene	-	-	21000000	-	-	-	NA	NA	NA	NA	NA	ND (330)	ND (330)
Dibenzo(a,h)anthracene	-		21000	-	-	-	NA	NA	NA	NA	NA	ND (330)	ND (330)
Dibenzofuran	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Diethyl phthalate	-	320000	740000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Dimethyl phthalate	-	790000	790000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Di-n-butylphthalate	-	760000	760000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Di-n-octyl phthalate	-	140000000	81000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Fluoranthene	-	720000	540000000	1000000000	880000000	4100000000	NA	NA	NA	NA	NA	ND (330)	ND (330)
Fluorene	-	890000	540000000	1000000000	150000000	4100000000	NA	NA	NA	NA	NA	ND (330)	ND (330)
Hexachlorobenzene	-	1800	94000	220000	56000	85000000	NA	NA	NA	NA	NA	NA	NA
Hexachlorobutadiene	-	77000	350000	350000	460000	180000000	NA	NA	NA	NA	NA	NA	NA
Hexachlorocyclopentadiene	-	36000	81000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Hexachloroethane	-	69000	1800000	370000	1400000	100000000	NA	NA	NA	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	-	-	210000	-	-	-	NA	NA	NA	NA	NA	ND (330)	ND (330)
Isophorone	-	74000	2400000	-	-	8200000000	NA	NA	NA	NA	NA	NA	NA
Naphthalene	-	50000	160000000	77000000	59000000	15000000000	NA	NA	NA	NA	NA	ND (330)	ND (330)
Nitrobenzene	-	330	340000	490000	4600000	1500000000	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	-	330	3500	-	-	2000000	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodiphenylamine	-	14000	5100000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Octachlorocyclopentene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Pentachlorophenol	-	3200	63000	-	-	130000000	NA	NA	NA	NA	NA	NA	NA
Phenanthrene	-	34000	16000000	28000000	150000	59000000	NA	NA	NA	NA	NA	ND (330)	ND (330)
Phenol	-	260000	12000000	-	-	18000000000	NA	NA	NA	NA	NA	NA	NA
Pyrene	-	470000	340000000	1000000000	770000000	2900000000	NA	NA	NA	NA	NA	ND (330)	ND (330)
Volatiles (ug/Kg)													
1,1,1-Trichloroethane	-	4000	460000	460000	4500000	29000000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
1,1,2,2-Tetrachloroethane	-	340	120000	23000	34000	68000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
1,1,2-Trichloroethane	-	100	440000	24000	57000	250000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
1,1-Dichloroethane	-	50000	790000	790000	36000000	240000000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
1,1-Dichloroethene	-	140	580000	330	3700	78000000	NA	NA	NA	NA	NA	ND (10)	ND (10)

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							F-34 11/14/96 AOI # 9	K-2 11/6/96 AOI # 9	K-32 11/6/96 AOI # 9	K-33 11/6/96 AOI # 9	K-35 11/6/96 AOI # 9	PFB-16 (3.3-3.9) 3/25/97 AOI # 9	PFB-17 (5.2-6.0) 3/25/97 AOI # 9
1,2-Dibromoethane (EDB)	-	10	290	3600	5800	18000000	NA	NA	NA	NA	NA	NA	NA
1,2-Dichlorobenzene	-	13000	210000	210000	46000000	44000000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
1,2-Dichloroethane	-	100	270000	11000	21000	150000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
1,2-Dichloropropane	-	100	360000	7400	30000	120000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
1,3-Dichlorobenzene	-	18000	200000	-	-	-	NA	NA	NA	NA	NA	ND (10)	ND (10)
1,4-Dichlorobenzene	-	1700	1000000	100000	260000	570000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
2-Butanone	-	760000	27000000	27000000	35000000	29000000000	NA	NA	NA	NA	NA	NA	NA
2-Chloroethyl vinyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	ND (100)	ND (100)
2-Hexanone	-	58000	2500000	1800000		1200000000	NA	NA	NA	NA	NA	NA	NA
4-Methyl-2-pentanone	-	100000	2700000	2700000	53000000	60000000000	NA	NA	NA	NA	NA	NA	NA
Acetone	-	42000	74000000	110000000	160000000	170000000000	NA	NA	NA	NA	NA	NA	NA
Benzene	-	100	400000	8400	45000	470000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
Bromodichloromethane	-	2000	400000	6400	31000	110000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
Bromoform	-	2000	870000	770000	3100000	3600000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
Bromomethane	-	580	1000000	1600	13000	150000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
Carbon disulfide	-	46000	280000	140000	1600000	21000000000	NA	NA	NA	NA	NA	NA	NA
Carbon tetrachloride	-	100	190000	990	12000	170000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
Chlorobenzene	-	2000	260000	220000	920000	2100000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
Chloroethane	-	18000	970000	970000	36000000	290000000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
Chloroform	-	2000	1500000	38000	150000	1600000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
Chloromethane	-	5400	1100000	12000	140000	6100000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
cis-1,2-Dichloroethene	-	1400	640000	640000	47000000	230000000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
cis-1,3-Dichloropropene	-	-	-	-	-	-	NA	NA	NA	NA	NA	ND (10)	ND (10)
Dibromochloromethane	-	2000	300000	21000	80000	160000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
Dichlorodifluoromethane (CFC-12)	-	270000	1000000	1000000	63000000	1500000000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
Ethylbenzene	-	1500	140000	140000	11000000	29000000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
Ethylene oxide	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
m&p-Xylene	-	-	-	-	-	-	NA	NA	NA	NA	NA	ND (30)	ND (30)
Methyl Tert Butyl Ether	-	800	6000000	6000000	31000000	88000000000	NA	NA	NA	NA	NA	NA	NA
Methylene chloride	-	100	2300000	240000	700000	8300000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
o-Xylene	-	-	-	-	-	-	NA	NA	NA	NA	NA	ND (30)	ND (30)
Styrene	-	2700	520000	520000	3200000	6600000000	NA	NA	NA	NA	NA	NA	NA
Tetrachloroethene	-	100	88000	60000	600000	6800000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
Toluene	-	16000	250000	250000	3300000	12000000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
trans-1,2-Dichloroethene	-	2000	1400000	1400000	37000000	230000000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
trans-1,3-Dichloropropene	-	-	-	-	-	-	NA	NA	NA	NA	NA	ND (10)	ND (10)
Trichloroethene	-	100	500000	37000	260000	2300000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
Trichlorofluoromethane (CFC-11)	-	150000	560000	560000	110000000	1700000000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
Vinyl chloride	-	40	11000	150	1500	47000000	NA	NA	NA	NA	NA	ND (10)	ND (10)
Xylene (total)	-	5600	150000	150000	54000000	130000000000	NA	NA	NA	NA	NA	NA	NA

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #					
							PFB-18 (1.4-2.0) 3/26/97 AOI # 9	PFB-19 (1.0-1.6) 3/26/97 AOI # 9	PFB-20 (1.6-2.3) 3/26/97 AOI # 9	PFW-12 (0.8-1.3) 3/21/97 AOI # 9	PFW-12 (6.2-6.7) 3/21/97 AOI # 9	PFB-23 (5.8-7) 4/24/97 AOI # 11
General Chemistry												
Cyanide (total) (ug/Kg)	-	4000	250000	-	-	250000	NA	NA	NA	NA	NA	NA
Total Solids (%)	-	-	-	-	-	-	90.1	88.77	90.21	86.83	88.64	87.13
Diesel Fuel (mg/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
Petroleum hydrocarbons (ug/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
Metals (ug/Kg)												
Arsenic	5800	23000	100000	-	-	910000	8000	7000	9000	9000	12000	12000
Barium	75000	1300000	320000000	-	-	150000000	54000	74000	88000	81000	100000	85000
Cadmium	1200	6000	4500000	-	-	2200000	830	860	900	670	820	1100
Chromium	18000	30000	22000000	-	-	-	18000	20000	18000	25000	20000	23000
Copper	32000	160000000	170000000	-	-	59000000	14000	15000	17000	16000	16000	16000
Lead	21000	1000	900000	-	-	44000000	17000	19000	17000	18000	19000	16000
Mercury	130	1700	1400000	-	-	-	ND (100)	ND (100)	ND (100)	ND (100)	ND (100)	ND (100)
Selenium	410	4000	23000000	-	-	59000000	ND (500)	ND (500)	ND (500)	ND (500)	ND (500)	ND (500)
Silver	1000	13000	21000000	-	-	2900000	800	900	800	800	1000	ND (500)
Zinc	47000	5000000	1000000000	-	-	-	42000	43000	56000	45000	48000	58000
PCBs (ug/Kg)												
Aroclor-1016	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1221	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1232	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1242	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1248	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1254	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1260	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Total PCBs	-	-	9900	16000000	820000	6500000	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)	NA
Semi - Volatiles (ug/Kg)												
1,2,4-Trichlorobenzene	-	4200	1100000	1100000	34000000	11000000000	NA	NA	NA	NA	NA	NA
2,4,6-Trichlorophenol	-	45000	14000000	-	-	1300000000	NA	NA	NA	NA	NA	NA
2,4-Dichlorophenol	-	7700	10000000	-	-	2300000000	NA	NA	NA	NA	NA	NA
2,4-Dimethylphenol	-	20000	230000000	-	-	2100000000	NA	NA	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
2,4-Dinitrotoluene	-	15000	220000	-	-	20000000	NA	NA	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
2-Chloronaphthalene	-	1800000	190000000	-	-	-	NA	NA	NA	NA	NA	NA
2-Chlorophenol	-	2600	4600000	-	-	-	NA	NA	NA	NA	NA	NA
2-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
2-Nitrophenol	-	1200	13000000	-	-	-	NA	NA	NA	NA	NA	NA
3,3-Dichlorobenzidine	-	2000	55000	-	-	8200000	NA	NA	NA	NA	NA	NA
3-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	-	1700	1600000	-	-	-	NA	NA	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
4-Chloro-3-methylphenol	-	16000	15000000	-	-	-	NA	NA	NA	NA	NA	NA
4-Chloroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
4-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #					
							PFB-18 (1.4-2.0) 3/26/97 AOI # 9	PFB-19 (1.0-1.6) 3/26/97 AOI # 9	PFB-20 (1.6-2.3) 3/26/97 AOI # 9	PFW-12 (0.8-1.3) 3/21/97 AOI # 9	PFW-12 (6.2-6.7) 3/21/97 AOI # 9	PFB-23 (5.8-7) 4/24/97 AOI # 11
4-Nitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
Acenaphthene	-	870000	810000000	350000000	97000000	6200000000	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)
Acenaphthylene	-	8500	16000000	3000000	2700000	1000000000	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)
Acetonitrile	-	8000	14000000	22000000	11000000	10000000000	NA	NA	NA	NA	NA	NA
Aniline	-	12000	4500000	-	-	29000000	NA	NA	NA	NA	NA	NA
Anthracene	-	41000	1000000000	1000000000	1600000000	29000000000	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)
Benzidine	-	1000	1000	-	-	59,000	NA	NA	NA	NA	NA	NA
Benzo(a)anthracene	-	-	210000	-	-	-	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)
Benzo(a)pyrene	-	-	21000	-	-	1900000	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)
Benzo(b)fluoranthene	-	-	210000	-	-	-	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)
Benzo(g,h,i)perylene	-	-	16000000	-	-	350000000	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)
Benzo(k)fluoranthene	-	-	2100000	-	-	-	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)
bis(2-Chloroethoxy)methane	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
bis(2-Chloroethyl)ether	-	330	23000	44000	13000	12000000	NA	NA	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	-	-	10000000	-	-	8900000000	NA	NA	NA	NA	NA	NA
Butyl benzylphthalate	-	310000	310000	-	-	21000000000	NA	NA	NA	NA	NA	NA
Chrysene	-	-	21000000	-	-	-	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)
Dibenzo(a,h)anthracene	-		21000	-	-	-	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)
Dibenzofuran	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
Diethyl phthalate	-	320000	740000	-	-	1500000000	NA	NA	NA	NA	NA	NA
Dimethyl phthalate	-	790000	790000	-	-	1500000000	NA	NA	NA	NA	NA	NA
Di-n-butylphthalate	-	760000	760000	-	-	1500000000	NA	NA	NA	NA	NA	NA
Di-n-octyl phthalate	-	140000000	81000000	-	-	-	NA	NA	NA	NA	NA	NA
Fluoranthene	-	720000	540000000	1000000000	880000000	4100000000	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)
Fluorene	-	890000	540000000	1000000000	150000000	4100000000	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)
Hexachlorobenzene	-	1800	94000	220000	56000	85000000	NA	NA	NA	NA	NA	NA
Hexachlorobutadiene	-	77000	350000	350000	460000	180000000	NA	NA	NA	NA	NA	NA
Hexachlorocyclopentadiene	-	36000	81000	-	-	-	NA	NA	NA	NA	NA	NA
Hexachloroethane	-	69000	1800000	370000	1400000	100000000	NA	NA	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	-	-	210000	-	-	-	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)
Isophorone	-	74000	2400000	-	-	8200000000	NA	NA	NA	NA	NA	NA
Naphthalene	-	50000	160000000	77000000	59000000	15000000000	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)
Nitrobenzene	-	330	340000	490000	4600000	1500000000	NA	NA	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	-	330	3500	-	-	2000000	NA	NA	NA	NA	NA	NA
N-Nitrosodiphenylamine	-	14000	5100000	-	-	-	NA	NA	NA	NA	NA	NA
Octachlorocyclopentene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
Pentachlorophenol	-	3200	63000	-	-	130000000	NA	NA	NA	NA	NA	NA
Phenanthrene	-	34000	16000000	28000000	150000	59000000	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)
Phenol	-	260000	12000000	-	-	18000000000	NA	NA	NA	NA	NA	NA
Pyrene	-	470000	340000000	1000000000	770000000	2900000000	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)	ND (330)
Volatiles (ug/Kg)												
1,1,1-Trichloroethane	-	4000	460000	460000	4500000	29000000000	ND (10)	ND (10)	ND (10)	420	ND (10)	NA
1,1,2,2-Tetrachloroethane	-	340	120000	23000	34000	68000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
1,1,2-Trichloroethane	-	100	440000	24000	57000	250000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
1,1-Dichloroethane	-	50000	790000	790000	36000000	240000000000	ND (10)	ND (10)	ND (10)	97	ND (10)	NA
1,1-Dichloroethene	-	140	580000	330	3700	78000000	ND (10)	ND (10)	ND (10)	11	ND (10)	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #					
							PFB-18 (1.4-2.0) 3/26/97 AOI # 9	PFB-19 (1.0-1.6) 3/26/97 AOI # 9	PFB-20 (1.6-2.3) 3/26/97 AOI # 9	PFW-12 (0.8-1.3) 3/21/97 AOI # 9	PFW-12 (6.2-6.7) 3/21/97 AOI # 9	PFB-23 (5.8-7) 4/24/97 AOI # 11
1,2-Dibromoethane (EDB)	-	10	290	3600	5800	18000000	NA	NA	NA	NA	NA	NA
1,2-Dichlorobenzene	-	13000	210000	210000	46000000	44000000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
1,2-Dichloroethane	-	100	270000	11000	21000	150000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
1,2-Dichloropropane	-	100	360000	7400	30000	120000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
1,3-Dichlorobenzene	-	18000	200000	-	-	-	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
1,4-Dichlorobenzene	-	1700	1000000	100000	260000	570000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
2-Butanone	-	760000	27000000	27000000	35000000	29000000000	NA	NA	NA	NA	NA	NA
2-Chloroethyl vinyl ether	-	-	-	-	-	-	ND (100)	ND (100)	ND (100)	ND (100)	ND (100)	NA
2-Hexanone	-	58000	2500000	1800000		1200000000	NA	NA	NA	NA	NA	NA
4-Methyl-2-pentanone	-	100000	2700000	2700000	53000000	60000000000	NA	NA	NA	NA	NA	NA
Acetone	-	42000	74000000	110000000	160000000	170000000000	NA	NA	NA	NA	NA	NA
Benzene	-	100	400000	8400	45000	470000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
Bromodichloromethane	-	2000	400000	6400	31000	110000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
Bromoform	-	2000	870000	770000	3100000	3600000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
Bromomethane	-	580	1000000	1600	13000	150000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
Carbon disulfide	-	46000	280000	140000	1600000	21000000000	NA	NA	NA	NA	NA	NA
Carbon tetrachloride	-	100	190000	990	12000	170000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
Chlorobenzene	-	2000	260000	220000	920000	2100000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
Chloroethane	-	18000	970000	970000	36000000	290000000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
Chloroform	-	2000	1500000	38000	150000	1600000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
Chloromethane	-	5400	1100000	12000	140000	6100000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
cis-1,2-Dichloroethene	-	1400	640000	640000	47000000	230000000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
cis-1,3-Dichloropropene	-	-	-	-	-	-	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
Dibromochloromethane	-	2000	300000	21000	80000	160000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
Dichlorodifluoromethane (CFC-12)	-	270000	1000000	1000000	63000000	1500000000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
Ethylbenzene	-	1500	140000	140000	11000000	29000000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
Ethylene oxide	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
m&p-Xylene	-	-	-	-	-	-	ND (30)	ND (30)	ND (30)	ND (30)	ND (30)	NA
Methyl Tert Butyl Ether	-	800	6000000	6000000	31000000	88000000000	NA	NA	NA	NA	NA	NA
Methylene chloride	-	100	2300000	240000	700000	8300000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
o-Xylene	-	-	-	-	-	-	ND (30)	ND (30)	ND (30)	ND (30)	ND (30)	NA
Styrene	-	2700	520000	520000	3200000	6600000000	NA	NA	NA	NA	NA	NA
Tetrachloroethene	-	100	88000	60000	600000	6800000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
Toluene	-	16000	250000	250000	3300000	12000000000	ND (10)	ND (10)	ND (10)	17	ND (10)	NA
trans-1,2-Dichloroethene	-	2000	1400000	1400000	37000000	230000000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
trans-1,3-Dichloropropene	-	-	-	-	-	-	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
Trichloroethene	-	100	500000	37000	260000	2300000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
Trichlorofluoromethane (CFC-11)	-	150000	560000	560000	110000000	1700000000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
Vinyl chloride	-	40	11000	150	1500	47000000	ND (10)	ND (10)	ND (10)	ND (10)	ND (10)	NA
Xylene (total)	-	5600	150000	150000	54000000	130000000000	NA	NA	NA	NA	NA	NA

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #					
							PFB-24 (1.2-2.5) 4/24/97 AOI # 11	GP-6 (0-4) 8/30/86 AOI # 13	GP-6 (0-4) 8/30/86 AOI # 13	MW-1 (2-4) 11/6/96 AOI # 14	PFW-2 (0-1.5) 3/14/97 AOI # 14	PFW-5 (0-1.7) 3/18/97 AOI # 15
General Chemistry												
Cyanide (total) (ug/Kg)	-	4000	250000	-	-	250000	NA	NA	NA	NA	NA	NA
Total Solids (%)	-	-	-	-	-	-	89.88	NA	NA	NA	89.88	88.91
Diesel Fuel (mg/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
Petroleum hydrocarbons (ug/Kg)	-	-	-	-	-	-	NA	NA	NA	336000	NA	NA
Metals (ug/Kg)												
Arsenic	5800	23000	100000	-	-	910000	11000	NA	NA	NA	18000	5000
Barium	75000	1300000	320000000	-	-	150000000	64000	NA	NA	NA	54000	22000
Cadmium	1200	6000	4500000	-	-	2200000	1900	NA	NA	NA	570	490
Chromium	18000	30000	22000000	-	-	-	19000	NA	NA	NA	15000	9100
Copper	32000	160000000	170000000	-	-	59000000	14000	NA	NA	NA	13000	8100
Lead	21000	1000	900000	-	-	44000000	19000	NA	NA	NA	14000	11000
Mercury	130	1700	1400000	-	-	-	ND (100)	NA	NA	NA	ND (100)	ND (100)
Selenium	410	4000	23000000	-	-	59000000	ND (500)	NA	NA	NA	ND (500)	ND (500)
Silver	1000	13000	21000000	-	-	2900000	1200	NA	NA	NA	ND (500)	ND (500)
Zinc	47000	5000000	1000000000	-	-	-	45000	NA	NA	NA	42000	30000
PCBs (ug/Kg)												
Aroclor-1016	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1221	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1232	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1242	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1248	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1254	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1260	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Total PCBs	-	-	9900	16000000	820000	6500000	NA	NA	NA	NA	ND (330)	NA
Semi - Volatiles (ug/Kg)												
1,2,4-Trichlorobenzene	-	4200	1100000	1100000	34000000	11000000000	NA	ND (330)	NA	NA	NA	NA
2,4,6-Trichlorophenol	-	45000	14000000	-	-	1300000000	NA	ND (330)	NA	NA	NA	NA
2,4-Dichlorophenol	-	7700	10000000	-	-	2300000000	NA	ND (330)	NA	NA	NA	NA
2,4-Dimethylphenol	-	20000	230000000	-	-	2100000000	NA	ND (330)	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	-	-	-	NA	ND (1700)	NA	NA	NA	NA
2,4-Dinitrotoluene	-	15000	220000	-	-	20000000	NA	ND (330)	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	-	-	-	NA	ND (330)	NA	NA	NA	NA
2-Chloronaphthalene	-	1800000	190000000	-	-	-	NA	ND (330)	NA	NA	NA	NA
2-Chlorophenol	-	2600	4600000	-	-	-	NA	ND (330)	NA	NA	NA	NA
2-Nitroaniline	-	-	-	-	-	-	NA	ND (1700)	NA	NA	NA	NA
2-Nitrophenol	-	1200	13000000	-	-	-	NA	ND (330)	NA	NA	NA	NA
3,3-Dichlorobenzidine	-	2000	55000	-	-	8200000	NA	ND (2000)	NA	NA	NA	NA
3-Nitroaniline	-	-	-	-	-	-	NA	ND (1700)	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	-	1700	1600000	-	-	-	NA	ND (1700)	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	-	-	-	NA	ND (330)	NA	NA	NA	NA
4-Chloro-3-methylphenol	-	16000	15000000	-	-	-	NA	ND (330)	NA	NA	NA	NA
4-Chloroaniline	-	-	-	-	-	-	NA	ND (1300)	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	-	-	-	NA	ND (330)	NA	NA	NA	NA
4-Nitroaniline	-	-	-	-	-	-	NA	ND (1700)	NA	NA	NA	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #					
							PFB-24 (1.2-2.5) 4/24/97 AOI # 11	GP-6 (0-4) 8/30/86 AOI # 13	GP-6 (0-4) 8/30/86 AOI # 13	MW-1 (2-4) 11/6/96 AOI # 14	PFW-2 (0-1.5) 3/14/97 AOI # 14	PFW-5 (0-1.7) 3/18/97 AOI # 15
4-Nitrophenol	-	-	-	-	-	-	NA	ND (1700)	NA	NA	NA	NA
Acenaphthene	-	870000	810000000	350000000	97000000	6200000000	ND (330)	ND (330)	NA	NA	ND (330)	NA
Acenaphthylene	-	8500	16000000	3000000	2700000	1000000000	ND (330)	ND (330)	NA	NA	ND (330)	NA
Acetonitrile	-	8000	14000000	22000000	11000000	10000000000	NA	ND (100)	NA	NA	NA	NA
Aniline	-	12000	4500000	-	-	29000000	NA	ND (1700)	NA	NA	NA	NA
Anthracene	-	41000	1000000000	1000000000	1600000000	29000000000	ND (330)	ND (330)	NA	NA	ND (330)	NA
Benzidine	-	1000	1000	-	-	59,000	NA	ND (5000)	NA	NA	NA	NA
Benzo(a)anthracene	-	-	210000	-	-	-	2200	ND (330)	NA	NA	ND (330)	NA
Benzo(a)pyrene	-	-	21000	-	-	1900000	ND (330)	ND (330)	NA	NA	ND (330)	NA
Benzo(b)fluoranthene	-	-	210000	-	-	-	ND (330)	ND (330)	NA	NA	ND (330)	NA
Benzo(g,h,i)perylene	-	-	16000000	-	-	350000000	ND (330)	ND (330)	NA	NA	ND (330)	NA
Benzo(k)fluoranthene	-	-	2100000	-	-	-	ND (330)	ND (330)	NA	NA	ND (330)	NA
bis(2-Chloroethoxy)methane	-	-	-	-	-	-	NA	ND (330)	NA	NA	NA	NA
bis(2-Chloroethyl)ether	-	330	23000	44000	13000	12000000	NA	ND (330)	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	-	-	-	NA	ND (330)	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	-	-	10000000	-	-	8900000000	NA	ND (330)	NA	NA	NA	NA
Butyl benzylphthalate	-	310000	310000	-	-	21000000000	NA	ND (330)	NA	NA	NA	NA
Chrysene	-	-	21000000	-	-	-	4800	ND (330)	NA	NA	ND (330)	NA
Dibenzo(a,h)anthracene	-		21000	-	-	-	ND (330)	ND (330)	NA	NA	ND (330)	NA
Dibenzofuran	-	-	-	-	-	-	NA	ND (330)	NA	NA	NA	NA
Diethyl phthalate	-	320000	740000	-	-	1500000000	NA	ND (330)	NA	NA	NA	NA
Dimethyl phthalate	-	790000	790000	-	-	1500000000	NA	ND (330)	NA	NA	NA	NA
Di-n-butylphthalate	-	760000	760000	-	-	1500000000	NA	ND (330)	NA	NA	NA	NA
Di-n-octyl phthalate	-	140000000	81000000	-	-	-	NA	ND (330)	NA	NA	NA	NA
Fluoranthene	-	720000	540000000	1000000000	880000000	4100000000	9600	ND (330)	NA	NA	ND (330)	NA
Fluorene	-	890000	540000000	1000000000	150000000	4100000000	2600	ND (330)	NA	NA	ND (330)	NA
Hexachlorobenzene	-	1800	94000	220000	56000	85000000	NA	ND (330)	NA	NA	NA	NA
Hexachlorobutadiene	-	77000	350000	350000	460000	180000000	NA	ND (330)	NA	NA	NA	NA
Hexachlorocyclopentadiene	-	36000	81000	-	-	-	NA	ND (330)	NA	NA	NA	NA
Hexachloroethane	-	69000	1800000	370000	1400000	100000000	NA	ND (330)	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	-	-	210000	-	-	-	ND (330)	ND (330)	NA	NA	ND (330)	NA
Isophorone	-	74000	2400000	-	-	8200000000	NA	ND (330)	NA	NA	NA	NA
Naphthalene	-	50000	160000000	77000000	59000000	15000000000	1900	ND (330)	NA	NA	ND (330)	NA
Nitrobenzene	-	330	340000	490000	4600000	1500000000	NA	ND (330)	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	-	330	3500	-	-	2000000	NA	ND (330)	NA	NA	NA	NA
N-Nitrosodiphenylamine	-	14000	5100000	-	-	-	NA	ND (330)	NA	NA	NA	NA
Octachlorocyclopentene	-	-	-	-	-	-	NA	ND (330)	NA	NA	NA	NA
Pentachlorophenol	-	3200	63000	-	-	130000000	NA	ND (3400)	NA	NA	NA	NA
Phenanthrene	-	34000	16000000	28000000	150000	59000000	ND (330)	ND (330)	NA	NA	ND (330)	NA
Phenol	-	260000	12000000	-	-	18000000000	NA	ND (330)	NA	NA	NA	NA
Pyrene	-	470000	340000000	1000000000	770000000	2900000000	27000	ND (330)	NA	NA	ND (330)	NA
Volatiles (ug/Kg)												
1,1,1-Trichloroethane	-	4000	460000	460000	4500000	29000000000	NA	ND (10)	NA	ND	ND (10)	ND (10)
1,1,2,2-Tetrachloroethane	-	340	120000	23000	34000	68000000	NA	ND (10)	NA	NA	ND (10)	ND (10)
1,1,2-Trichloroethane	-	100	440000	24000	57000	250000000	NA	ND (10)	NA	NA	ND (10)	ND (10)
1,1-Dichloroethane	-	50000	790000	790000	36000000	240000000000	NA	ND (10)	NA	ND	ND (10)	ND (10)
1,1-Dichloroethene	-	140	580000	330	3700	78000000	NA	ND (10)	NA	ND	ND (10)	ND (10)

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels (2)	Industrial Drinking Water Protection Criteria (2)	Industrial Direct Contact Criteria (2)	Soil Volatilization to Indoor Air Inhalation Criteria (2)	Volatile Soil Inhalation Criteria (2)	Particulate Soil Inhalation Criteria (2)	Sample Location (depth ft. bgs) / Sample Date / AOI #					
							PFB-24 (1.2-2.5) 4/24/97 AOI # 11	GP-6 (0-4) 8/30/86 AOI # 13	GP-6 (0-4) 8/30/86 AOI # 13	MW-1 (2-4) 11/6/96 AOI # 14	PFW-2 (0-1.5) 3/14/97 AOI # 14	PFW-5 (0-1.7) 3/18/97 AOI # 15
1,2-Dibromoethane (EDB)	-	10	290	3600	5800	18000000	NA	ND (10)	NA	NA	NA	NA
1,2-Dichlorobenzene	-	13000	210000	210000	46000000	44000000000	NA	ND (10)	NA	NA	ND (10)	ND (10)
1,2-Dichloroethane	-	100	270000	11000	21000	150000000	NA	ND (10)	NA	NA	ND (10)	ND (10)
1,2-Dichloropropane	-	100	360000	7400	30000	120000000	NA	ND (10)	NA	NA	ND (10)	ND (10)
1,3-Dichlorobenzene	-	18000	200000	-	-	-	NA	ND (10)	NA	NA	ND (10)	ND (10)
1,4-Dichlorobenzene	-	1700	1000000	100000	260000	570000000	NA	ND (10)	NA	NA	ND (10)	ND (10)
2-Butanone	-	760000	27000000	27000000	35000000	29000000000	NA	ND (100)	NA	NA	NA	NA
2-Chloroethyl vinyl ether	-	-	-	-	-	-	NA	NA	NA	NA	ND (100)	ND (100)
2-Hexanone	-	58000	2500000	1800000		1200000000	NA	ND (100)	NA	NA	NA	NA
4-Methyl-2-pentanone	-	100000	2700000	2700000	53000000	60000000000	NA	ND (100)	NA	NA	NA	NA
Acetone	-	42000	74000000	110000000	160000000	170000000000	NA	ND (100)	NA	NA	NA	NA
Benzene	-	100	400000	8400	45000	470000000	NA	ND (10)	NA	NA	ND (10)	ND (10)
Bromodichloromethane	-	2000	400000	6400	31000	110000000	NA	ND (10)	NA	NA	ND (10)	ND (10)
Bromoform	-	2000	870000	770000	3100000	3600000000	NA	ND (10)	NA	NA	ND (10)	ND (10)
Bromomethane	-	580	1000000	1600	13000	150000000	NA	ND (10)	NA	NA	ND (10)	ND (10)
Carbon disulfide	-	46000	280000	140000	1600000	21000000000	NA	ND (100)	NA	NA	NA	NA
Carbon tetrachloride	-	100	190000	990	12000	170000000	NA	ND (10)	NA	NA	ND (10)	ND (10)
Chlorobenzene	-	2000	260000	220000	920000	2100000000	NA	ND (10)	NA	NA	ND (10)	ND (10)
Chloroethane	-	18000	970000	970000	36000000	290000000000	NA	ND (10)	NA	NA	ND (10)	ND (10)
Chloroform	-	2000	1500000	38000	150000	1600000000	NA	ND (10)	NA	ND	ND (10)	ND (10)
Chloromethane	-	5400	1100000	12000	140000	6100000000	NA	ND (10)	NA	NA	ND (10)	ND (10)
cis-1,2-Dichloroethene	-	1400	640000	640000	47000000	230000000000	NA	ND (10)	NA	NA	ND (10)	ND (10)
cis-1,3-Dichloropropene	-	-	-	-	-	-	NA	ND (10)	NA	NA	ND (10)	ND (10)
Dibromochloromethane	-	2000	300000	21000	80000	160000000	NA	ND (10)	NA	NA	ND (10)	ND (10)
Dichlorodifluoromethane (CFC-12)	-	270000	1000000	1000000	63000000	1500000000000	NA	NA	NA	NA	ND (10)	ND (10)
Ethylbenzene	-	1500	140000	140000	11000000	29000000000	NA	ND (10)	NA	1.3	ND (10)	ND (10)
Ethylene oxide	-	-	-	-	-	-	NA	ND (1000)	NA	NA	NA	NA
m&p-Xylene	-	-	-	-	-	-	NA	NA	NA	NA	ND (30)	ND (30)
Methyl Tert Butyl Ether	-	800	6000000	6000000	31000000	880000000000	NA	ND (100)	NA	NA	NA	NA
Methylene chloride	-	100	2300000	240000	700000	8300000000	NA	37	NA	14.3	ND (10)	ND (10)
o-Xylene	-	-	-	-	-	-	NA	NA	NA	NA	ND (30)	ND (30)
Styrene	-	2700	520000	520000	3200000	6600000000	NA	ND (10)	NA	NA	NA	NA
Tetrachloroethene	-	100	88000	60000	600000	6800000000	NA	ND (10)	NA	ND	ND (10)	ND (10)
Toluene	-	16000	250000	250000	3300000	12000000000	NA	ND (10)	NA	4.6	ND (10)	ND (10)
trans-1,2-Dichloroethene	-	2000	1400000	1400000	37000000	230000000000	NA	NA	ND (10)	ND	ND (10)	ND (10)
trans-1,3-Dichloropropene	-	-	-	-	-	-	NA	ND (10)	NA	NA	ND (10)	ND (10)
Trichloroethene	-	100	500000	37000	260000	2300000000	NA	ND (10)	NA	ND	ND (10)	ND (10)
Trichlorofluoromethane (CFC-11)	-	150000	560000	560000	110000000	1700000000000	NA	NA	NA	NA	ND (10)	ND (10)
Vinyl chloride	-	40	11000	150	1500	47000000	NA	ND (10)	NA	NA	ND (10)	ND (10)
Xylene (total)	-	5600	150000	150000	54000000	130000000000	NA	ND (30)	NA	8.1	NA	NA

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							SB-9 (3-5) 11/6/96 AOI # 15	MW-2 (8-10) 11/6/96 AOI # 16	SB-2 (0-2) 11/7/96 AOI # 17	SB-2 (0-2) 11/7/96 AOI # 17	SB-2 (0-2) 11/7/96 AOI # 17	GP-3 (1-4) 8/30/96 AOI # 18	GP-3 (1-4) 8/30/96 AOI # 18
General Chemistry													
Cyanide (total) (ug/Kg)	-	4000	250000	-	-	250000	NA	NA	NA	NA	NA	NA	NA
Total Solids (%)	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Diesel Fuel (mg/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	NA	ND (6.6)	NA
Petroleum hydrocarbons (ug/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Metals (ug/Kg)													
Arsenic	5800	23000	100000	-	-	910000	NA	NA	NA	NA	NA	8500	NA
Barium	75000	1300000	320000000	-	-	150000000	NA	NA	NA	NA	NA	10000	NA
Cadmium	1200	6000	4500000	-	-	2200000	NA	NA	NA	NA	NA	82	NA
Chromium	18000	30000	22000000	-	-	-	NA	NA	NA	NA	NA	4500	NA
Copper	32000	160000000	170000000	-	-	59000000	NA	NA	NA	NA	NA	7600	NA
Lead	21000	1000	900000	-	-	44000000	NA	NA	NA	NA	NA	4100	NA
Mercury	130	1700	1400000	-	-	-	NA	NA	NA	NA	NA	ND (100)	NA
Selenium	410	4000	23000000	-	-	59000000	NA	NA	NA	NA	NA	940	NA
Silver	1000	13000	21000000	-	-	2900000	NA	NA	NA	NA	NA	ND (500)	NA
Zinc	47000	5000000	1000000000	-	-	-	NA	NA	NA	NA	NA	25000	NA
PCBs (ug/Kg)													
Aroclor-1016	-	-	9900	-	-	-	NA	NA	ND (330)	NA	NA	NA	NA
Aroclor-1221	-	-	9900	-	-	-	NA	NA	ND (330)	NA	NA	NA	NA
Aroclor-1232	-	-	9900	-	-	-	NA	NA	ND (330)	NA	NA	NA	NA
Aroclor-1242	-	-	9900	-	-	-	NA	NA	ND (330)	NA	NA	NA	NA
Aroclor-1248	-	-	9900	-	-	-	NA	NA	ND (330)	NA	NA	NA	NA
Aroclor-1254	-	-	9900	-	-	-	NA	NA	NA	ND (330)	ND	NA	NA
Aroclor-1260	-	-	9900	-	-	-	NA	NA	NA	ND (330)	150	NA	NA
Total PCBs	-	-	9900	16000000	820000	6500000	NA	NA	NA	ND (330)	ND	NA	NA
Semi - Volatiles (ug/Kg)													
1,2,4-Trichlorobenzene	-	4200	1100000	1100000	34000000	11000000000	NA	NA	NA	NA	NA	NA	NA
2,4,6-Trichlorophenol	-	45000	14000000	-	-	1300000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dichlorophenol	-	7700	10000000	-	-	2300000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dimethylphenol	-	20000	230000000	-	-	2100000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrotoluene	-	15000	220000	-	-	20000000	NA	NA	NA	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Chloronaphthalene	-	1800000	190000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Chlorophenol	-	2600	4600000	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Nitrophenol	-	1200	13000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
3,3-Dichlorobenzidine	-	2000	55000	-	-	8200000	NA	NA	NA	NA	NA	NA	NA
3-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	-	1700	1600000	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chloro-3-methylphenol	-	16000	15000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chloroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) Sample Date AOI # SB-9 (3-5) 11/6/96 AOI # 15	MW-2 (8-10) 11/6/96 AOI # 16	SB-2 (0-2) 11/7/96 AOI # 17	SB-2 (0-2) 11/7/96 AOI # 17	SB-2 (0-2) 11/7/96 AOI # 17	GP-3 (1-4) 8/30/96 AOI # 18	GP-3 (1-4) 8/30/96 AOI # 18
4-Nitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Acenaphthene	-	870000	810000000	350000000	97000000	6200000000	NA	NA	NA	NA	NA	NA	NA
Acenaphthylene	-	8500	16000000	3000000	2700000	1000000000	NA	NA	NA	NA	NA	NA	NA
Acetonitrile	-	8000	14000000	22000000	11000000	10000000000	NA	NA	NA	NA	NA	ND (100)	NA
Aniline	-	12000	4500000	-	-	29000000	NA	NA	NA	NA	NA	NA	NA
Anthracene	-	41000	1000000000	1000000000	1600000000	29000000000	NA	NA	NA	NA	NA	NA	NA
Benzidine	-	1000	1000	-	-	59,000	NA	NA	NA	NA	NA	NA	NA
Benzo(a)anthracene	-	-	210000	-	-	-	11	NA	NA	NA	NA	NA	NA
Benzo(a)pyrene	-	-	21000	-	-	1900000	ND	NA	NA	NA	NA	NA	NA
Benzo(b)fluoranthene	-	-	210000	-	-	-	14	NA	NA	NA	NA	NA	NA
Benzo(g,h,i)perylene	-	-	16000000	-	-	350000000	NA	NA	NA	NA	NA	NA	NA
Benzo(k)fluoranthene	-	-	2100000	-	-	-	ND	NA	NA	NA	NA	NA	NA
bis(2-Chloroethoxy)methane	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroethyl)ether	-	330	23000	44000	13000	12000000	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	-	-	10000000	-	-	8900000000	NA	NA	NA	NA	NA	NA	NA
Butyl benzylphthalate	-	310000	310000	-	-	21000000000	NA	NA	NA	NA	NA	NA	NA
Chrysene	-	-	21000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Dibenzo(a,h)anthracene	-		21000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Dibenzofuran	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Diethyl phthalate	-	320000	740000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Dimethyl phthalate	-	790000	790000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Di-n-butylphthalate	-	760000	760000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Di-n-octyl phthalate	-	140000000	81000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Fluoranthene	-	720000	540000000	1000000000	880000000	4100000000	NA	NA	NA	NA	NA	NA	NA
Fluorene	-	890000	540000000	1000000000	150000000	4100000000	NA	NA	NA	NA	NA	NA	NA
Hexachlorobenzene	-	1800	94000	220000	56000	85000000	NA	NA	NA	NA	NA	NA	NA
Hexachlorobutadiene	-	77000	350000	350000	460000	180000000	NA	NA	NA	NA	NA	NA	NA
Hexachlorocyclopentadiene	-	36000	81000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Hexachloroethane	-	69000	1800000	370000	1400000	100000000	NA	NA	NA	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	-	-	210000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Isophorone	-	74000	2400000	-	-	8200000000	NA	NA	NA	NA	NA	NA	NA
Naphthalene	-	50000	160000000	77000000	59000000	15000000000	NA	NA	NA	NA	NA	NA	NA
Nitrobenzene	-	330	340000	490000	4600000	1500000000	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	-	330	3500	-	-	2000000	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodiphenylamine	-	14000	5100000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Octachlorocyclopentene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Pentachlorophenol	-	3200	63000	-	-	130000000	NA	NA	NA	NA	NA	NA	NA
Phenanthrene	-	34000	16000000	28000000	150000	59000000	NA	NA	NA	NA	NA	NA	NA
Phenol	-	260000	12000000	-	-	18000000000	NA	NA	NA	NA	NA	NA	NA
Pyrene	-	470000	340000000	1000000000	770000000	2900000000	NA	NA	NA	NA	NA	NA	NA
Volatiles (ug/Kg)													
1,1,1-Trichloroethane	-	4000	460000	460000	4500000	29000000000	ND	ND	NA	NA	NA	ND (10)	NA
1,1,2,2-Tetrachloroethane	-	340	120000	23000	34000	68000000	NA	NA	NA	NA	NA	ND (10)	NA
1,1,2-Trichloroethane	-	100	440000	24000	57000	250000000	NA	NA	NA	NA	NA	ND (10)	NA
1,1-Dichloroethane	-	50000	790000	790000	36000000	240000000000	ND	ND	NA	NA	NA	ND (10)	NA
1,1-Dichloroethene	-	140	580000	330	3700	78000000	ND	ND	NA	NA	NA	ND (10)	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							SB-9 (3-5) 11/6/96 AOI # 15	MW-2 (8-10) 11/6/96 AOI # 16	SB-2 (0-2) 11/7/96 AOI # 17	SB-2 (0-2) 11/7/96 AOI # 17	SB-2 (0-2) 11/7/96 AOI # 17	GP-3 (1-4) 8/30/96 AOI # 18	GP-3 (1-4) 8/30/96 AOI # 18
1,2-Dibromoethane (EDB)	-	10	290	3600	5800	18000000	NA	NA	NA	NA	NA	ND (10)	NA
1,2-Dichlorobenzene	-	13000	210000	210000	46000000	44000000000	NA	NA	NA	NA	NA	ND (10)	NA
1,2-Dichloroethane	-	100	270000	11000	21000	150000000	NA	NA	NA	NA	NA	ND (10)	NA
1,2-Dichloropropane	-	100	360000	7400	30000	120000000	NA	NA	NA	NA	NA	ND (10)	NA
1,3-Dichlorobenzene	-	18000	200000	-	-	-	NA	NA	NA	NA	NA	ND (10)	NA
1,4-Dichlorobenzene	-	1700	1000000	100000	260000	570000000	NA	NA	NA	NA	NA	ND (10)	NA
2-Butanone	-	760000	27000000	27000000	35000000	29000000000	NA	NA	NA	NA	NA	ND (100)	NA
2-Chloroethyl vinyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Hexanone	-	58000	2500000	1800000		1200000000	NA	NA	NA	NA	NA	ND (100)	NA
4-Methyl-2-pentanone	-	100000	2700000	2700000	53000000	60000000000	NA	NA	NA	NA	NA	ND (100)	NA
Acetone	-	42000	74000000	110000000	160000000	170000000000	NA	NA	NA	NA	NA	ND (100)	NA
Benzene	-	100	400000	8400	45000	470000000	NA	NA	NA	NA	NA	ND (10)	NA
Bromodichloromethane	-	2000	400000	6400	31000	110000000	NA	NA	NA	NA	NA	ND (10)	NA
Bromoform	-	2000	870000	770000	3100000	3600000000	NA	NA	NA	NA	NA	ND (10)	NA
Bromomethane	-	580	1000000	1600	13000	150000000	NA	NA	NA	NA	NA	ND (10)	NA
Carbon disulfide	-	46000	280000	140000	1600000	21000000000	NA	NA	NA	NA	NA	ND (100)	NA
Carbon tetrachloride	-	100	190000	990	12000	170000000	NA	NA	NA	NA	NA	ND (10)	NA
Chlorobenzene	-	2000	260000	220000	920000	2100000000	NA	NA	NA	NA	NA	ND (10)	NA
Chloroethane	-	18000	970000	970000	36000000	290000000000	NA	NA	NA	NA	NA	ND (10)	NA
Chloroform	-	2000	1500000	38000	150000	1600000000	ND	ND	NA	NA	NA	ND (10)	NA
Chloromethane	-	5400	1100000	12000	140000	6100000000	NA	NA	NA	NA	NA	ND (10)	NA
cis-1,2-Dichloroethene	-	1400	640000	640000	47000000	230000000000	NA	NA	NA	NA	NA	ND (10)	NA
cis-1,3-Dichloropropene	-	-	-	-	-	-	NA	NA	NA	NA	NA	ND (10)	NA
Dibromochloromethane	-	2000	300000	21000	80000	160000000	NA	NA	NA	NA	NA	ND (10)	NA
Dichlorodifluoromethane (CFC-12)	-	270000	1000000	1000000	63000000	1500000000000	NA	NA	NA	NA	NA	NA	NA
Ethylbenzene	-	1500	140000	140000	11000000	29000000000	ND	ND	NA	NA	NA	ND (10)	NA
Ethylene oxide	-	-	-	-	-	-	NA	NA	NA	NA	NA	ND (1000)	NA
m&p-Xylene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Methyl Tert Butyl Ether	-	800	6000000	6000000	31000000	88000000000	NA	NA	NA	NA	NA	ND (100)	NA
Methylene chloride	-	100	2300000	240000	700000	8300000000	83.1	ND	NA	NA	NA	14	NA
o-Xylene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Styrene	-	2700	520000	520000	3200000	6600000000	NA	NA	NA	NA	NA	ND (10)	NA
Tetrachloroethene	-	100	88000	60000	600000	68000000000	ND	ND	NA	NA	NA	ND (10)	NA
Toluene	-	16000	250000	250000	3300000	12000000000	2.9	ND	NA	NA	NA	ND (10)	NA
trans-1,2-Dichloroethene	-	2000	1400000	1400000	37000000	230000000000	ND	ND	NA	NA	NA	NA	ND (10)
trans-1,3-Dichloropropene	-	-	-	-	-	-	NA	NA	NA	NA	NA	ND (10)	NA
Trichloroethene	-	100	500000	37000	260000	2300000000	ND	ND	NA	NA	NA	ND (10)	NA
Trichlorofluoromethane (CFC-11)	-	150000	560000	560000	110000000	1700000000000	NA	NA	NA	NA	NA	NA	NA
Vinyl chloride	-	40	11000	150	1500	47000000	NA	NA	NA	NA	NA	ND (10)	NA
Xylene (total)	-	5600	150000	150000	54000000	130000000000	ND	ND	NA	NA	NA	ND (30)	NA

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							GP-4 (0-4) 8/30/96 AOI # 18	GP-4 (0-4) 8/30/96 AOI # 18	MW-3 (2-4) 11/6/96 AOI # 18	PFW-4 (1.5-3) 3/17/97 AOI # 18	SB-17 (1-3) 11/6/96 AOI # 18	SB-6 (1-3) 11/6/96 AOI # 18	SB-7 (1-3) 11/6/96 AOI # 18
General Chemistry													
Cyanide (total) (ug/Kg)	-	4000	250000	-	-	250000	NA	NA	NA	NA	NA	NA	NA
Total Solids (%)	-	-	-	-	-	-	NA	NA	NA	86.13	NA	NA	NA
Diesel Fuel (mg/Kg)	-	-	-	-	-	-	11	NA	NA	NA	NA	NA	NA
Petroleum hydrocarbons (ug/Kg)	-	-	-	-	-	-	NA	NA	396000	NA	308000	384000	556000
Metals (ug/Kg)													
Arsenic	5800	23000	100000	-	-	910000	7000	NA	NA	8000	NA	NA	NA
Barium	75000	1300000	320000000	-	-	150000000	73000	NA	NA	51000	NA	NA	NA
Cadmium	1200	6000	4500000	-	-	2200000	88	NA	NA	670	NA	NA	NA
Chromium	18000	30000	22000000	-	-	-	13000	NA	NA	17000	NA	NA	NA
Copper	32000	160000000	170000000	-	-	59000000	12000	NA	NA	13000	NA	NA	NA
Lead	21000	1000	900000.	-	-	44000000	10000	NA	NA	17000	NA	NA	NA
Mercury	130	1700	1400000	-	-	-	ND (100)	NA	NA	ND (100)	NA	NA	NA
Selenium	410	4000	23000000	-	-	59000000	800	NA	NA	ND (500)	NA	NA	NA
Silver	1000	13000	21000000	-	-	2900000	ND (500)	NA	NA	ND (500)	NA	NA	NA
Zinc	47000	5000000	1000000000	-	-	-	42000	NA	NA	38000	NA	NA	NA
PCBs (ug/Kg)													
Aroclor-1016	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1221	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1232	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1242	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1248	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1254	-	-	9900	-	-	-	NA	NA	ND	NA	ND	ND	ND
Aroclor-1260	-	-	9900	-	-	-	NA	NA	ND	NA	ND	ND	ND
Total PCBs	-	-	9900	16000000	820000	6500000	NA	NA	ND	ND (330)	ND	ND	ND
Semi - Volatiles (ug/Kg)													
1,2,4-Trichlorobenzene	-	4200	1100000	1100000	34000000	11000000000	NA	NA	NA	NA	NA	NA	NA
2,4,6-Trichlorophenol	-	45000	14000000	-	-	1300000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dichlorophenol	-	7700	10000000	-	-	2300000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dimethylphenol	-	20000	230000000	-	-	2100000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrotoluene	-	15000	220000	-	-	20000000	NA	NA	NA	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Chloronaphthalene	-	1800000	190000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Chlorophenol	-	2600	4600000	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Nitrophenol	-	1200	13000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
3,3-Dichlorobenzidine	-	2000	55000	-	-	8200000	NA	NA	NA	NA	NA	NA	NA
3-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	-	1700	1600000	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chloro-3-methylphenol	-	16000	15000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chloroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							GP-4 (0-4) 8/30/96 AOI # 18	GP-4 (0-4) 8/30/96 AOI # 18	MW-3 (2-4) 11/6/96 AOI # 18	PFW-4 (1.5-3) 3/17/97 AOI # 18	SB-17 (1-3) 11/6/96 AOI # 18	SB-6 (1-3) 11/6/96 AOI # 18	SB-7 (1-3) 11/6/96 AOI # 18
4-Nitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Acenaphthene	-	870000	810000000	350000000	97000000	6200000000	NA	NA	NA	ND (330)	NA	NA	NA
Acenaphthylene	-	8500	16000000	3000000	2700000	1000000000	NA	NA	NA	ND (330)	NA	NA	NA
Acetonitrile	-	8000	14000000	22000000	11000000	10000000000	ND (100)	NA	NA	NA	NA	NA	NA
Aniline	-	12000	4500000	-	-	29000000	NA	NA	NA	NA	NA	NA	NA
Anthracene	-	41000	1000000000	1000000000	1600000000	29000000000	NA	NA	NA	ND (330)	NA	NA	NA
Benzidine	-	1000	1000	-	-	59,000	NA	NA	NA	NA	NA	NA	NA
Benzo(a)anthracene	-	-	210000	-	-	-	NA	NA	ND	ND (330)	NA	NA	NA
Benzo(a)pyrene	-	-	21000	-	-	1900000	NA	NA	ND	ND (330)	NA	NA	NA
Benzo(b)fluoranthene	-	-	210000	-	-	-	NA	NA	ND	ND (330)	NA	NA	NA
Benzo(g,h,i)perylene	-	-	16000000	-	-	350000000	NA	NA	NA	ND (330)	NA	NA	NA
Benzo(k)fluoranthene	-	-	2100000	-	-	-	NA	NA	ND	ND (330)	NA	NA	NA
bis(2-Chloroethoxy)methane	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroethyl)ether	-	330	23000	44000	13000	12000000	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	-	-	10000000	-	-	8900000000	NA	NA	NA	NA	NA	NA	NA
Butyl benzylphthalate	-	310000	310000	-	-	21000000000	NA	NA	NA	NA	NA	NA	NA
Chrysene	-	-	21000000	-	-	-	NA	NA	NA	ND (330)	NA	NA	NA
Dibenzo(a,h)anthracene	-		21000	-	-	-	NA	NA	NA	ND (330)	NA	NA	NA
Dibenzofuran	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Diethyl phthalate	-	320000	740000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Dimethyl phthalate	-	790000	790000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Di-n-butylphthalate	-	760000	760000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Di-n-octyl phthalate	-	140000000	81000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Fluoranthene	-	720000	540000000	1000000000	880000000	4100000000	NA	NA	NA	ND (330)	NA	NA	NA
Fluorene	-	890000	540000000	1000000000	150000000	4100000000	NA	NA	NA	ND (330)	NA	NA	NA
Hexachlorobenzene	-	1800	94000	220000	56000	85000000	NA	NA	NA	NA	NA	NA	NA
Hexachlorobutadiene	-	77000	350000	350000	460000	180000000	NA	NA	NA	NA	NA	NA	NA
Hexachlorocyclopentadiene	-	36000	81000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Hexachloroethane	-	69000	1800000	370000	1400000	100000000	NA	NA	NA	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	-	-	210000	-	-	-	NA	NA	NA	ND (330)	NA	NA	NA
Isophorone	-	74000	2400000	-	-	8200000000	NA	NA	NA	NA	NA	NA	NA
Naphthalene	-	50000	160000000	77000000	59000000	15000000000	NA	NA	NA	ND (330)	NA	NA	NA
Nitrobenzene	-	330	340000	490000	4600000	1500000000	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	-	330	3500	-	-	2000000	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodiphenylamine	-	14000	5100000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Octachlorocyclopentene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Pentachlorophenol	-	3200	63000	-	-	130000000	NA	NA	NA	NA	NA	NA	NA
Phenanthrene	-	34000	16000000	28000000	150000	59000000	NA	NA	NA	ND (330)	NA	NA	NA
Phenol	-	260000	12000000	-	-	18000000000	NA	NA	NA	NA	NA	NA	NA
Pyrene	-	470000	340000000	1000000000	770000000	2900000000	NA	NA	NA	ND (330)	NA	NA	NA
Volatiles (ug/Kg)													
1,1,1-Trichloroethane	-	4000	460000	460000	4500000	29000000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
1,1,2,2-Tetrachloroethane	-	340	120000	23000	34000	68000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
1,1,2-Trichloroethane	-	100	440000	24000	57000	250000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
1,1-Dichloroethane	-	50000	790000	790000	36000000	240000000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
1,1-Dichloroethene	-	140	580000	330	3700	78000000	ND (10)	NA	NA	ND (10)	NA	NA	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels (2)	Industrial Drinking Water Protection Criteria (2)	Industrial Direct Contact Criteria (2)	Soil Volatilization to Indoor Air Inhalation Criteria (2)	Volatile Soil Inhalation Criteria (2)	Particulate Soil Inhalation Criteria (2)	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							GP-4 (0-4) 8/30/96 AOI # 18	GP-4 (0-4) 8/30/96 AOI # 18	MW-3 (2-4) 11/6/96 AOI # 18	PFW-4 (1.5-3) 3/17/97 AOI # 18	SB-17 (1-3) 11/6/96 AOI # 18	SB-6 (1-3) 11/6/96 AOI # 18	SB-7 (1-3) 11/6/96 AOI # 18
1,2-Dibromoethane (EDB)	-	10	290	3600	5800	18000000	ND (10)	NA	NA	NA	NA	NA	NA
1,2-Dichlorobenzene	-	13000	210000	210000	46000000	44000000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
1,2-Dichloroethane	-	100	270000	11000	21000	150000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
1,2-Dichloropropane	-	100	360000	7400	30000	120000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
1,3-Dichlorobenzene	-	18000	200000	-	-	-	ND (10)	NA	NA	ND (10)	NA	NA	NA
1,4-Dichlorobenzene	-	1700	1000000	100000	260000	570000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
2-Butanone	-	760000	27000000	27000000	35000000	29000000000	ND (100)	NA	NA	NA	NA	NA	NA
2-Chloroethyl vinyl ether	-	-	-	-	-	-	NA	NA	NA	ND (100)	NA	NA	NA
2-Hexanone	-	58000	2500000	1800000		1200000000	ND (100)	NA	NA	NA	NA	NA	NA
4-Methyl-2-pentanone	-	100000	2700000	2700000	53000000	60000000000	ND (100)	NA	NA	NA	NA	NA	NA
Acetone	-	42000	74000000	110000000	160000000	170000000000	ND (100)	NA	NA	NA	NA	NA	NA
Benzene	-	100	400000	8400	45000	470000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
Bromodichloromethane	-	2000	400000	6400	31000	110000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
Bromoform	-	2000	870000	770000	3100000	3600000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
Bromomethane	-	580	1000000	1600	13000	150000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
Carbon disulfide	-	46000	280000	140000	1600000	21000000000	ND (100)	NA	NA	NA	NA	NA	NA
Carbon tetrachloride	-	100	190000	990	12000	170000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
Chlorobenzene	-	2000	260000	220000	920000	2100000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
Chloroethane	-	18000	970000	970000	36000000	290000000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
Chloroform	-	2000	1500000	38000	150000	1600000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
Chloromethane	-	5400	1100000	12000	140000	6100000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
cis-1,2-Dichloroethene	-	1400	640000	640000	47000000	230000000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
cis-1,3-Dichloropropene	-	-	-	-	-	-	ND (10)	NA	NA	ND (10)	NA	NA	NA
Dibromochloromethane	-	2000	300000	21000	80000	160000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
Dichlorodifluoromethane (CFC-12)	-	270000	1000000	1000000	63000000	1500000000000	NA	NA	NA	ND (10)	NA	NA	NA
Ethylbenzene	-	1500	140000	140000	11000000	29000000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
Ethylene oxide	-	-	-	-	-	-	ND (1000)	NA	NA	NA	NA	NA	NA
m&p-Xylene	-	-	-	-	-	-	NA	NA	NA	ND (30)	NA	NA	NA
Methyl Tert Butyl Ether	-	800	6000000	6000000	31000000	88000000000	ND (100)	NA	NA	NA	NA	NA	NA
Methylene chloride	-	100	2300000	240000	700000	8300000000	17	NA	NA	ND (10)	NA	NA	NA
o-Xylene	-	-	-	-	-	-	NA	NA	NA	ND (30)	NA	NA	NA
Styrene	-	2700	520000	520000	3200000	66000000000	ND (10)	NA	NA	NA	NA	NA	NA
Tetrachloroethene	-	100	88000	60000	600000	68000000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
Toluene	-	16000	250000	250000	3300000	12000000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
trans-1,2-Dichloroethene	-	2000	1400000	1400000	37000000	230000000000	NA	ND (10)	NA	ND (10)	NA	NA	NA
trans-1,3-Dichloropropene	-	-	-	-	-	-	ND (10)	NA	NA	ND (10)	NA	NA	NA
Trichloroethene	-	100	500000	37000	260000	2300000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
Trichlorofluoromethane (CFC-11)	-	150000	560000	560000	110000000	1700000000000	NA	NA	NA	ND (10)	NA	NA	NA
Vinyl chloride	-	40	11000	150	1500	47000000	ND (10)	NA	NA	ND (10)	NA	NA	NA
Xylene (total)	-	5600	150000	150000	54000000	130000000000	ND (30)	NA	NA	NA	NA	NA	NA

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							SB-8 (1-3) 11/6/96 AOI # 18	PFB-21 (0-0.9) 4/24/97 AOI # 21	MW-5 (3-5) 11/6/96 None	MW-5 (8-10) 11/6/96 None	MW-6 (3-5) 11/6/96 None	MW-6 (8-10) 11/6/96 None	MW-7 (4-6) 11/6/96 None
General Chemistry													
Cyanide (total) (ug/Kg)	-	4000	250000	-	-	250000	NA	NA	NA	NA	NA	NA	NA
Total Solids (%)	-	-	-	-	-	-	NA	87.04	NA	NA	NA	NA	NA
Diesel Fuel (mg/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Petroleum hydrocarbons (ug/Kg)	-	-	-	-	-	-	ND	NA	ND	ND	ND	313000	360000
Metals (ug/Kg)													
Arsenic	5800	23000	100000	-	-	910000	NA	NA	7120	5340	5370	4650	6400
Barium	75000	1300000	320000000	-	-	150000000	NA	NA	52200	38400	49500	41000	68000
Cadmium	1200	6000	4500000	-	-	2200000	NA	NA	ND	ND	ND	ND	ND
Chromium	18000	30000	22000000	-	-	-	NA	NA	11700	12900	11200	12600	12900
Copper	32000	160000000	170000000	-	-	59000000	NA	NA	ND	ND	ND	ND	ND
Lead	21000	1000	900000	-	-	44000000	NA	NA	3720	6920	ND	3800	5610
Mercury	130	1700	1400000	-	-	-	NA	NA	ND	ND	30	ND	ND
Selenium	410	4000	23000000	-	-	59000000	NA	NA	260	ND	ND	ND	ND
Silver	1000	13000	21000000	-	-	2900000	NA	NA	ND	ND	ND	ND	ND
Zinc	47000	5000000	1000000000	-	-	-	NA	NA	ND	ND	ND	ND	ND
PCBs (ug/Kg)													
Aroclor-1016	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1221	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1232	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1242	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1248	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1254	-	-	9900	-	-	-	ND	NA	NA	NA	NA	NA	NA
Aroclor-1260	-	-	9900	-	-	-	ND	NA	NA	NA	NA	NA	NA
Total PCBs	-	-	9900	16000000	820000	6500000	ND	NA	NA	NA	NA	NA	NA
Semi - Volatiles (ug/Kg)													
1,2,4-Trichlorobenzene	-	4200	1100000	1100000	34000000	11000000000	NA	NA	NA	NA	NA	NA	NA
2,4,6-Trichlorophenol	-	45000	14000000	-	-	1300000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dichlorophenol	-	7700	10000000	-	-	2300000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dimethylphenol	-	20000	230000000	-	-	2100000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrotoluene	-	15000	220000	-	-	20000000	NA	NA	NA	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Chloronaphthalene	-	1800000	190000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Chlorophenol	-	2600	4600000	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Nitrophenol	-	1200	13000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
3,3-Dichlorobenzidine	-	2000	55000	-	-	8200000	NA	NA	NA	NA	NA	NA	NA
3-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	-	1700	1600000	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chloro-3-methylphenol	-	16000	15000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chloroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							SB-8 (1-3) 11/6/96 AOI # 18	PFB-21 (0-0.9) 4/24/97 AOI # 21	MW-5 (3-5) 11/6/96 None	MW-5 (8-10) 11/6/96 None	MW-6 (3-5) 11/6/96 None	MW-6 (8-10) 11/6/96 None	MW-7 (4-6) 11/6/96 None
4-Nitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Acenaphthene	-	870000	810000000	350000000	97000000	6200000000	NA	ND (540)	NA	NA	NA	NA	NA
Acenaphthylene	-	8500	16000000	3000000	2700000	1000000000	NA	ND (2200)	NA	NA	NA	NA	NA
Acetonitrile	-	8000	14000000	22000000	11000000	10000000000	NA	NA	NA	NA	NA	NA	NA
Aniline	-	12000	4500000	-	-	29000000	NA	NA	NA	NA	NA	NA	NA
Anthracene	-	41000	1000000000	1000000000	1600000000	29000000000	NA	ND (2000)	NA	NA	NA	NA	NA
Benzidine	-	1000	1000	-	-	59,000	NA	NA	NA	NA	NA	NA	NA
Benzo(a)anthracene	-	-	210000	-	-	-	NA	ND (850)	NA	NA	NA	NA	NA
Benzo(a)pyrene	-	-	21000	-	-	1900000	NA	ND (3400)	NA	NA	NA	NA	NA
Benzo(b)fluoranthene	-	-	210000	-	-	-	NA	ND (3600)	NA	NA	NA	NA	NA
Benzo(g,h,i)perylene	-	-	16000000	-	-	350000000	NA	ND (1200)	NA	NA	NA	NA	NA
Benzo(k)fluoranthene	-	-	2100000	-	-	-	NA	ND (1600)	NA	NA	NA	NA	NA
bis(2-Chloroethoxy)methane	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroethyl)ether	-	330	23000	44000	13000	12000000	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	-	-	10000000	-	-	8900000000	NA	NA	NA	NA	NA	NA	NA
Butyl benzylphthalate	-	310000	310000	-	-	21000000000	NA	NA	NA	NA	NA	NA	NA
Chrysene	-	-	21000000	-	-	-	NA	23000	NA	NA	NA	NA	NA
Dibenzo(a,h)anthracene	-		21000	-	-	-	NA	ND (2900)	NA	NA	NA	NA	NA
Dibenzofuran	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Diethyl phthalate	-	320000	740000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Dimethyl phthalate	-	790000	790000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Di-n-butylphthalate	-	760000	760000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Di-n-octyl phthalate	-	140000000	81000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Fluoranthene	-	720000	540000000	1000000000	880000000	4100000000	NA	ND (1400)	NA	NA	NA	NA	NA
Fluorene	-	890000	540000000	1000000000	150000000	4100000000	NA	ND (3300)	NA	NA	NA	NA	NA
Hexachlorobenzene	-	1800	94000	220000	56000	85000000	NA	NA	NA	NA	NA	NA	NA
Hexachlorobutadiene	-	77000	350000	350000	460000	180000000	NA	NA	NA	NA	NA	NA	NA
Hexachlorocyclopentadiene	-	36000	81000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Hexachloroethane	-	69000	1800000	370000	1400000	100000000	NA	NA	NA	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	-	-	210000	-	-	-	NA	ND (3000)	NA	NA	NA	NA	NA
Isophorone	-	74000	2400000	-	-	8200000000	NA	NA	NA	NA	NA	NA	NA
Naphthalene	-	50000	160000000	77000000	59000000	15000000000	NA	ND (930)	NA	NA	NA	NA	NA
Nitrobenzene	-	330	340000	490000	4600000	1500000000	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	-	330	3500	-	-	2000000	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodiphenylamine	-	14000	5100000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Octachlorocyclopentene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Pentachlorophenol	-	3200	63000	-	-	130000000	NA	NA	NA	NA	NA	NA	NA
Phenanthrene	-	34000	16000000	28000000	150000	59000000	NA	ND (1100)	NA	NA	NA	NA	NA
Phenol	-	260000	12000000	-	-	18000000000	NA	NA	NA	NA	NA	NA	NA
Pyrene	-	470000	340000000	1000000000	770000000	2900000000	NA	ND (870)	NA	NA	NA	NA	NA
Volatiles (ug/Kg)													
1,1,1-Trichloroethane	-	4000	460000	460000	4500000	29000000000	NA	ND (10)	ND	ND	ND	ND	ND
1,1,2,2-Tetrachloroethane	-	340	120000	23000	34000	68000000	NA	ND (10)	NA	NA	NA	NA	NA
1,1,2-Trichloroethane	-	100	440000	24000	57000	250000000	NA	ND (10)	NA	NA	NA	NA	NA
1,1-Dichloroethane	-	50000	790000	790000	36000000	240000000000	NA	ND (10)	ND	ND	ND	ND	ND
1,1-Dichloroethene	-	140	580000	330	3700	78000000	NA	ND (10)	ND	ND	ND	ND	ND

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels (2)	Industrial Drinking Water Protection Criteria (2)	Industrial Direct Contact Criteria (2)	Soil Volatilization to Indoor Air Inhalation Criteria (2)	Volatile Soil Inhalation Criteria (2)	Particulate Soil Inhalation Criteria (2)	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							SB-8 (1-3) 11/6/96 AOI # 18	PFB-21 (0-0.9) 4/24/97 AOI # 21	MW-5 (3-5) 11/6/96 None	MW-5 (8-10) 11/6/96 None	MW-6 (3-5) 11/6/96 None	MW-6 (8-10) 11/6/96 None	MW-7 (4-6) 11/6/96 None
1,2-Dibromoethane (EDB)	-	10	290	3600	5800	18000000	NA	NA	NA	NA	NA	NA	NA
1,2-Dichlorobenzene	-	13000	210000	210000	46000000	44000000000	NA	ND (10)	NA	NA	NA	NA	NA
1,2-Dichloroethane	-	100	270000	11000	21000	150000000	NA	ND (10)	NA	NA	NA	NA	NA
1,2-Dichloropropane	-	100	360000	7400	30000	120000000	NA	ND (10)	NA	NA	NA	NA	NA
1,3-Dichlorobenzene	-	18000	200000	-	-	-	NA	ND (10)	NA	NA	NA	NA	NA
1,4-Dichlorobenzene	-	1700	1000000	100000	260000	570000000	NA	ND (10)	NA	NA	NA	NA	NA
2-Butanone	-	760000	27000000	27000000	35000000	29000000000	NA	NA	NA	NA	NA	NA	NA
2-Chloroethyl vinyl ether	-	-	-	-	-	-	NA	ND (100)	NA	NA	NA	NA	NA
2-Hexanone	-	58000	2500000	1800000		1200000000	NA	NA	NA	NA	NA	NA	NA
4-Methyl-2-pentanone	-	100000	2700000	2700000	53000000	60000000000	NA	NA	NA	NA	NA	NA	NA
Acetone	-	42000	74000000	110000000	160000000	170000000000	NA	NA	NA	NA	NA	NA	NA
Benzene	-	100	400000	8400	45000	470000000	NA	ND (10)	NA	NA	NA	NA	NA
Bromodichloromethane	-	2000	400000	6400	31000	110000000	NA	ND (10)	NA	NA	NA	NA	NA
Bromoform	-	2000	870000	770000	3100000	3600000000	NA	ND (10)	NA	NA	NA	NA	NA
Bromomethane	-	580	1000000	1600	13000	150000000	NA	ND (10)	NA	NA	NA	NA	NA
Carbon disulfide	-	46000	280000	140000	1600000	21000000000	NA	NA	NA	NA	NA	NA	NA
Carbon tetrachloride	-	100	190000	990	12000	170000000	NA	ND (10)	NA	NA	NA	NA	NA
Chlorobenzene	-	2000	260000	220000	920000	2100000000	NA	ND (10)	NA	NA	NA	NA	NA
Chloroethane	-	18000	970000	970000	36000000	290000000000	NA	ND (10)	NA	NA	NA	NA	NA
Chloroform	-	2000	1500000	38000	150000	1600000000	NA	ND (10)	ND	ND	ND	ND	ND
Chloromethane	-	5400	1100000	12000	140000	6100000000	NA	ND (10)	NA	NA	NA	NA	NA
cis-1,2-Dichloroethene	-	1400	640000	640000	47000000	230000000000	NA	ND (10)	NA	NA	NA	NA	NA
cis-1,3-Dichloropropene	-	-	-	-	-	-	NA	ND (10)	NA	NA	NA	NA	NA
Dibromochloromethane	-	2000	300000	21000	80000	160000000	NA	ND (10)	NA	NA	NA	NA	NA
Dichlorodifluoromethane (CFC-12)	-	270000	1000000	1000000	63000000	1500000000000	NA	ND (10)	NA	NA	NA	NA	NA
Ethylbenzene	-	1500	140000	140000	11000000	29000000000	NA	ND (10)	ND	ND	ND	ND	ND
Ethylene oxide	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
m&p-Xylene	-	-	-	-	-	-	NA	95	NA	NA	NA	NA	NA
Methyl Tert Butyl Ether	-	800	6000000	6000000	31000000	88000000000	NA	NA	NA	NA	NA	NA	NA
Methylene chloride	-	100	2300000	240000	700000	8300000000	NA	ND (10)	ND	ND	ND	37.1	15.2
o-Xylene	-	-	-	-	-	-	NA	210	NA	NA	NA	NA	NA
Styrene	-	2700	520000	520000	3200000	6600000000	NA	NA	NA	NA	NA	NA	NA
Tetrachloroethene	-	100	88000	60000	600000	6800000000	NA	ND (10)	ND	ND	ND	ND	ND
Toluene	-	16000	250000	250000	3300000	12000000000	NA	ND (10)	ND	ND	ND	ND	0.9
trans-1,2-Dichloroethene	-	2000	1400000	1400000	37000000	230000000000	NA	ND (10)	ND	ND	ND	ND	ND
trans-1,3-Dichloropropene	-	-	-	-	-	-	NA	ND (10)	NA	NA	NA	NA	NA
Trichloroethene	-	100	500000	37000	260000	2300000000	NA	ND (10)	ND	ND	ND	ND	1.6
Trichlorofluoromethane (CFC-11)	-	150000	560000	560000	110000000	1700000000000	NA	ND (10)	NA	NA	NA	NA	NA
Vinyl chloride	-	40	11000	150	1500	47000000	NA	ND (10)	NA	NA	NA	NA	NA
Xylene (total)	-	5600	150000	150000	54000000	130000000000	NA	NA	ND	ND	ND	ND	ND

Notes:
PCBs - Polychlorinated Biphenyls.
TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.
ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.
NA - Parameter not analyzed.
(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							MW-8 (4-6) 11/6/96 None	MW-8 (8-10) 11/6/96 None	MW-9 (4-6) 11/6/96 None	MW-10 (4-6) 11/15/96 None	MW-10 (4-6) 11/5/96 AOI # 15	MW-10 (4-6) 11/5/96 None	MW-10 (4-6) 11/5/96 None
General Chemistry													
Cyanide (total) (ug/Kg)	-	4000	250000	-	-	250000	NA	NA	NA	NA	NA	NA	NA
Total Solids (%)	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	87.6
Diesel Fuel (mg/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Petroleum hydrocarbons (ug/Kg)	-	-	-	-	-	-	419000	ND	ND	ND	NA	56000	NA
Metals (ug/Kg)													
Arsenic	5800	23000	100000	-	-	910000	4190	6330	8270	4520	16000	NA	NA
Barium	75000	1300000	320000000	-	-	150000000	41700	39400	54200	42700	34000	NA	NA
Cadmium	1200	6000	4500000	-	-	2200000	ND	ND	ND	ND	110	NA	NA
Chromium	18000	30000	22000000	-	-	-	11800	12000	12000	12900	7200	NA	NA
Copper	32000	160000000	170000000	-	-	59000000	ND	ND	ND	ND	18000	NA	NA
Lead	21000	1000	900000	-	-	44000000	103000	7450	8890	6640	12000	NA	NA
Mercury	130	1700	1400000	-	-	-	ND	ND	ND	ND	ND (100)	NA	NA
Selenium	410	4000	23000000	-	-	59000000	370	ND	ND	ND	560	NA	NA
Silver	1000	13000	21000000	-	-	2900000	ND	ND	ND	ND	ND (500)	NA	NA
Zinc	47000	5000000	1000000000	-	-	-	ND	ND	ND	ND	30000	NA	NA
PCBs (ug/Kg)													
Aroclor-1016	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1221	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1232	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1242	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1248	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1254	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Aroclor-1260	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA	NA
Total PCBs	-	-	9900	16000000	820000	6500000	NA	NA	NA	NA	NA	NA	NA
Semi - Volatiles (ug/Kg)													
1,2,4-Trichlorobenzene	-	4200	1100000	1100000	34000000	11000000000	NA	NA	NA	NA	NA	NA	NA
2,4,6-Trichlorophenol	-	45000	14000000	-	-	1300000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dichlorophenol	-	7700	10000000	-	-	2300000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dimethylphenol	-	20000	230000000	-	-	2100000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrotoluene	-	15000	220000	-	-	20000000	NA	NA	NA	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Chloronaphthalene	-	1800000	190000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Chlorophenol	-	2600	4600000	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Nitrophenol	-	1200	13000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
3,3-Dichlorobenzidine	-	2000	55000	-	-	8200000	NA	NA	NA	NA	NA	NA	NA
3-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	-	1700	1600000	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chloro-3-methylphenol	-	16000	15000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chloroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							MW-8 (4-6) 11/6/96 None	MW-8 (8-10) 11/6/96 None	MW-9 (4-6) 11/6/96 None	MW-10 (4-6) 11/15/96 None	MW-10 (4-6) 11/5/96 AOI # 15	MW-10 (4-6) 11/5/96 None	MW-10 (4-6) 11/5/96 None
4-Nitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Acenaphthene	-	870000	810000000	350000000	97000000	6200000000	NA	NA	NA	NA	NA	NA	NA
Acenaphthylene	-	8500	16000000	3000000	2700000	1000000000	NA	NA	NA	NA	NA	NA	NA
Acetonitrile	-	8000	14000000	22000000	11000000	10000000000	NA	NA	NA	NA	NA	NA	NA
Aniline	-	12000	4500000	-	-	29000000	NA	NA	NA	NA	NA	NA	NA
Anthracene	-	41000	1000000000	1000000000	1600000000	29000000000	NA	NA	NA	NA	NA	NA	NA
Benzidine	-	1000	1000	-	-	59,000	NA	NA	NA	NA	NA	NA	NA
Benzo(a)anthracene	-	-	210000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Benzo(a)pyrene	-	-	21000	-	-	1900000	NA	NA	NA	NA	NA	NA	NA
Benzo(b)fluoranthene	-	-	210000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Benzo(g,h,i)perylene	-	-	16000000	-	-	350000000	NA	NA	NA	NA	NA	NA	NA
Benzo(k)fluoranthene	-	-	2100000	-	-	-	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroethoxy)methane	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroethyl)ether	-	330	23000	44000	13000	12000000	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	-	-	10000000	-	-	8900000000	NA	NA	NA	NA	NA	NA	NA
Butyl benzylphthalate	-	310000	310000	-	-	21000000000	NA	NA	NA	NA	NA	NA	NA
Chrysene	-	-	21000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Dibenzo(a,h)anthracene	-		21000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Dibenzofuran	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Diethyl phthalate	-	320000	740000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Dimethyl phthalate	-	790000	790000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Di-n-butylphthalate	-	760000	760000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Di-n-octyl phthalate	-	140000000	81000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Fluoranthene	-	720000	540000000	1000000000	880000000	4100000000	NA	NA	NA	NA	NA	NA	NA
Fluorene	-	890000	540000000	1000000000	150000000	4100000000	NA	NA	NA	NA	NA	NA	NA
Hexachlorobenzene	-	1800	94000	220000	56000	85000000	NA	NA	NA	NA	NA	NA	NA
Hexachlorobutadiene	-	77000	350000	350000	460000	180000000	NA	NA	NA	NA	NA	NA	NA
Hexachlorocyclopentadiene	-	36000	81000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Hexachloroethane	-	69000	1800000	370000	1400000	100000000	NA	NA	NA	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	-	-	210000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Isophorone	-	74000	2400000	-	-	8200000000	NA	NA	NA	NA	NA	NA	NA
Naphthalene	-	50000	160000000	77000000	59000000	15000000000	NA	NA	NA	NA	NA	NA	NA
Nitrobenzene	-	330	340000	490000	4600000	1500000000	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	-	330	3500	-	-	2000000	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodiphenylamine	-	14000	5100000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Octachlorocyclopentene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Pentachlorophenol	-	3200	63000	-	-	130000000	NA	NA	NA	NA	NA	NA	NA
Phenanthrene	-	34000	16000000	28000000	150000	59000000	NA	NA	NA	NA	NA	NA	NA
Phenol	-	260000	12000000	-	-	18000000000	NA	NA	NA	NA	NA	NA	NA
Pyrene	-	470000	340000000	1000000000	770000000	2900000000	NA	NA	NA	NA	NA	NA	NA
Volatiles (ug/Kg)													
1,1,1-Trichloroethane	-	4000	460000	460000	4500000	29000000000	ND	ND	ND	ND	ND (10)	NA	NA
1,1,2,2-Tetrachloroethane	-	340	120000	23000	34000	68000000	NA	NA	NA	NA	ND (10)	NA	NA
1,1,2-Trichloroethane	-	100	440000	24000	57000	250000000	NA	NA	NA	NA	ND (10)	NA	NA
1,1-Dichloroethane	-	50000	790000	790000	36000000	240000000000	ND	ND	ND	ND	ND (10)	NA	NA
1,1-Dichloroethene	-	140	580000	330	3700	78000000	ND	ND	ND	ND	ND (10)	NA	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							MW-8 (4-6) 11/6/96 None	MW-8 (8-10) 11/6/96 None	MW-9 (4-6) 11/6/96 None	MW-10 (4-6) 11/15/96 None	MW-10 (4-6) 11/5/96 AOI # 15	MW-10 (4-6) 11/5/96 None	MW-10 (4-6) 11/5/96 None
1,2-Dibromoethane (EDB)	-	10	290	3600	5800	18000000	NA	NA	NA	NA	NA	NA	NA
1,2-Dichlorobenzene	-	13000	210000	210000	46000000	44000000000	NA	NA	NA	NA	ND (10)	NA	NA
1,2-Dichloroethane	-	100	270000	11000	21000	150000000	NA	NA	NA	NA	ND (10)	NA	NA
1,2-Dichloropropane	-	100	360000	7400	30000	120000000	NA	NA	NA	NA	ND (10)	NA	NA
1,3-Dichlorobenzene	-	18000	200000	-	-	-	NA	NA	NA	NA	ND (10)	NA	NA
1,4-Dichlorobenzene	-	1700	1000000	100000	260000	570000000	NA	NA	NA	NA	ND (10)	NA	NA
2-Butanone	-	760000	27000000	27000000	35000000	29000000000	NA	NA	NA	NA	ND (100)	NA	NA
2-Chloroethyl vinyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Hexanone	-	58000	2500000	1800000		1200000000	NA	NA	NA	NA	ND (100)	NA	NA
4-Methyl-2-pentanone	-	100000	2700000	2700000	53000000	60000000000	NA	NA	NA	NA	ND (100)	NA	NA
Acetone	-	42000	74000000	110000000	160000000	170000000000	NA	NA	NA	NA	ND (100)	NA	NA
Benzene	-	100	400000	8400	45000	470000000	NA	NA	NA	NA	ND (10)	NA	NA
Bromodichloromethane	-	2000	400000	6400	31000	110000000	NA	NA	NA	NA	ND (10)	NA	NA
Bromoform	-	2000	870000	770000	3100000	3600000000	NA	NA	NA	NA	ND (10)	NA	NA
Bromomethane	-	580	1000000	1600	13000	150000000	NA	NA	NA	NA	ND (10)	NA	NA
Carbon disulfide	-	46000	280000	140000	1600000	21000000000	NA	NA	NA	NA	ND (100)	NA	NA
Carbon tetrachloride	-	100	190000	990	12000	170000000	NA	NA	NA	NA	ND (10)	NA	NA
Chlorobenzene	-	2000	260000	220000	920000	2100000000	NA	NA	NA	NA	ND (10)	NA	NA
Chloroethane	-	18000	970000	970000	36000000	290000000000	NA	NA	NA	NA	ND (10)	NA	NA
Chloroform	-	2000	1500000	38000	150000	1600000000	ND	ND	ND	ND	NA	ND (10)	NA
Chloromethane	-	5400	1100000	12000	140000	6100000000	NA	NA	NA	NA	ND (10)	NA	NA
cis-1,2-Dichloroethene	-	1400	640000	640000	47000000	230000000000	NA	NA	NA	NA	ND (10)	NA	NA
cis-1,3-Dichloropropene	-	-	-	-	-	-	NA	NA	NA	NA	ND (10)	NA	NA
Dibromochloromethane	-	2000	300000	21000	80000	160000000	NA	NA	NA	NA	ND (10)	NA	NA
Dichlorodifluoromethane (CFC-12)	-	270000	1000000	1000000	63000000	1500000000000	NA	NA	NA	NA	NA	NA	NA
Ethylbenzene	-	1500	140000	140000	11000000	29000000000	ND	5.8	ND	ND	NA	ND (10)	NA
Ethylene oxide	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
m&p-Xylene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Methyl Tert Butyl Ether	-	800	6000000	6000000	31000000	88000000000	NA	NA	NA	NA	NA	NA	NA
Methylene chloride	-	100	2300000	240000	700000	8300000000	54.1	11.7	8.8	ND	ND (10)	NA	NA
o-Xylene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Styrene	-	2700	520000	520000	3200000	6600000000	NA	NA	NA	NA	ND (10)	NA	NA
Tetrachloroethene	-	100	88000	60000	600000	6800000000	4.4	ND	ND	1	ND (10)	NA	NA
Toluene	-	16000	250000	250000	3300000	12000000000	ND	ND	ND	ND	ND (10)	NA	NA
trans-1,2-Dichloroethene	-	2000	1400000	1400000	37000000	230000000000	ND	ND	ND	ND	ND (10)	NA	NA
trans-1,3-Dichloropropene	-	-	-	-	-	-	NA	NA	NA	NA	ND (10)	NA	NA
Trichloroethene	-	100	500000	37000	260000	2300000000	ND	ND	0.9	1.1	ND (10)	NA	NA
Trichlorofluoromethane (CFC-11)	-	150000	560000	560000	110000000	1700000000000	NA	NA	NA	NA	NA	NA	NA
Vinyl chloride	-	40	11000	150	1500	47000000	NA	NA	NA	NA	ND (10)	NA	NA
Xylene (total)	-	5600	150000	150000	54000000	130000000000	ND	30.5	ND	ND	ND (30)	NA	NA

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI # GP-1 (4-8) 8/30/96 None	GP-2 (0-2) 8/30/96 None	GP-5 (0-4) 8/30/96 None	GP-5 (0-4) 8/30/96 None	GP-7 (10-12) 8/30/96 None
General Chemistry											
Cyanide (total) (ug/Kg)	-	4000	250000	-	-	250000	NA	NA	NA	NA	NA
Total Solids (%)	-	-	-	-	-	-	NA	NA	NA	NA	NA
Diesel Fuel (mg/Kg)	-	-	-	-	-	-	ND (6.6)	ND (6.6)	ND (6.6)	NA	ND (6.6)
Petroleum hydrocarbons (ug/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	NA
Metals (ug/Kg)											
Arsenic	5800	23000	100000	-	-	910000	8500	6600	3500	NA	7800
Barium	75000	1300000	320000000	-	-	150000000	57000	73000	64000	NA	86000
Cadmium	1200	6000	4500000	-	-	2200000	140	130	120	NA	160
Chromium	18000	30000	22000000	-	-	-	14000	11000	14000	NA	9500
Copper	32000	160000000	170000000	-	-	59000000	14000	13000	16000	NA	13000
Lead	21000	1000	900000	-	-	44000000	8800	8100	10000	NA	7500
Mercury	130	1700	1400000	-	-	-	ND (100)	ND (100)	ND (100)	NA	ND (100)
Selenium	410	4000	23000000	-	-	59000000	720	660	ND (500)	NA	680
Silver	1000	13000	21000000	-	-	2900000	ND (500)	ND (500)	ND (500)	NA	ND (500)
Zinc	47000	5000000	1000000000	-	-	-	40000	39000	49000	NA	39000
PCBs (ug/Kg)											
Aroclor-1016	-	-	9900	-	-	-	NA	NA	NA	NA	NA
Aroclor-1221	-	-	9900	-	-	-	NA	NA	NA	NA	NA
Aroclor-1232	-	-	9900	-	-	-	NA	NA	NA	NA	NA
Aroclor-1242	-	-	9900	-	-	-	NA	NA	NA	NA	NA
Aroclor-1248	-	-	9900	-	-	-	NA	NA	NA	NA	NA
Aroclor-1254	-	-	9900	-	-	-	NA	NA	NA	NA	NA
Aroclor-1260	-	-	9900	-	-	-	NA	NA	NA	NA	NA
Total PCBs	-	-	9900	16000000	820000	6500000	NA	NA	NA	NA	NA
Semi - Volatiles (ug/Kg)											
1,2,4-Trichlorobenzene	-	4200	1100000	1100000	34000000	11000000000	NA	NA	NA	NA	NA
2,4,6-Trichlorophenol	-	45000	14000000	-	-	1300000000	NA	NA	NA	NA	NA
2,4-Dichlorophenol	-	7700	10000000	-	-	2300000000	NA	NA	NA	NA	NA
2,4-Dimethylphenol	-	20000	230000000	-	-	2100000000	NA	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA
2,4-Dinitrotoluene	-	15000	220000	-	-	20000000	NA	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	-	-	-	NA	NA	NA	NA	NA
2-Chloronaphthalene	-	1800000	190000000	-	-	-	NA	NA	NA	NA	NA
2-Chlorophenol	-	2600	4600000	-	-	-	NA	NA	NA	NA	NA
2-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA
2-Nitrophenol	-	1200	13000000	-	-	-	NA	NA	NA	NA	NA
3,3-Dichlorobenzidine	-	2000	55000	-	-	8200000	NA	NA	NA	NA	NA
3-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	-	1700	1600000	-	-	-	NA	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA
4-Chloro-3-methylphenol	-	16000	15000000	-	-	-	NA	NA	NA	NA	NA
4-Chloroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA
4-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #				
							GP-1 (4-8) 8/30/96 None	GP-2 (0-2) 8/30/96 None	GP-5 (0-4) 8/30/96 None	GP-5 (0-4) 8/30/96 None	GP-7 (10-12) 8/30/96 None
4-Nitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA
Acenaphthene	-	870000	810000000	350000000	97000000	6200000000	NA	NA	NA	NA	NA
Acenaphthylene	-	8500	16000000	3000000	2700000	1000000000	NA	NA	NA	NA	NA
Acetonitrile	-	8000	14000000	22000000	11000000	10000000000	NA	NA	ND (100)	NA	NA
Aniline	-	12000	4500000	-	-	29000000	NA	NA	NA	NA	NA
Anthracene	-	41000	1000000000	1000000000	1600000000	29000000000	NA	NA	NA	NA	NA
Benzidine	-	1000	1000	-	-	59,000	NA	NA	NA	NA	NA
Benzo(a)anthracene	-	-	210000	-	-	-	NA	NA	NA	NA	NA
Benzo(a)pyrene	-	-	21000	-	-	1900000	NA	NA	NA	NA	NA
Benzo(b)fluoranthene	-	-	210000	-	-	-	NA	NA	NA	NA	NA
Benzo(g,h,i)perylene	-	-	16000000	-	-	350000000	NA	NA	NA	NA	NA
Benzo(k)fluoranthene	-	-	2100000	-	-	-	NA	NA	NA	NA	NA
bis(2-Chloroethoxy)methane	-	-	-	-	-	-	NA	NA	NA	NA	NA
bis(2-Chloroethyl)ether	-	330	23000	44000	13000	12000000	NA	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	-	-	-	NA	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	-	-	10000000	-	-	8900000000	NA	NA	NA	NA	NA
Butyl benzylphthalate	-	310000	310000	-	-	21000000000	NA	NA	NA	NA	NA
Chrysene	-	-	21000000	-	-	-	NA	NA	NA	NA	NA
Dibenzo(a,h)anthracene	-		21000	-	-	-	NA	NA	NA	NA	NA
Dibenzofuran	-	-	-	-	-	-	NA	NA	NA	NA	NA
Diethyl phthalate	-	320000	740000	-	-	1500000000	NA	NA	NA	NA	NA
Dimethyl phthalate	-	790000	790000	-	-	1500000000	NA	NA	NA	NA	NA
Di-n-butylphthalate	-	760000	760000	-	-	1500000000	NA	NA	NA	NA	NA
Di-n-octyl phthalate	-	140000000	81000000	-	-	-	NA	NA	NA	NA	NA
Fluoranthene	-	720000	540000000	1000000000	880000000	4100000000	NA	NA	NA	NA	NA
Fluorene	-	890000	540000000	1000000000	150000000	4100000000	NA	NA	NA	NA	NA
Hexachlorobenzene	-	1800	94000	220000	56000	85000000	NA	NA	NA	NA	NA
Hexachlorobutadiene	-	77000	350000	350000	460000	180000000	NA	NA	NA	NA	NA
Hexachlorocyclopentadiene	-	36000	81000	-	-	-	NA	NA	NA	NA	NA
Hexachloroethane	-	69000	1800000	370000	1400000	100000000	NA	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	-	-	210000	-	-	-	NA	NA	NA	NA	NA
Isophorone	-	74000	2400000	-	-	8200000000	NA	NA	NA	NA	NA
Naphthalene	-	50000	160000000	77000000	59000000	15000000000	NA	NA	NA	NA	NA
Nitrobenzene	-	330	340000	490000	4600000	1500000000	NA	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	-	330	3500	-	-	2000000	NA	NA	NA	NA	NA
N-Nitrosodiphenylamine	-	14000	5100000	-	-	-	NA	NA	NA	NA	NA
Octachlorocyclopentene	-	-	-	-	-	-	NA	NA	NA	NA	NA
Pentachlorophenol	-	3200	63000	-	-	130000000	NA	NA	NA	NA	NA
Phenanthrene	-	34000	16000000	28000000	150000	59000000	NA	NA	NA	NA	NA
Phenol	-	260000	12000000	-	-	18000000000	NA	NA	NA	NA	NA
Pyrene	-	470000	340000000	1000000000	770000000	2900000000	NA	NA	NA	NA	NA
Volatiles (ug/Kg)											
1,1,1-Trichloroethane	-	4000	460000	460000	4500000	29000000000	NA	NA	ND (10)	NA	NA
1,1,2,2-Tetrachloroethane	-	340	120000	23000	34000	68000000	NA	NA	ND (10)	NA	NA
1,1,2-Trichloroethane	-	100	440000	24000	57000	250000000	NA	NA	ND (10)	NA	NA
1,1-Dichloroethane	-	50000	790000	790000	36000000	240000000000	NA	NA	ND (10)	NA	NA
1,1-Dichloroethene	-	140	580000	330	3700	78000000	NA	NA	ND (10)	NA	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI # GP-1 (4-8) 8/30/96 None	GP-2 (0-2) 8/30/96 None	GP-5 (0-4) 8/30/96 None	GP-5 (0-4) 8/30/96 None	GP-7 (10-12) 8/30/96 None
1,2-Dibromoethane (EDB)	-	10	290	3600	5800	18000000	NA	NA	ND (10)	NA	NA
1,2-Dichlorobenzene	-	13000	210000	210000	46000000	44000000000	NA	NA	ND (10)	NA	NA
1,2-Dichloroethane	-	100	270000	11000	21000	150000000	NA	NA	ND (10)	NA	NA
1,2-Dichloropropane	-	100	360000	7400	30000	120000000	NA	NA	ND (10)	NA	NA
1,3-Dichlorobenzene	-	18000	200000	-	-	-	NA	NA	ND (10)	NA	NA
1,4-Dichlorobenzene	-	1700	1000000	100000	260000	570000000	NA	NA	ND (10)	NA	NA
2-Butanone	-	760000	27000000	27000000	35000000	29000000000	NA	NA	ND (100)	NA	NA
2-Chloroethyl vinyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA
2-Hexanone	-	58000	2500000	1800000		1200000000	NA	NA	ND (100)	NA	NA
4-Methyl-2-pentanone	-	100000	2700000	2700000	53000000	60000000000	NA	NA	ND (100)	NA	NA
Acetone	-	42000	74000000	110000000	160000000	170000000000	NA	NA	ND (100)	NA	NA
Benzene	-	100	400000	8400	45000	470000000	NA	NA	ND (10)	NA	NA
Bromodichloromethane	-	2000	400000	6400	31000	110000000	NA	NA	ND (10)	NA	NA
Bromoform	-	2000	870000	770000	3100000	3600000000	NA	NA	ND (10)	NA	NA
Bromomethane	-	580	1000000	1600	13000	150000000	NA	NA	ND (10)	NA	NA
Carbon disulfide	-	46000	280000	140000	1600000	21000000000	NA	NA	ND (100)	NA	NA
Carbon tetrachloride	-	100	190000	990	12000	170000000	NA	NA	ND (10)	NA	NA
Chlorobenzene	-	2000	260000	220000	920000	2100000000	NA	NA	ND (10)	NA	NA
Chloroethane	-	18000	970000	970000	36000000	290000000000	NA	NA	ND (10)	NA	NA
Chloroform	-	2000	1500000	38000	150000	1600000000	NA	NA	ND (10)	NA	NA
Chloromethane	-	5400	1100000	12000	140000	6100000000	NA	NA	ND (10)	NA	NA
cis-1,2-Dichloroethene	-	1400	640000	640000	47000000	230000000000	NA	NA	ND (10)	NA	NA
cis-1,3-Dichloropropene	-	-	-	-	-	-	NA	NA	ND (10)	NA	NA
Dibromochloromethane	-	2000	300000	21000	80000	160000000	NA	NA	ND (10)	NA	NA
Dichlorodifluoromethane (CFC-12)	-	270000	1000000	1000000	63000000	1500000000000	NA	NA	NA	NA	NA
Ethylbenzene	-	1500	140000	140000	11000000	29000000000	NA	NA	ND (10)	NA	NA
Ethylene oxide	-	-	-	-	-	-	NA	NA	ND (1000)	NA	NA
m&p-Xylene	-	-	-	-	-	-	NA	NA	NA	NA	NA
Methyl Tert Butyl Ether	-	800	6000000	6000000	31000000	88000000000	NA	NA	ND (100)	NA	NA
Methylene chloride	-	100	2300000	240000	700000	8300000000	NA	NA	17	NA	NA
o-Xylene	-	-	-	-	-	-	NA	NA	NA	NA	NA
Styrene	-	2700	520000	520000	3200000	6600000000	NA	NA	ND (10)	NA	NA
Tetrachloroethene	-	100	88000	60000	600000	6800000000	NA	NA	ND (10)	NA	NA
Toluene	-	16000	250000	250000	3300000	12000000000	NA	NA	ND (10)	NA	NA
trans-1,2-Dichloroethene	-	2000	1400000	1400000	37000000	230000000000	NA	NA	NA	ND (10)	NA
trans-1,3-Dichloropropene	-	-	-	-	-	-	NA	NA	ND (10)	NA	NA
Trichloroethene	-	100	500000	37000	260000	2300000000	NA	NA	ND (10)	NA	NA
Trichlorofluoromethane (CFC-11)	-	150000	560000	560000	110000000	1700000000000	NA	NA	NA	NA	NA
Vinyl chloride	-	40	11000	150	1500	47000000	NA	NA	ND (10)	NA	NA
Xylene (total)	-	5600	150000	150000	54000000	130000000000	NA	NA	ND (30)	NA	NA

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI # HD-1 11/6/96 None	MW-12 (10-12) 11/6/96 None	MW-12 (10-12) 11/6/96 None	MW-12 (10-12) 11/6/96 None	PFW-8 (0-2) 3/19/97 None	PFW-8 (6-7.8) 3/19/97 None
General Chemistry												
Cyanide (total) (ug/Kg)	-	4000	250000	-	-	250000	NA	NA	NA	88300	NA	NA
Total Solids (%)	-	-	-	-	-	-	NA	89	NA	NA	87.9	88.03
Diesel Fuel (mg/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
Petroleum hydrocarbons (ug/Kg)	-	-	-	-	-	-	978000	NA	ND (40000)	ND	NA	NA
Metals (ug/Kg)												
Arsenic	5800	23000	100000	-	-	910000	3630	5700	NA	6880	6000	NA
Barium	75000	1300000	320000000	-	-	150000000	20100	58000	NA	54900	61000	NA
Cadmium	1200	6000	4500000	-	-	2200000	ND	92	NA	ND	680	NA
Chromium	18000	30000	22000000	-	-	-	25200	8300	NA	11300	15000	NA
Copper	32000	160000000	170000000	-	-	59000000	ND	22000	NA	ND	11000	NA
Lead	21000	1000	900000	-	-	44000000	175000	13000	NA	6380	15000	NA
Mercury	130	1700	1400000	-	-	-	ND	NA	ND (100)	ND	ND (100)	NA
Selenium	410	4000	23000000	-	-	59000000	ND	590	NA	300	ND (500)	NA
Silver	1000	13000	21000000	-	-	2900000	ND	NA	ND (500)	ND	600	NA
Zinc	47000	5000000	1000000000	-	-	-	ND	26000	NA	ND	37000	NA
PCBs (ug/Kg)												
Aroclor-1016	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1221	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1232	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1242	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1248	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1254	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Aroclor-1260	-	-	9900	-	-	-	NA	NA	NA	NA	NA	NA
Total PCBs	-	-	9900	16000000	820000	6500000	NA	NA	NA	NA	ND (330)	NA
Semi - Volatiles (ug/Kg)												
1,2,4-Trichlorobenzene	-	4200	1100000	1100000	34000000	11000000000	NA	NA	NA	NA	NA	NA
2,4,6-Trichlorophenol	-	45000	14000000	-	-	1300000000	NA	NA	NA	NA	NA	NA
2,4-Dichlorophenol	-	7700	10000000	-	-	2300000000	NA	NA	NA	NA	NA	NA
2,4-Dimethylphenol	-	20000	230000000	-	-	2100000000	NA	NA	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
2,4-Dinitrotoluene	-	15000	220000	-	-	20000000	NA	NA	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
2-Chloronaphthalene	-	1800000	190000000	-	-	-	NA	NA	NA	NA	NA	NA
2-Chlorophenol	-	2600	4600000	-	-	-	NA	NA	NA	NA	NA	NA
2-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
2-Nitrophenol	-	1200	13000000	-	-	-	NA	NA	NA	NA	NA	NA
3,3-Dichlorobenzidine	-	2000	55000	-	-	8200000	NA	NA	NA	NA	NA	NA
3-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	-	1700	1600000	-	-	-	NA	NA	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
4-Chloro-3-methylphenol	-	16000	15000000	-	-	-	NA	NA	NA	NA	NA	NA
4-Chloroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
4-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI # HD-1 11/6/96 None	MW-12 (10-12) 11/6/96 None	MW-12 (10-12) 11/6/96 None	MW-12 (10-12) 11/6/96 None	PFW-8 (0-2) 3/19/97 None	PFW-8 (6-7.8) 3/19/97 None
4-Nitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
Acenaphthene	-	870000	810000000	350000000	97000000	6200000000	NA	NA	NA	NA	ND (330)	NA
Acenaphthylene	-	8500	16000000	3000000	2700000	1000000000	NA	NA	NA	NA	ND (330)	NA
Acetonitrile	-	8000	14000000	22000000	11000000	10000000000	NA	NA	NA	NA	NA	NA
Aniline	-	12000	4500000	-	-	29000000	NA	NA	NA	NA	NA	NA
Anthracene	-	41000	1000000000	1000000000	1600000000	29000000000	NA	NA	NA	NA	ND (330)	NA
Benzidine	-	1000	1000	-	-	59,000	NA	NA	NA	NA	NA	NA
Benzo(a)anthracene	-	-	210000	-	-	-	NA	NA	NA	NA	ND (330)	NA
Benzo(a)pyrene	-	-	21000	-	-	1900000	NA	NA	NA	NA	ND (330)	NA
Benzo(b)fluoranthene	-	-	210000	-	-	-	NA	NA	NA	NA	ND (330)	NA
Benzo(g,h,i)perylene	-	-	16000000	-	-	350000000	NA	NA	NA	NA	ND (330)	NA
Benzo(k)fluoranthene	-	-	2100000	-	-	-	NA	NA	NA	NA	NA	NA
bis(2-Chloroethoxy)methane	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
bis(2-Chloroethyl)ether	-	330	23000	44000	13000	12000000	NA	NA	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	-	-	10000000	-	-	8900000000	NA	NA	NA	NA	NA	NA
Butyl benzylphthalate	-	310000	310000	-	-	21000000000	NA	NA	NA	NA	NA	NA
Chrysene	-	-	21000000	-	-	-	NA	NA	NA	NA	ND (330)	NA
Dibenzo(a,h)anthracene	-		21000	-	-	-	NA	NA	NA	NA	ND (330)	NA
Dibenzofuran	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
Diethyl phthalate	-	320000	740000	-	-	1500000000	NA	NA	NA	NA	NA	NA
Dimethyl phthalate	-	790000	790000	-	-	1500000000	NA	NA	NA	NA	NA	NA
Di-n-butylphthalate	-	760000	760000	-	-	1500000000	NA	NA	NA	NA	NA	NA
Di-n-octyl phthalate	-	140000000	81000000	-	-	-	NA	NA	NA	NA	NA	NA
Fluoranthene	-	720000	540000000	1000000000	880000000	4100000000	NA	NA	NA	NA	ND (330)	NA
Fluorene	-	890000	540000000	1000000000	150000000	4100000000	NA	NA	NA	NA	ND (330)	NA
Hexachlorobenzene	-	1800	94000	220000	56000	85000000	NA	NA	NA	NA	NA	NA
Hexachlorobutadiene	-	77000	350000	350000	460000	180000000	NA	NA	NA	NA	NA	NA
Hexachlorocyclopentadiene	-	36000	81000	-	-	-	NA	NA	NA	NA	NA	NA
Hexachloroethane	-	69000	1800000	370000	1400000	100000000	NA	NA	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	-	-	210000	-	-	-	NA	NA	NA	NA	ND (330)	NA
Isophorone	-	74000	2400000	-	-	8200000000	NA	NA	NA	NA	NA	NA
Naphthalene	-	50000	160000000	77000000	59000000	15000000000	NA	NA	NA	NA	ND (330)	NA
Nitrobenzene	-	330	340000	490000	4600000	1500000000	NA	NA	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	-	330	3500	-	-	2000000	NA	NA	NA	NA	NA	NA
N-Nitrosodiphenylamine	-	14000	5100000	-	-	-	NA	NA	NA	NA	NA	NA
Octachlorocyclopentene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
Pentachlorophenol	-	3200	63000	-	-	130000000	NA	NA	NA	NA	NA	NA
Phenanthrene	-	34000	16000000	28000000	150000	59000000	NA	NA	NA	NA	ND (330)	NA
Phenol	-	260000	12000000	-	-	18000000000	NA	NA	NA	NA	NA	NA
Pyrene	-	470000	340000000	1000000000	770000000	2900000000	NA	NA	NA	NA	ND (330)	NA
Volatiles (ug/Kg)												
1,1,1-Trichloroethane	-	4000	460000	460000	4500000	29000000000	NA	NA	ND (10)	ND	NA	ND (10)
1,1,2,2-Tetrachloroethane	-	340	120000	23000	34000	68000000	NA	ND (10)	NA	NA	NA	ND (10)
1,1,2-Trichloroethane	-	100	440000	24000	57000	250000000	NA	ND (10)	NA	NA	NA	ND (10)
1,1-Dichloroethane	-	50000	790000	790000	36000000	240000000000	NA	NA	ND (10)	ND	NA	ND (10)
1,1-Dichloroethene	-	140	580000	330	3700	78000000	NA	NA	ND (10)	ND	NA	ND (10)

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI # HD-1 11/6/96 None	MW-12 (10-12) 11/6/96 None	MW-12 (10-12) 11/6/96 None	MW-12 (10-12) 11/6/96 None	PFW-8 (0-2) 3/19/97 None	PFW-8 (6-7.8) 3/19/97 None
1,2-Dibromoethane (EDB)	-	10	290	3600	5800	18000000	NA	NA	NA	NA	NA	NA
1,2-Dichlorobenzene	-	13000	210000	210000	46000000	44000000000	NA	ND (10)	NA	NA	NA	ND (10)
1,2-Dichloroethane	-	100	270000	11000	21000	150000000	NA	ND (10)	NA	NA	NA	ND (10)
1,2-Dichloropropane	-	100	360000	7400	30000	120000000	NA	ND (10)	NA	NA	NA	ND (10)
1,3-Dichlorobenzene	-	18000	200000	-	-	-	NA	ND (10)	NA	NA	NA	ND (10)
1,4-Dichlorobenzene	-	1700	1000000	100000	260000	570000000	NA	ND (10)	NA	NA	NA	ND (10)
2-Butanone	-	760000	27000000	27000000	35000000	29000000000	NA	ND (100)	NA	NA	NA	NA
2-Chloroethyl vinyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	ND (100)
2-Hexanone	-	58000	2500000	1800000		1200000000	NA	ND (100)	NA	NA	NA	NA
4-Methyl-2-pentanone	-	100000	2700000	2700000	53000000	60000000000	NA	ND (100)	NA	NA	NA	NA
Acetone	-	42000	74000000	110000000	160000000	170000000000	NA	ND (100)	NA	NA	NA	NA
Benzene	-	100	400000	8400	45000	470000000	NA	ND (10)	NA	NA	NA	ND (10)
Bromodichloromethane	-	2000	400000	6400	31000	110000000	NA	ND (10)	NA	NA	NA	ND (10)
Bromoform	-	2000	870000	770000	3100000	3600000000	NA	ND (10)	NA	NA	NA	ND (10)
Bromomethane	-	580	1000000	1600	13000	150000000	NA	ND (10)	NA	NA	NA	ND (10)
Carbon disulfide	-	46000	280000	140000	1600000	21000000000	NA	ND (100)	NA	NA	NA	NA
Carbon tetrachloride	-	100	190000	990	12000	170000000	NA	ND (10)	NA	NA	NA	ND (10)
Chlorobenzene	-	2000	260000	220000	920000	2100000000	NA	ND (10)	NA	NA	NA	ND (10)
Chloroethane	-	18000	970000	970000	36000000	2900000000000	NA	ND (10)	NA	NA	NA	ND (10)
Chloroform	-	2000	1500000	38000	150000	1600000000	NA	ND (10)	NA	ND	NA	ND (10)
Chloromethane	-	5400	1100000	12000	140000	6100000000	NA	ND (10)	NA	NA	NA	ND (10)
cis-1,2-Dichloroethene	-	1400	640000	640000	47000000	230000000000	NA	ND (10)	NA	NA	NA	ND (10)
cis-1,3-Dichloropropene	-	-	-	-	-	-	NA	ND (10)	NA	NA	NA	ND (10)
Dibromochloromethane	-	2000	300000	21000	80000	160000000	NA	ND (10)	NA	NA	NA	ND (10)
Dichlorodifluoromethane (CFC-12)	-	270000	1000000	1000000	63000000	1500000000000	NA	NA	NA	NA	NA	ND (10)
Ethylbenzene	-	1500	140000	140000	11000000	29000000000	NA	ND (10)	NA	ND	NA	ND (10)
Ethylene oxide	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA
m&p-Xylene	-	-	-	-	-	-	NA	NA	NA	NA	NA	ND (30)
Methyl Tert Butyl Ether	-	800	6000000	6000000	31000000	88000000000	NA	NA	NA	NA	NA	NA
Methylene chloride	-	100	2300000	240000	700000	8300000000	NA	ND (10)	NA	ND	NA	ND (10)
o-Xylene	-	-	-	-	-	-	NA	NA	NA	NA	NA	ND (30)
Styrene	-	2700	520000	520000	3200000	6600000000	NA	ND (10)	NA	NA	NA	NA
Tetrachloroethene	-	100	88000	60000	600000	6800000000	NA	ND (10)	NA	ND	NA	ND (10)
Toluene	-	16000	250000	250000	3300000	12000000000	NA	ND (10)	NA	ND	NA	ND (10)
trans-1,2-Dichloroethene	-	2000	1400000	1400000	37000000	230000000000	NA	ND (10)	NA	ND	NA	ND (10)
trans-1,3-Dichloropropene	-	-	-	-	-	-	NA	ND (10)	NA	NA	NA	ND (10)
Trichloroethene	-	100	500000	37000	260000	2300000000	NA	ND (10)	NA	ND	NA	ND (10)
Trichlorofluoromethane (CFC-11)	-	150000	560000	560000	110000000	1700000000000	NA	NA	NA	NA	NA	ND (10)
Vinyl chloride	-	40	11000	150	1500	47000000	NA	ND (10)	NA	NA	NA	ND (10)
Xylene (total)	-	5600	150000	150000	54000000	130000000000	NA	ND (30)	NA	ND	NA	NA

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	*Statewide Default Background Levels* [2]	*Industrial Drinking Water Protection Criteria* [2]	*Industrial Direct Contact Criteria* [2]	*Soil Volatilization to Indoor Air Inhalation Criteria* [2]	*Volatile Soil Inhalation Criteria* [2]	*Particulate Soil Inhalation Criteria* [2]	*Sample Location (depth ft. bgs)* / *Sample Date* / *AOI #*						
							Q-12 11/6/96 None	*SPL* 11/6/96 None	*WWB (0.75-2.75)* 11/5/96 None	*WWB (0.75-2.75)* 11/5/96 None	*WWB (0.75-2.75)* 11/5/96 None	*WWT-1* 11/6/96 None	*WWT-2* 11/6/96 None
General Chemistry													
Cyanide (total) (ug/Kg)	-	4000	250000	-	-	250000	NA	NA	NA	NA	NA	ND	ND
Total Solids (%)	-	-	-	-	-	-	NA	NA	87.1	NA	NA	NA	NA
Diesel Fuel (mg/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Petroleum hydrocarbons (ug/Kg)	-	-	-	-	-	-	NA	NA	NA	NA	ND	NA	NA
Metals (ug/Kg)													
Arsenic	5800	23000	100000	-	-	910000	5980	6030	NA	NA	NA	5370	6160
Barium	75000	1300000	320000000	-	-	150000000	44100	60600	NA	NA	NA	66600	45200
Cadmium	1200	6000	4500000	-	-	2200000	ND	ND	NA	NA	NA	ND	ND
Chromium	18000	30000	22000000	-	-	-	11600	13900	NA	NA	NA	19800	20900
Copper	32000	160000000	170000000	-	-	59000000	ND	ND	NA	NA	NA	ND	ND
Lead	21000	1000	900000	-	-	44000000	7200	6040	NA	NA	NA	6400	ND
Mercury	130	1700	1400000	-	-	-	ND	ND	NA	NA	NA	ND	ND
Selenium	410	4000	23000000	-	-	59000000	ND	ND	NA	NA	NA	ND	ND
Silver	1000	13000	21000000	-	-	2900000	ND	ND	NA	NA	NA	ND	ND
Zinc	47000	5000000	1000000000	-	-	-	ND	ND	NA	NA	NA	ND	ND
PCBs (ug/Kg)													
Aroclor-1016	-	-	9900	-	-	-	NA	NA	ND (330)	NA	NA	NA	NA
Aroclor-1221	-	-	9900	-	-	-	NA	NA	ND (330)	NA	NA	NA	NA
Aroclor-1232	-	-	9900	-	-	-	NA	NA	ND (330)	NA	NA	NA	NA
Aroclor-1242	-	-	9900	-	-	-	NA	NA	ND (330)	NA	NA	NA	NA
Aroclor-1248	-	-	9900	-	-	-	NA	NA	ND (330)	NA	NA	NA	NA
Aroclor-1254	-	-	9900	-	-	-	NA	NA	NA	ND (330)	ND	NA	NA
Aroclor-1260	-	-	9900	-	-	-	NA	NA	NA	ND (330)	ND	NA	NA
Total PCBs	-	-	9900	16000000	820000	6500000	NA	NA	NA	ND (330)	ND	NA	NA
Semi - Volatiles (ug/Kg)													
1,2,4-Trichlorobenzene	-	4200	1100000	1100000	34000000	11000000000	NA	NA	NA	NA	NA	NA	NA
2,4,6-Trichlorophenol	-	45000	14000000	-	-	1300000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dichlorophenol	-	7700	10000000	-	-	2300000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dimethylphenol	-	20000	230000000	-	-	2100000000	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrotoluene	-	15000	220000	-	-	20000000	NA	NA	NA	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Chloronaphthalene	-	1800000	190000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Chlorophenol	-	2600	4600000	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Nitrophenol	-	1200	13000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
3,3-Dichlorobenzidine	-	2000	55000	-	-	8200000	NA	NA	NA	NA	NA	NA	NA
3-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	-	1700	1600000	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chloro-3-methylphenol	-	16000	15000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chloroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
4-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							Q-12 11/6/96 None	SPL 11/6/96 None	WWB (0.75-2.75) 11/5/96 None	WWB (0.75-2.75) 11/5/96 None	WWB (0.75-2.75) 11/5/96 None	WWT-1 11/6/96 None	WWT-2 11/6/96 None
4-Nitrophenol	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Acenaphthene	-	870000	810000000	350000000	97000000	6200000000	NA	NA	ND (330)	NA	NA	NA	NA
Acenaphthylene	-	8500	16000000	3000000	2700000	1000000000	NA	NA	ND (330)	NA	NA	NA	NA
Acetonitrile	-	8000	14000000	22000000	11000000	10000000000	NA	NA	NA	NA	NA	NA	NA
Aniline	-	12000	4500000	-	-	29000000	NA	NA	NA	NA	NA	NA	NA
Anthracene	-	41000	1000000000	1000000000	1600000000	29000000000	NA	NA	ND (330)	NA	NA	NA	NA
Benzidine	-	1000	1000	-	-	59,000	NA	NA	NA	NA	NA	NA	NA
Benzo(a)anthracene	-	-	210000	-	-	-	NA	NA	NA	ND (330)	ND	NA	NA
Benzo(a)pyrene	-	-	21000	-	-	1900000	NA	NA	ND (330)	NA	ND	NA	NA
Benzo(b)fluoranthene	-	-	210000	-	-	-	NA	NA	ND (330)	NA	ND	NA	NA
Benzo(g,h,i)perylene	-	-	16000000	-	-	350000000	NA	NA	ND (330)	NA	NA	NA	NA
Benzo(k)fluoranthene	-	-	2100000	-	-	-	NA	NA	ND (330)	NA	ND	NA	NA
bis(2-Chloroethoxy)methane	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroethyl)ether	-	330	23000	44000	13000	12000000	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	-	-	10000000	-	-	8900000000	NA	NA	NA	NA	NA	NA	NA
Butyl benzylphthalate	-	310000	310000	-	-	21000000000	NA	NA	NA	NA	NA	NA	NA
Chrysene	-	-	21000000	-	-	-	NA	NA	ND (330)	NA	NA	NA	NA
Dibenzo(a,h)anthracene	-		21000	-	-	-	NA	NA	ND (330)	NA	NA	NA	NA
Dibenzofuran	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Diethyl phthalate	-	320000	740000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Dimethyl phthalate	-	790000	790000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Di-n-butylphthalate	-	760000	760000	-	-	1500000000	NA	NA	NA	NA	NA	NA	NA
Di-n-octyl phthalate	-	140000000	81000000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Fluoranthene	-	720000	540000000	1000000000	880000000	4100000000	NA	NA	ND (330)	NA	NA	NA	NA
Fluorene	-	890000	540000000	1000000000	150000000	4100000000	NA	NA	ND (330)	NA	NA	NA	NA
Hexachlorobenzene	-	1800	94000	220000	56000	85000000	NA	NA	NA	NA	NA	NA	NA
Hexachlorobutadiene	-	77000	350000	350000	460000	180000000	NA	NA	NA	NA	NA	NA	NA
Hexachlorocyclopentadiene	-	36000	81000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Hexachloroethane	-	69000	1800000	370000	1400000	100000000	NA	NA	NA	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	-	-	210000	-	-	-	NA	NA	ND (330)	NA	NA	NA	NA
Isophorone	-	74000	2400000	-	-	8200000000	NA	NA	NA	NA	NA	NA	NA
Naphthalene	-	50000	160000000	77000000	59000000	15000000000	NA	NA	ND (330)	NA	NA	NA	NA
Nitrobenzene	-	330	340000	490000	4600000	1500000000	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	-	330	3500	-	-	2000000	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodiphenylamine	-	14000	5100000	-	-	-	NA	NA	NA	NA	NA	NA	NA
Octachlorocyclopentene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Pentachlorophenol	-	3200	63000	-	-	130000000	NA	NA	NA	NA	NA	NA	NA
Phenanthrene	-	34000	16000000	28000000	150000	59000000	NA	NA	ND (330)	NA	NA	NA	NA
Phenol	-	260000	12000000	-	-	18000000000	NA	NA	NA	NA	NA	NA	NA
Pyrene	-	470000	340000000	1000000000	770000000	2900000000	NA	NA	ND (330)	NA	NA	NA	NA
Volatiles (ug/Kg)													
1,1,1-Trichloroethane	-	4000	460000	460000	4500000	29000000000	ND	ND	NA	NA	NA	ND	ND
1,1,2,2-Tetrachloroethane	-	340	120000	23000	34000	68000000	NA	NA	NA	NA	NA	NA	NA
1,1,2-Trichloroethane	-	100	440000	24000	57000	250000000	NA	NA	NA	NA	NA	NA	NA
1,1-Dichloroethane	-	50000	790000	790000	36000000	240000000000	ND	ND	NA	NA	NA	ND	ND
1,1-Dichloroethene	-	140	580000	330	3700	78000000	ND	ND	NA	NA	NA	ND	ND

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #						
							Q-12 11/6/96 None	SPL 11/6/96 None	WWB (0.75-2.75) 11/5/96 None	WWB (0.75-2.75) 11/5/96 None	WWB (0.75-2.75) 11/5/96 None	WWT-1 11/6/96 None	WWT-2 11/6/96 None
1,2-Dibromoethane (EDB)	-	10	290	3600	5800	18000000	NA	NA	NA	NA	NA	NA	NA
1,2-Dichlorobenzene	-	13000	210000	210000	46000000	44000000000	NA	NA	NA	NA	NA	NA	NA
1,2-Dichloroethane	-	100	270000	11000	21000	150000000	NA	NA	NA	NA	NA	NA	NA
1,2-Dichloropropane	-	100	360000	7400	30000	120000000	NA	NA	NA	NA	NA	NA	NA
1,3-Dichlorobenzene	-	18000	200000	-	-	-	NA	NA	NA	NA	NA	NA	NA
1,4-Dichlorobenzene	-	1700	1000000	100000	260000	570000000	NA	NA	NA	NA	NA	NA	NA
2-Butanone	-	760000	27000000	27000000	35000000	29000000000	NA	NA	NA	NA	NA	NA	NA
2-Chloroethyl vinyl ether	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
2-Hexanone	-	58000	2500000	1800000		1200000000	NA	NA	NA	NA	NA	NA	NA
4-Methyl-2-pentanone	-	100000	2700000	2700000	53000000	60000000000	NA	NA	NA	NA	NA	NA	NA
Acetone	-	42000	74000000	110000000	160000000	170000000000	NA	NA	NA	NA	NA	NA	NA
Benzene	-	100	400000	8400	45000	470000000	NA	NA	NA	NA	NA	NA	NA
Bromodichloromethane	-	2000	400000	6400	31000	110000000	NA	NA	NA	NA	NA	NA	NA
Bromoform	-	2000	870000	770000	3100000	3600000000	NA	NA	NA	NA	NA	NA	NA
Bromomethane	-	580	1000000	1600	13000	150000000	NA	NA	NA	NA	NA	NA	NA
Carbon disulfide	-	46000	280000	140000	1600000	21000000000	NA	NA	NA	NA	NA	NA	NA
Carbon tetrachloride	-	100	190000	990	12000	170000000	NA	NA	NA	NA	NA	NA	NA
Chlorobenzene	-	2000	260000	220000	920000	2100000000	NA	NA	NA	NA	NA	NA	NA
Chloroethane	-	18000	970000	970000	36000000	290000000000	NA	NA	NA	NA	NA	NA	NA
Chloroform	-	2000	1500000	38000	150000	1600000000	ND	ND	NA	NA	NA	ND	1.1
Chloromethane	-	5400	1100000	12000	140000	6100000000	NA	NA	NA	NA	NA	NA	NA
cis-1,2-Dichloroethene	-	1400	640000	640000	47000000	230000000000	NA	NA	NA	NA	NA	NA	NA
cis-1,3-Dichloropropene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Dibromochloromethane	-	2000	300000	21000	80000	160000000	NA	NA	NA	NA	NA	NA	NA
Dichlorodifluoromethane (CFC-12)	-	270000	1000000	1000000	63000000	1500000000000	NA	NA	NA	NA	NA	NA	NA
Ethylbenzene	-	1500	140000	140000	11000000	29000000000	ND	ND	NA	NA	NA	ND	ND
Ethylene oxide	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
m&p-Xylene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Methyl Tert Butyl Ether	-	800	6000000	6000000	31000000	88000000000	NA	NA	NA	NA	NA	NA	NA
Methylene chloride	-	100	2300000	240000	700000	8300000000	12.3	55	NA	NA	NA	ND	ND
o-Xylene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Styrene	-	2700	520000	520000	3200000	6600000000	NA	NA	NA	NA	NA	NA	NA
Tetrachloroethene	-	100	88000	60000	600000	6800000000	0.8	ND	NA	NA	NA	ND	3.5
Toluene	-	16000	250000	250000	3300000	12000000000	ND	ND	NA	NA	NA	ND	ND
trans-1,2-Dichloroethene	-	2000	1400000	1400000	37000000	230000000000	ND	ND	NA	NA	NA	ND	ND
trans-1,3-Dichloropropene	-	-	-	-	-	-	NA	NA	NA	NA	NA	NA	NA
Trichloroethene	-	100	500000	37000	260000	2300000000	0.8	ND	NA	NA	NA	ND	ND
Trichlorofluoromethane (CFC-11)	-	150000	560000	560000	110000000	1700000000000	NA	NA	NA	NA	NA	NA	NA
Vinyl chloride	-	40	11000	150	1500	47000000	NA	NA	NA	NA	NA	NA	NA
Xylene (total)	-	5600	150000	150000	54000000	130000000000	ND	ND	NA	NA	NA	ND	ND

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #			
							WWT-3 11/6/96	WWT-4 11/6/96	MW-4 (6-8) 11/6/96	PFW-9 (0-2) 3/19/97
							None	None	None	None
General Chemistry										
Cyanide (total) (ug/Kg)	-	4000	250000	-	-	250000	ND	ND	NA	NA
Total Solids (%)	-	-	-	-	-	-	NA	NA	NA	86.04
Diesel Fuel (mg/Kg)	-	-	-	-	-	-	NA	NA	NA	NA
Petroleum hydrocarbons (ug/Kg)	-	-	-	-	-	-	NA	NA	NA	NA
Metals (ug/Kg)										
Arsenic	5800	23000	100000	-	-	910000	6530	6950	NA	9000
Barium	75000	1300000	320000000	-	-	150000000	56000	44400	NA	57000
Cadmium	1200	6000	4500000	-	-	2200000	ND	ND	NA	590
Chromium	18000	30000	22000000	-	-	-	14500	13400	NA	16000
Copper	32000	160000000	170000000	-	-	59000000	ND	ND	NA	13000
Lead	21000	1000	900000	-	-	44000000	4100	ND	NA	16000
Mercury	130	1700	1400000	-	-	-	ND	ND	NA	ND (100)
Selenium	410	4000	23000000	-	-	59000000	ND	ND	NA	ND (500)
Silver	1000	13000	21000000	-	-	2900000	ND	ND	NA	ND (500)
Zinc	47000	5000000	1000000000	-	-	-	ND	ND	NA	80000
PCBs (ug/Kg)										
Aroclor-1016	-	-	9900	-	-	-	NA	NA	NA	NA
Aroclor-1221	-	-	9900	-	-	-	NA	NA	NA	NA
Aroclor-1232	-	-	9900	-	-	-	NA	NA	NA	NA
Aroclor-1242	-	-	9900	-	-	-	NA	NA	NA	NA
Aroclor-1248	-	-	9900	-	-	-	NA	NA	NA	NA
Aroclor-1254	-	-	9900	-	-	-	NA	NA	NA	NA
Aroclor-1260	-	-	9900	-	-	-	NA	NA	NA	NA
Total PCBs	-	-	9900	16000000	820000	6500000	NA	NA	NA	NA
Semi - Volatiles (ug/Kg)										
1,2,4-Trichlorobenzene	-	4200	1100000	1100000	34000000	11000000000	NA	NA	NA	NA
2,4,6-Trichlorophenol	-	45000	14000000	-	-	1300000000	NA	NA	NA	NA
2,4-Dichlorophenol	-	7700	10000000	-	-	2300000000	NA	NA	NA	NA
2,4-Dimethylphenol	-	20000	230000000	-	-	2100000000	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	-	-	-	NA	NA	NA	NA
2,4-Dinitrotoluene	-	15000	220000	-	-	20000000	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	-	-	-	NA	NA	NA	NA
2-Chloronaphthalene	-	1800000	190000000	-	-	-	NA	NA	NA	NA
2-Chlorophenol	-	2600	4600000	-	-	-	NA	NA	NA	NA
2-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA
2-Nitrophenol	-	1200	13000000	-	-	-	NA	NA	NA	NA
3,3-Dichlorobenzidine	-	2000	55000	-	-	8200000	NA	NA	NA	NA
3-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	-	1700	1600000	-	-	-	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA
4-Chloro-3-methylphenol	-	16000	15000000	-	-	-	NA	NA	NA	NA
4-Chloroaniline	-	-	-	-	-	-	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	-	-	-	NA	NA	NA	NA
4-Nitroaniline	-	-	-	-	-	-	NA	NA	NA	NA

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels [2]	Industrial Drinking Water Protection Criteria [2]	Industrial Direct Contact Criteria [2]	Soil Volatilization to Indoor Air Inhalation Criteria [2]	Volatile Soil Inhalation Criteria [2]	Particulate Soil Inhalation Criteria [2]	Sample Location (depth ft. bgs) Sample Date AOI # WWT-3 11/6/96 None	WWT-4 11/6/96 None	MW-4 (6-8) 11/6/96 None	PFW-9 (0-2) 3/19/97 None
4-Nitrophenol	-	-	-	-	-	-	NA	NA	NA	NA
Acenaphthene	-	870000	810000000	350000000	97000000	6200000000	NA	NA	NA	NA
Acenaphthylene	-	8500	16000000	3000000	2700000	1000000000	NA	NA	NA	NA
Acetonitrile	-	8000	14000000	22000000	11000000	10000000000	NA	NA	NA	NA
Aniline	-	12000	4500000	-	-	29000000	NA	NA	NA	NA
Anthracene	-	41000	1000000000	1000000000	1600000000	29000000000	NA	NA	NA	NA
Benzidine	-	1000	1000	-	-	59,000	NA	NA	NA	NA
Benzo(a)anthracene	-	-	210000	-	-	-	NA	NA	NA	NA
Benzo(a)pyrene	-	-	21000	-	-	1900000	NA	NA	NA	NA
Benzo(b)fluoranthene	-	-	210000	-	-	-	NA	NA	NA	NA
Benzo(g,h,i)perylene	-	-	16000000	-	-	350000000	NA	NA	NA	NA
Benzo(k)fluoranthene	-	-	2100000	-	-	-	NA	NA	NA	NA
bis(2-Chloroethoxy)methane	-	-	-	-	-	-	NA	NA	NA	NA
bis(2-Chloroethyl)ether	-	330	23000	44000	13000	12000000	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	-	-	-	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	-	-	10000000	-	-	8900000000	NA	NA	NA	NA
Butyl benzylphthalate	-	310000	310000	-	-	21000000000	NA	NA	NA	NA
Chrysene	-	-	21000000	-	-	-	NA	NA	NA	NA
Dibenzo(a,h)anthracene	-		21000	-	-	-	NA	NA	NA	NA
Dibenzofuran	-	-	-	-	-	-	NA	NA	NA	NA
Diethyl phthalate	-	320000	740000	-	-	1500000000	NA	NA	NA	NA
Dimethyl phthalate	-	790000	790000	-	-	1500000000	NA	NA	NA	NA
Di-n-butylphthalate	-	760000	760000	-	-	1500000000	NA	NA	NA	NA
Di-n-octyl phthalate	-	140000000	81000000	-	-	-	NA	NA	NA	NA
Fluoranthene	-	720000	540000000	1000000000	880000000	4100000000	NA	NA	NA	NA
Fluorene	-	890000	540000000	1000000000	150000000	4100000000	NA	NA	NA	NA
Hexachlorobenzene	-	1800	94000	220000	56000	85000000	NA	NA	NA	NA
Hexachlorobutadiene	-	77000	350000	350000	460000	180000000	NA	NA	NA	NA
Hexachlorocyclopentadiene	-	36000	81000	-	-	-	NA	NA	NA	NA
Hexachloroethane	-	69000	1800000	370000	1400000	100000000	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	-	-	210000	-	-	-	NA	NA	NA	NA
Isophorone	-	74000	2400000	-	-	8200000000	NA	NA	NA	NA
Naphthalene	-	50000	160000000	77000000	59000000	15000000000	NA	NA	NA	NA
Nitrobenzene	-	330	340000	490000	4600000	1500000000	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	-	330	3500	-	-	2000000	NA	NA	NA	NA
N-Nitrosodiphenylamine	-	14000	5100000	-	-	-	NA	NA	NA	NA
Octachlorocyclopentene	-	-	-	-	-	-	NA	NA	NA	NA
Pentachlorophenol	-	3200	63000	-	-	130000000	NA	NA	NA	NA
Phenanthrene	-	34000	16000000	28000000	150000	59000000	NA	NA	NA	NA
Phenol	-	260000	12000000	-	-	18000000000	NA	NA	NA	NA
Pyrene	-	470000	340000000	1000000000	770000000	2900000000	NA	NA	NA	NA
Volatiles (ug/Kg)										
1,1,1-Trichloroethane	-	4000	460000	460000	4500000	29000000000	ND	1.57	ND	ND (10)
1,1,2,2-Tetrachloroethane	-	340	120000	23000	34000	68000000	NA	NA	NA	ND (10)
1,1,2-Trichloroethane	-	100	440000	24000	57000	250000000	NA	NA	NA	ND (10)
1,1-Dichloroethane	-	50000	790000	790000	36000000	240000000000	ND	36	ND	ND (10)
1,1-Dichloroethene	-	140	580000	330	3700	78000000	ND	0.97	ND	ND (10)

TABLE H.1

SOIL ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Statewide Default Background Levels (2)	Industrial Drinking Water Protection Criteria (2)	Industrial Direct Contact Criteria (2)	Soil Volatilization to Indoor Air Inhalation Criteria (2)	Volatile Soil Inhalation Criteria (2)	Particulate Soil Inhalation Criteria (2)	Sample Location (depth ft. bgs) / Sample Date / AOI #			
							WWT-3 11/6/96 None	WWT-4 11/6/96 None	MW-4 (6-8) 11/6/96 None	PFW-9 (0-2) 3/19/97 None
1,2-Dibromoethane (EDB)	-	10	290	3600	5800	18000000	NA	NA	NA	NA
1,2-Dichlorobenzene	-	13000	210000	210000	46000000	44000000000	NA	NA	NA	ND (10)
1,2-Dichloroethane	-	100	270000	11000	21000	150000000	NA	NA	NA	ND (10)
1,2-Dichloropropane	-	100	360000	7400	30000	120000000	NA	NA	NA	ND (10)
1,3-Dichlorobenzene	-	18000	200000	-	-	-	NA	NA	NA	ND (10)
1,4-Dichlorobenzene	-	1700	1000000	100000	260000	570000000	NA	NA	NA	ND (10)
2-Butanone	-	760000	27000000	27000000	35000000	29000000000	NA	NA	NA	NA
2-Chloroethyl vinyl ether	-	-	-	-	-	-	NA	NA	NA	ND (100)
2-Hexanone	-	58000	2500000	1800000		1200000000	NA	NA	NA	NA
4-Methyl-2-pentanone	-	100000	2700000	2700000	53000000	60000000000	NA	NA	NA	NA
Acetone	-	42000	74000000	110000000	160000000	170000000000	NA	NA	NA	NA
Benzene	-	100	400000	8400	45000	470000000	NA	NA	NA	ND (10)
Bromodichloromethane	-	2000	400000	6400	31000	110000000	NA	NA	NA	ND (10)
Bromoform	-	2000	870000	770000	3100000	3600000000	NA	NA	NA	ND (10)
Bromomethane	-	580	1000000	1600	13000	150000000	NA	NA	NA	ND (10)
Carbon disulfide	-	46000	280000	140000	1600000	21000000000	NA	NA	NA	NA
Carbon tetrachloride	-	100	190000	990	12000	170000000	NA	NA	NA	ND (10)
Chlorobenzene	-	2000	260000	220000	920000	2100000000	NA	NA	NA	ND (10)
Chloroethane	-	18000	970000	970000	36000000	290000000000	NA	NA	NA	ND (10)
Chloroform	-	2000	1500000	38000	150000	1600000000	ND	ND	ND	ND (10)
Chloromethane	-	5400	1100000	12000	140000	6100000000	NA	NA	NA	ND (10)
cis-1,2-Dichloroethene	-	1400	640000	640000	47000000	230000000000	NA	NA	NA	ND (10)
cis-1,3-Dichloropropene	-	-	-	-	-	-	NA	NA	NA	ND (10)
Dibromochloromethane	-	2000	300000	21000	80000	160000000	NA	NA	NA	ND (10)
Dichlorodifluoromethane (CFC-12)	-	270000	1000000	1000000	63000000	1500000000000	NA	NA	NA	ND (10)
Ethylbenzene	-	1500	140000	140000	11000000	29000000000	ND	ND	ND	ND (10)
Ethylene oxide	-	-	-	-	-	-	NA	NA	NA	NA
m&p-Xylene	-	-	-	-	-	-	NA	NA	NA	ND (30)
Methyl Tert Butyl Ether	-	800	6000000	6000000	31000000	88000000000	NA	NA	NA	NA
Methylene chloride	-	100	2300000	240000	700000	8300000000	ND	ND	ND	ND (10)
o-Xylene	-	-	-	-	-	-	NA	NA	NA	ND (30)
Styrene	-	2700	520000	520000	3200000	6600000000	NA	NA	NA	NA
Tetrachloroethene	-	100	88000	60000	600000	6800000000	1.7	2.1	1.6	ND (10)
Toluene	-	16000	250000	250000	3300000	12000000000	ND	ND	ND	ND (10)
trans-1,2-Dichloroethene	-	2000	1400000	1400000	37000000	230000000000	ND	ND	ND	ND (10)
trans-1,3-Dichloropropene	-	-	-	-	-	-	NA	NA	NA	ND (10)
Trichloroethene	-	100	500000	37000	260000	2300000000	139	ND	ND	ND (10)
Trichlorofluoromethane (CFC-11)	-	150000	560000	560000	110000000	1700000000000	NA	NA	NA	ND (10)
Vinyl chloride	-	40	11000	150	1500	47000000	NA	NA	NA	ND (10)
Xylene (total)	-	5600	150000	150000	54000000	130000000000	ND	ND	ND	NA

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Groundwater Contact Criteria [2]	Industrial Drinking Water Criteria [2]	Industrial Groundwater Volatilization to Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #								
				MW-14 11/14/96 AOI # 1	MW-14 11/14/96 AOI # 1	MW-14 11/14/96 AOI # 1	PFW-6 3/31/97 AOI # 1	MW-13 11/14/96 AOI # 2	MW-13 11/14/96 AOI # 2	PFW-11 4/1/97 AOI # 3	PFW-10 3/31/97 AOI # 7	PFW-12 3/31/97 AOI # 9
General Chemistry (mg/L)												
Diesel Fuel	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Metals (ug/L)												
Arsenic (Dissolved)	4700	50	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Arsenic	4700	50	-	NA	NA	NA	ND (5)	NA	NA	ND (5)	ND (5)	ND (5)
Barium (Dissolved)	15000000	2000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Barium	15000000	2000	-	NA	NA	NA	1300	NA	NA	1100	2600	1200
Cadmium (Dissolved)	210000	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Cadmium	210000	5	-	NA	NA	NA	ND (0.5)	NA	NA	0.6	ND (0.5)	ND (0.5)
Chromium (Dissolved)	1000000	30000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Chromium	1000000	30000	-	NA	NA	NA	ND (50)	NA	NA	ND (50)	ND (50)	ND (50)
Copper (Dissolved)	8100000	1000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Copper	8100000	1000	-	NA	NA	NA	ND (25)	NA	NA	ND (25)	ND (25)	ND (25)
Lead (Dissolved)	-	4	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Lead	-	4	-	NA	NA	NA	ND (3)	NA	NA	ND (3)	4	ND (3)
Mercury (Dissolved)	56	2	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Mercury	56	2	-	NA	NA	NA	ND (0.2)	NA	NA	ND (0.2)	ND (0.2)	ND (0.2)
Selenium (Dissolved)	1100000	50	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Selenium	1100000	50	-	NA	NA	NA	ND (5)	NA	NA	ND (5)	ND (5)	ND (5)
Silver (Dissolved)	1000000	98	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Silver	1000000	98	-	NA	NA	NA	ND (0.5)	NA	NA	ND (0.5)	ND (0.5)	ND (0.5)
Zinc (Dissolved)	70000000	5000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Zinc	70000000	5000	-	NA	NA	NA	ND (20)	NA	NA	ND (20)	50	ND (20)
PCBs (ug/L)												
Aroclor-1016	-	-	-	NA	NA	NA	NA	ND (0.20)	NA	NA	NA	NA
Aroclor-1221	-	-	-	NA	NA	NA	NA	ND (0.20)	NA	NA	NA	NA
Aroclor-1232	-	-	-	NA	NA	NA	NA	ND (0.40)	NA	NA	NA	NA
Aroclor-1242	-	-	-	NA	NA	NA	NA	ND (0.20)	NA	NA	NA	NA
Aroclor-1248	-	-	-	NA	NA	NA	NA	ND (0.20)	NA	NA	NA	NA
Aroclor-1254	-	-	-	NA	NA	NA	NA	ND (0.20)	NA	NA	NA	NA
Aroclor-1260	-	-	-	NA	NA	NA	NA	ND (0.20)	NA	NA	NA	NA
Total PCBs	2.3	0.5	45	NA	NA	NA	NA	NA	ND	NA	NA	NA
Semi - Volatiles (ug/L)												
1,2,4-Trichlorobenzene	15000	70	300000	NA	NA	NA	NA	NA	NA	NA	NA	NA
2,4,5-Trichlorophenol	130000	2100	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2,4,6-Trichlorophenol	5500	320	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2,4-Dichlorophenol	40000	210	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2,4-Dimethylphenol	440000	1000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Groundwater Contact Criteria [2]	Industrial Drinking Water Criteria [2]	Industrial Groundwater Volatilization to Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #								
				MW-14 11/14/96 AOI # 1	MW-14 11/14/96 AOI # 1	MW-14 11/14/96 AOI # 1	PFW-6 3/31/97 AOI # 1	MW-13 11/14/96 AOI # 2	MW-13 11/14/96 AOI # 2	PFW-11 4/1/97 AOI # 3	PFW-10 3/31/97 AOI # 7	PFW-12 3/31/97 AOI # 9
2,4-Dinitrotoluene	1300	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2-Chloronaphthalene	6700	5200	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2-Chlorophenol	82000	130	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2-Methyl naphthalene	32000	750	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2-Methylphenol	710000	1000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2-Nitroaniline	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2-Nitrophenol	72000	58	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
3,3-Dichlorobenzidine	270	8	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
3-Nitroaniline	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	8800	20	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
4-Chloro-3-methylphenol	62000	420	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
4-Chloroaniline	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
4-Methylphenol	75000	100	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
4-Nitroaniline	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
4-Nitrophenol	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Acenaphthene	4200	3800	4200	NA	NA	NA	NA	NA	NA	NA	NA	NA
Acenaphthylene	3900	75	3900	NA	NA	NA	NA	NA	NA	NA	NA	NA
Anthracene	43	43	43	NA	NA	NA	NA	NA	NA	NA	NA	NA
Benzo(a)anthracene	5	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Benzo(a)pyrene	5	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Benzo(b)fluoranthene	5	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Benzo(g,h,i)perylene	5	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Benzo(k)fluoranthene	5	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroethoxy)methane	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroethyl)ether	2100	5	210000	NA	NA	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	47	6	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Butyl benzylphthalate	2700	2700	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Carbazole	2900	170	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Chrysene	5	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Dibenzo(a,h)anthracene	5	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Dibenzofuran	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Diethyl phthalate	1100000	16000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Dimethyl phthalate	4200000	210000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Di-n-butylphthalate	11000	2500	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Di-n-octyl phthalate	250	380	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Fluoranthene	210	210	210	NA	NA	NA	NA	NA	NA	NA	NA	NA
Fluorene	2000	2000	2000	NA	NA	NA	NA	NA	NA	NA	NA	NA

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	*Groundwater Contact Criteria* [2]	*Industrial Drinking Water Criteria* [2]	*Industrial Groundwater Volatilization to Inhalation Criteria* [2]	*Sample Location (depth ft. bgs)* / *Sample Date* / *AOI #*								
				MW-14 11/14/96 AOI # 1	*MW-14* 11/14/96 AOI # 1	*MW-14* 11/14/96 AOI # 1	*PFW-6* 3/31/97 AOI # 1	*MW-13* 11/14/96 AOI # 2	*MW-13* 11/14/96 AOI # 2	*PFW-11* 4/1/97 AOI # 3	*PFW-10* 3/31/97 AOI # 7	*PFW-12* 3/31/97 AOI # 9
Hexachlorobenzene	2	1	3000	NA	NA	NA	NA	NA	NA	NA	NA	NA
Hexachlorobutadiene	200	45	3200	NA	NA	NA	NA	NA	NA	NA	NA	NA
Hexachlorocyclopentadiene	1400	50	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Hexachloroethane	1500	250	50000	NA	NA	NA	NA	NA	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	5	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Isophorone	1100000	3700	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Naphthalene	31000	750	31000	NA	NA	NA	NA	NA	NA	NA	NA	NA
Nitrobenzene	9600	10	2100000	NA	NA	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	220	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodiphenylamine	30000	710	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Pentachlorophenol	85	1	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Phenanthrene	1000	75	1000	NA	NA	NA	NA	NA	NA	NA	NA	NA
Phenol	28000000	13000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Pyrene	140	140	140	NA	NA	NA	NA	NA	NA	NA	NA	NA
Volatiles (ug/L)												
1,1,1-Trichloroethane	220000	200	1300000	NA	ND (1)	ND	9	NA	0.7	ND (1)	ND (1)	ND (1)
1,1,2,2-Tetrachloroethane	2100	17	77000	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
1,1,2-Trichloroethane	9500	5	110000	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
1,1-Dichloroethane	2100000	2500	5100000	NA	ND (1)	ND	4	NA	ND	ND (1)	ND (1)	ND (1)
1,1-Dichloroethene	9000	7	1300	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
1,2-Dichlorobenzene	160000	600	160000	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
1,2-Dichloroethane	11000	5	59000	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
1,2-Dichloropropane	7500	5	36000	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
1,3-Dichlorobenzene	110000	600	-	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
1,4-Dichlorobenzene	2800	75	74000	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
2-Butanone	240000000	38000	240000000	ND (50)	NA	NA	NA	NA	NA	NA	NA	NA
2-Chloroethyl vinyl ether	-	-	-	NA	NA	NA	ND (10)	NA	NA	ND (10)	ND (10)	ND (10)
2-Hexanone	4800000	2900	8800000	ND (50)	NA	NA	NA	NA	NA	NA	NA	NA
4-Methyl-2-pentanone	12000000	5200	20000000	ND (50)	NA	NA	NA	NA	NA	NA	NA	NA
Acetone	31000000	2100	1000000000	ND (100)	NA	NA	NA	NA	NA	NA	NA	NA
Benzene	9400	5	36000	ND (5.0)	NA	NA	ND (5)	NA	NA	ND (5)	ND (5)	ND (5)
Bromodichloromethane	11000	100	38000	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
Bromoform	100000	100	3100000	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
Bromomethane	65000	29	9000	NA	ND (1)	ND	ND (1)	NA	6.7	ND (1)	ND (1)	ND (1)
Carbon disulfide	1100000	2300	550000	ND (50)	NA	NA	NA	NA	NA	NA	NA	NA
Carbon tetrachloride	1600	5	2400	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
Chlorobenzene	68000	100	470000	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
Chloroethane	200000	910	5700000	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
Chloroform	96000	100	180000	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
Chloromethane	110000	270	52000	NA	ND (1)	ND	ND (1)	NA	ND	ND (1)	ND (1)	ND (1)

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Groundwater Contact Criteria (2)	Industrial Drinking Water Criteria (2)	Industrial Groundwater Volatilization to Inhalation Criteria (2)	Sample Location (depth ft. bgs) / Sample Date / AOI #								
				MW-14 11/14/96 AOI # 1	MW-14 11/14/96 AOI # 1	MW-14 11/14/96 AOI # 1	PFW-6 3/31/97 AOI # 1	MW-13 11/14/96 AOI # 2	MW-13 11/14/96 AOI # 2	PFW-11 4/1/97 AOI # 3	PFW-10 3/31/97 AOI # 7	PFW-12 3/31/97 AOI # 9
cis-1,2-Dichloroethene	170000	70	3500000	4	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
cis-1,3-Dichloropropene	-	-	-	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
Dibromochloromethane	9500	100	110000	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
Dichlorodifluoromethane (CFC-12)	300000	4800	300000	NA	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
Ethylbenzene	170000	74	170000	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
m&p-Xylene	-	-	-	NA	NA	NA	ND (3)	NA	NA	ND (3)	ND (3)	ND (3)
Methylene chloride	110000	5	1400000	ND (5.0)	NA	NA	ND (5)	NA	NA	ND (5)	ND (5)	ND (5)
o-Xylene	-	-	-	NA	NA	NA	ND (3)	NA	NA	ND (3)	ND (3)	ND (3)
Styrene	3200	100	310000	ND (1.0)	NA	NA	NA	NA	NA	NA	NA	NA
Tetrachloroethene	5100	5	170000	NA	ND (1)	1.1	ND (1)	NA	ND	ND (1)	ND (1)	ND (1)
Toluene	530000	790	530000	NA	ND (1)	1.3	ND (1)	NA	1.1	ND (1)	ND (1)	ND (1)
trans-1,2-Dichloroethene	190000	100	6300000	NA	ND (1)	ND	ND (1)	NA	ND	ND (1)	ND (1)	ND (1)
trans-1,3-Dichloropropene	-	-	-	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
Trichloroethene	11000	5	97000	NA	6	21.3	ND (1)	NA	8.4	ND (1)	ND (1)	ND (1)
Trichlorofluoromethane (CFC-11)	1100000	7300	1100000	NA	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
Vinyl chloride	290	2	690	ND (1.0)	NA	NA	ND (1)	NA	NA	ND (1)	ND (1)	ND (1)
Xylene (total)	190000	280	190000	ND (3.0)	NA	NA	NA	NA	NA	NA	NA	NA

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Groundwater Contact Criteria [2]	Industrial Drinking Water Criteria [2]	Industrial Groundwater Volatilization to Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #								
				MW-1 11/14/96 AOI # 14	MW-1 11/14/96 AOI # 14	MW-1 11/14/96 AOI # 14	MW-2 11/14/96 AOI # 14	MW-2 11/14/96 AOI # 14	MW-2 11/14/96 AOI # 14	PFW-2 4/1/97 AOI # 14	PFW-2D 4/1/97 AOI # 14	PFW-5 3/31/97 AOI # 15
General Chemistry (mg/L)												
Diesel Fuel	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Metals (ug/L)												
Arsenic (Dissolved)	4700	50	-	NA	NA	NA	NA	ND (5)	NA	NA	NA	NA
Arsenic	4700	50	-	NA	NA	ND	NA	NA	ND	ND (5)	ND (5)	ND (5)
Barium (Dissolved)	15000000	2000	-	NA	NA	NA	NA	ND (200)	NA	NA	NA	NA
Barium	15000000	2000	-	NA	NA	ND	NA	NA	540	610	610	450
Cadmium (Dissolved)	210000	5	-	NA	NA	NA	ND (0.50)	NA	NA	NA	NA	NA
Cadmium	210000	5	-	NA	NA	NA	NA	NA	NA	ND (0.5)	ND (0.5)	1.4
Chromium (Dissolved)	1000000	30000	-	NA	NA	NA	ND (50)	NA	NA	NA	NA	NA
Chromium	1000000	30000	-	NA	NA	NA	NA	NA	NA	ND (50)	ND (50)	ND (50)
Copper (Dissolved)	8100000	1000	-	NA	NA	NA	NA	ND (25)	NA	NA	NA	NA
Copper	8100000	1000	-	NA	NA	ND	NA	NA	ND	ND (25)	ND (25)	ND (25)
Lead (Dissolved)	-	4	-	NA	NA	NA	NA	ND (3)	NA	NA	NA	NA
Lead	-	4	-	NA	NA	ND	NA	NA	ND	ND (3)	ND (3)	ND (3)
Mercury (Dissolved)	56	2	-	NA	NA	NA	ND (0.20)	NA	NA	NA	NA	NA
Mercury	56	2	-	NA	NA	NA	NA	NA	NA	ND (0.2)	ND (0.2)	ND (0.2)
Selenium (Dissolved)	1100000	50	-	NA	NA	NA	9.5	NA	NA	NA	NA	NA
Selenium	1100000	50	-	NA	NA	ND	NA	NA	ND	ND (5)	ND (5)	ND (5)
Silver (Dissolved)	1000000	98	-	NA	NA	NA	ND (0.50)	NA	NA	NA	NA	NA
Silver	1000000	98	-	NA	NA	NA	NA	NA	NA	ND (0.5)	ND (0.5)	ND (0.5)
Zinc (Dissolved)	70000000	5000	-	NA	NA	NA	NA	ND (20)	NA	NA	NA	NA
Zinc	70000000	5000	-	NA	NA	ND	NA	NA	ND	ND (20)	ND (20)	ND (20)
PCBs (ug/L)												
Aroclor-1016	-	-	-	ND (0.20)	NA	NA	NA	NA	NA	NA	NA	NA
Aroclor-1221	-	-	-	ND (0.20)	NA	NA	NA	NA	NA	NA	NA	NA
Aroclor-1232	-	-	-	ND (0.40)	NA	NA	NA	NA	NA	NA	NA	NA
Aroclor-1242	-	-	-	ND (0.20)	NA	NA	NA	NA	NA	NA	NA	NA
Aroclor-1248	-	-	-	ND (0.20)	NA	NA	NA	NA	NA	NA	NA	NA
Aroclor-1254	-	-	-	ND (0.20)	NA	NA	NA	NA	NA	NA	NA	NA
Aroclor-1260	-	-	-	ND (0.20)	NA	NA	NA	NA	NA	NA	NA	NA
Total PCBs	2.3	0.5	45	NA	ND (.2)	NA	NA	NA	NA	NA	NA	NA
Semi - Volatiles (ug/L)												
1,2,4-Trichlorobenzene	15000	70	300000	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
2,4,5-Trichlorophenol	130000	2100	-	NA	NA	NA	ND (50)	NA	NA	NA	NA	NA
2,4,6-Trichlorophenol	5500	320	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
2,4-Dichlorophenol	40000	210	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
2,4-Dimethylphenol	440000	1000	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	NA	NA	NA	ND (20)	NA	NA	NA	NA	NA

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Groundwater Contact Criteria [2]	Industrial Drinking Water Criteria [2]	Industrial Groundwater Volatilization to Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #								
				MW-1 11/14/96 AOI # 14	MW-1 11/14/96 AOI # 14	MW-1 11/14/96 AOI # 14	MW-2 11/14/96 AOI # 14	MW-2 11/14/96 AOI # 14	MW-2 11/14/96 AOI # 14	PFW-2 4/1/97 AOI # 14	PFW-2D 4/1/97 AOI # 14	PFW-5 3/31/97 AOI # 15
2,4-Dinitrotoluene	1300	5	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
2-Chloronaphthalene	6700	5200	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
2-Chlorophenol	82000	130	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
2-Methyl naphthalene	32000	750	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
2-Methylphenol	710000	1000	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
2-Nitroaniline	-	-	-	NA	NA	NA	ND (20)	NA	NA	NA	NA	NA
2-Nitrophenol	72000	58	-	NA	NA	NA	NA	ND (5)	1.8	NA	NA	NA
3,3-Dichlorobenzidine	270	8	-	NA	NA	NA	ND (20)	NA	NA	NA	NA	NA
3-Nitroaniline	-	-	-	NA	NA	NA	ND (20)	NA	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	8800	20	-	NA	NA	NA	ND (20)	NA	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
4-Chloro-3-methylphenol	62000	420	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
4-Chloroaniline	-	-	-	NA	NA	NA	ND (20)	NA	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
4-Methylphenol	75000	100	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
4-Nitroaniline	-	-	-	NA	NA	NA	ND (20)	NA	NA	NA	NA	NA
4-Nitrophenol	-	-	-	NA	NA	NA	ND (20)	NA	NA	NA	NA	NA
Acenaphthene	4200	3800	4200	NA	NA	NA	NA	ND (5)	0.16	ND (5)	NA	NA
Acenaphthylene	3900	75	3900	NA	NA	NA	ND (5.0)	NA	NA	ND (5)	NA	NA
Anthracene	43	43	43	NA	NA	NA	ND (5.0)	NA	NA	ND (5)	NA	NA
Benzo(a)anthracene	5	5	-	NA	NA	NA	ND (5.0)	NA	NA	ND (5)	NA	NA
Benzo(a)pyrene	5	5	-	NA	NA	NA	ND (5.0)	NA	NA	ND (5)	NA	NA
Benzo(b)fluoranthene	5	5	-	NA	NA	NA	ND (5.0)	NA	NA	ND (5)	NA	NA
Benzo(g,h,i)perylene	5	5	-	NA	NA	NA	ND (5.0)	NA	NA	ND (5)	NA	NA
Benzo(k)fluoranthene	5	5	-	NA	NA	NA	ND (5.0)	NA	NA	ND (5)	NA	NA
bis(2-Chloroethoxy)methane	-	-	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
bis(2-Chloroethyl)ether	2100	5	210000	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	47	6	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
Butyl benzylphthalate	2700	2700	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
Carbazole	2900	170	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
Chrysene	5	5	-	NA	NA	NA	ND (5.0)	NA	NA	ND (5)	NA	NA
Dibenzo(a,h)anthracene	5	5	-	NA	NA	NA	ND (5.0)	NA	NA	ND (5)	NA	NA
Dibenzofuran	-	-	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
Diethyl phthalate	1100000	16000	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
Dimethyl phthalate	4200000	210000	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
Di-n-butylphthalate	11000	2500	-	NA	NA	NA	NA	ND (5)	0.7	NA	NA	NA
Di-n-octyl phthalate	250	380	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
Fluoranthene	210	210	210	NA	NA	NA	ND (5.0)	NA	NA	ND (5)	NA	NA
Fluorene	2000	2000	2000	NA	NA	NA	NA	ND (5)	0.14	ND (5)	NA	NA

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Groundwater Contact Criteria [2]	Industrial Drinking Water Criteria [2]	Industrial Groundwater Volatilization to Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #								
				MW-1 11/14/96 AOI # 14	MW-1 11/14/96 AOI # 14	MW-1 11/14/96 AOI # 14	MW-2 11/14/96 AOI # 14	MW-2 11/14/96 AOI # 14	MW-2 11/14/96 AOI # 14	PFW-2 4/1/97 AOI # 14	PFW-2D 4/1/97 AOI # 14	PFW-5 3/31/97 AOI # 15
Hexachlorobenzene	2	1	3000	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
Hexachlorobutadiene	200	45	3200	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
Hexachlorocyclopentadiene	1400	50	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
Hexachloroethane	1500	250	50000	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	5	5	-	NA	NA	NA	ND (5.0)	NA	NA	ND (5)	NA	NA
Isophorone	1100000	3700	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
Naphthalene	31000	750	31000	NA	NA	NA	NA	ND (5)	0.17	ND (5)	NA	NA
Nitrobenzene	9600	10	2100000	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	220	5	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
N-Nitrosodiphenylamine	30000	710	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
Pentachlorophenol	85	1	-	NA	NA	NA	ND (20)	NA	NA	NA	NA	NA
Phenanthrene	1000	75	1000	NA	NA	NA	NA	ND (5)	0.6	ND (5)	NA	NA
Phenol	28000000	13000	-	NA	NA	NA	ND (5.0)	NA	NA	NA	NA	NA
Pyrene	140	140	140	NA	NA	NA	ND (5.0)	NA	NA	ND (5)	NA	NA
Volatiles (ug/L)												
1,1,1-Trichloroethane	220000	200	1300000	NA	ND (1)	ND	NA	ND (1)	ND	ND (1)	ND (1)	ND (1)
1,1,2,2-Tetrachloroethane	2100	17	77000	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
1,1,2-Trichloroethane	9500	5	110000	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
1,1-Dichloroethane	2100000	2500	5100000	NA	ND (1)	1	NA	ND (1)	ND	ND (1)	ND (1)	ND (1)
1,1-Dichloroethene	9000	7	1300	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
1,2-Dichlorobenzene	160000	600	160000	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
1,2-Dichloroethane	11000	5	59000	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
1,2-Dichloropropane	7500	5	36000	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
1,3-Dichlorobenzene	110000	600	-	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
1,4-Dichlorobenzene	2800	75	74000	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
2-Butanone	240000000	38000	240000000	ND (50)	NA	NA	ND (50)	NA	NA	NA	NA	NA
2-Chloroethyl vinyl ether	-	-	-	NA	NA	NA	NA	NA	NA	ND (10)	ND (10)	ND (10)
2-Hexanone	4800000	2900	8800000	ND (50)	NA	NA	ND (50)	NA	NA	NA	NA	NA
4-Methyl-2-pentanone	12000000	5200	20000000	ND (50)	NA	NA	ND (50)	NA	NA	NA	NA	NA
Acetone	31000000	2100	1000000000	ND (100)	NA	NA	ND (100)	NA	NA	NA	NA	NA
Benzene	9400	5	36000	ND (5.0)	NA	NA	ND (5.0)	NA	NA	ND (5)	ND (5)	ND (5)
Bromodichloromethane	11000	100	38000	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
Bromoform	100000	100	3100000	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
Bromomethane	65000	29	9000	NA	ND (1)	ND	NA	ND (1)	ND	ND (1)	ND (1)	ND (1)
Carbon disulfide	1100000	2300	550000	ND (50)	NA	NA	ND (50)	NA	NA	NA	NA	NA
Carbon tetrachloride	1600	5	2400	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
Chlorobenzene	68000	100	470000	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
Chloroethane	200000	910	5700000	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
Chloroform	96000	100	180000	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
Chloromethane	110000	270	52000	NA	ND (1)	ND	NA	ND (1)	ND	ND (1)	ND (1)	ND (1)

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Groundwater Contact Criteria (2)	Industrial Drinking Water Criteria (2)	Industrial Groundwater Volatilization to Inhalation Criteria (2)	Sample Location (depth ft. bgs) / Sample Date / AOI #								
				MW-1 11/14/96 AOI # 14	MW-1 11/14/96 AOI # 14	MW-1 11/14/96 AOI # 14	MW-2 11/14/96 AOI # 14	MW-2 11/14/96 AOI # 14	MW-2 11/14/96 AOI # 14	PFW-2 4/1/97 AOI # 14	PFW-2D 4/1/97 AOI # 14	PFW-5 3/31/97 AOI # 15
cis-1,2-Dichloroethene	170000	70	3500000	5	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
cis-1,3-Dichloropropene	-	-	-	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
Dibromochloromethane	9500	100	110000	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
Dichlorodifluoromethane (CFC-12)	300000	4800	300000	NA	NA	NA	NA	NA	NA	ND (1)	ND (1)	ND (1)
Ethylbenzene	170000	74	170000	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
m&p-Xylene	-	-	-	NA	NA	NA	NA	NA	NA	ND (3)	ND (3)	ND (3)
Methylene chloride	110000	5	1400000	ND (5.0)	NA	NA	ND (5.0)	NA	NA	ND (5)	ND (5)	ND (5)
o-Xylene	-	-	-	NA	NA	NA	NA	NA	NA	ND (3)	ND (3)	ND (3)
Styrene	3200	100	310000	ND (1.0)	NA	NA	ND (1.0)	NA	NA	NA	NA	NA
Tetrachloroethene	5100	5	170000	NA	ND (1)	ND	NA	ND (1)	0.5	ND (1)	ND (1)	ND (1)
Toluene	530000	790	530000	NA	ND (1)	0.6	NA	ND (1)	0.7	ND (1)	ND (1)	ND (1)
trans-1,2-Dichloroethene	190000	100	6300000	NA	ND (1)	1.7	NA	ND (1)	ND	ND (1)	ND (1)	ND (1)
trans-1,3-Dichloropropene	-	-	-	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
Trichloroethene	11000	5	97000	NA	ND (1)	4.8	NA	ND (1)	4.4	ND (1)	ND (1)	ND (1)
Trichlorofluoromethane (CFC-11)	1100000	7300	1100000	NA	NA	NA	NA	NA	NA	ND (1)	ND (1)	ND (1)
Vinyl chloride	290	2	690	ND (1.0)	NA	NA	ND (1.0)	NA	NA	ND (1)	ND (1)	ND (1)
Xylene (total)	190000	280	190000	ND (3.0)	NA	NA	ND (3.0)	NA	NA	NA	NA	NA

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Groundwater Contact Criteria [2]	Industrial Drinking Water Criteria [2]	Industrial Groundwater Volatilization to Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #								
				PFW-9 4/1/97 AOI # 16	MW-3 11/14/96 AOI # 18	MW-3 11/14/96 AOI # 18	MW-3 11/14/96 AOI # 18	PFW-4 4/1/97 AOI # 18	PFB-21 GW 4/25/97 AOI # 21	MW-10 11/13/96 None	MW-10 4/1/97 None	MW-6 11/13/96 None
General Chemistry (mg/L)												
Diesel Fuel	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Metals (ug/L)												
Arsenic (Dissolved)	4700	50	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Arsenic	4700	50	-	ND (5)	NA	13	ND	ND (5)	NA	ND	ND (5)	NA
Barium (Dissolved)	15000000	2000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Barium	15000000	2000	-	970	NA	ND (200)	270	1600	NA	260	1600	NA
Cadmium (Dissolved)	210000	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Cadmium	210000	5	-	ND (0.5)	ND (0.50)	NA	NA	ND (0.5)	NA	NA	ND (0.5)	NA
Chromium (Dissolved)	1000000	30000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Chromium	1000000	30000	-	ND (50)	ND (50)	NA	NA	ND (50)	NA	NA	ND (50)	NA
Copper (Dissolved)	8100000	1000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Copper	8100000	1000	-	ND (25)	32	NA	ND	ND (25)	NA	ND	ND (25)	NA
Lead (Dissolved)	-	4	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Lead	-	4	-	9	12	NA	ND	ND (3)	NA	14	ND (3)	NA
Mercury (Dissolved)	56	2	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Mercury	56	2	-	ND (0.2)	ND (0.20)	NA	NA	ND (0.2)	NA	NA	ND (0.2)	NA
Selenium (Dissolved)	1100000	50	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Selenium	1100000	50	-	ND (5)	NA	10	ND	ND (5)	NA	ND	ND (5)	NA
Silver (Dissolved)	1000000	98	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Silver	1000000	98	-	ND (0.5)	ND (0.50)	NA	NA	ND (0.5)	NA	NA	ND (0.5)	NA
Zinc (Dissolved)	70000000	5000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Zinc	70000000	5000	-	ND (20)	NA	110	ND	20	NA	ND	ND (20)	NA
PCBs (ug/L)												
Aroclor-1016	-	-	-	NA	ND (0.20)	NA	NA	NA	NA	NA	NA	ND (0.20)
Aroclor-1221	-	-	-	NA	ND (0.20)	NA	NA	NA	NA	NA	NA	ND (0.20)
Aroclor-1232	-	-	-	NA	ND (0.40)	NA	NA	NA	NA	NA	NA	ND (0.40)
Aroclor-1242	-	-	-	NA	ND (0.20)	NA	NA	NA	NA	NA	NA	ND (0.20)
Aroclor-1248	-	-	-	NA	ND (0.20)	NA	NA	NA	NA	NA	NA	ND (0.20)
Aroclor-1254	-	-	-	NA	ND (0.20)	NA	NA	NA	NA	NA	NA	ND (0.20)
Aroclor-1260	-	-	-	NA	ND (0.20)	NA	NA	NA	NA	NA	NA	ND (0.20)
Total PCBs	2.3	0.5	45	NA	NA	ND (.2)	ND	NA	NA	ND	NA	NA
Semi - Volatiles (ug/L)												
1,2,4-Trichlorobenzene	15000	70	300000	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
2,4,5-Trichlorophenol	130000	2100	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (50)
2,4,6-Trichlorophenol	5500	320	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
2,4-Dichlorophenol	40000	210	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
2,4-Dimethylphenol	440000	1000	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
2,4-Dinitrophenol	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (20)

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Groundwater Contact Criteria [2]	Industrial Drinking Water Criteria [2]	Industrial Groundwater Volatilization to Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #								
				PFW-9 4/1/97 AOI # 16	MW-3 11/14/96 AOI # 18	MW-3 11/14/96 AOI # 18	MW-3 11/14/96 AOI # 18	PFW-4 4/1/97 AOI # 18	PFB-21 GW 4/25/97 AOI # 21	MW-10 11/13/96 None	MW-10 4/1/97 None	MW-6 11/13/96 None
2,4-Dinitrotoluene	1300	5	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
2,6-Dinitrotoluene	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
2-Chloronaphthalene	6700	5200	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
2-Chlorophenol	82000	130	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
2-Methyl naphthalene	32000	750	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
2-Methylphenol	710000	1000	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
2-Nitroaniline	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (20)
2-Nitrophenol	72000	58	-	NA	NA	NA	NA	NA	NA	ND	NA	NA
3,3-Dichlorobenzidine	270	8	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (20)
3-Nitroaniline	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (20)
4,6-Dinitro-2-methylphenol	8800	20	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (20)
4-Bromophenyl phenyl ether	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
4-Chloro-3-methylphenol	62000	420	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
4-Chloroaniline	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (20)
4-Chlorophenyl phenyl ether	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
4-Methylphenol	75000	100	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
4-Nitroaniline	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (20)
4-Nitrophenol	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (20)
Acenaphthene	4200	3800	4200	NA	NA	NA	NA	NA	ND (5)	ND	NA	NA
Acenaphthylene	3900	75	3900	NA	NA	NA	NA	NA	ND (5)	NA	NA	ND (5.0)
Anthracene	43	43	43	NA	NA	NA	NA	NA	ND (5)	NA	NA	ND (5.0)
Benzo(a)anthracene	5	5	-	NA	NA	NA	NA	NA	ND (5)	NA	NA	ND (5.0)
Benzo(a)pyrene	5	5	-	NA	NA	NA	NA	NA	ND (5)	NA	NA	ND (5.0)
Benzo(b)fluoranthene	5	5	-	NA	NA	NA	NA	NA	ND (5)	NA	NA	ND (5.0)
Benzo(g,h,i)perylene	5	5	-	NA	NA	NA	NA	NA	ND (5)	NA	NA	ND (5.0)
Benzo(k)fluoranthene	5	5	-	NA	NA	NA	NA	NA	ND (5)	NA	NA	ND (5.0)
bis(2-Chloroethoxy)methane	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
bis(2-Chloroethyl)ether	2100	5	210000	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
bis(2-Chloroisopropyl)ether	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
bis(2-Ethylhexyl)phthalate	47	6	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
Butyl benzylphthalate	2700	2700	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
Carbazole	2900	170	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
Chrysene	5	5	-	NA	NA	NA	NA	NA	ND (5)	NA	NA	ND (5.0)
Dibenzo(a,h)anthracene	5	5	-	NA	NA	NA	NA	NA	ND (5)	NA	NA	ND (5.0)
Dibenzofuran	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
Diethyl phthalate	1100000	16000	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
Dimethyl phthalate	4200000	210000	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
Di-n-butylphthalate	11000	2500	-	NA	NA	NA	NA	NA	NA	ND	NA	NA
Di-n-octyl phthalate	250	380	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
Fluoranthene	210	210	210	NA	NA	NA	NA	NA	ND (5)	NA	NA	ND (5.0)
Fluorene	2000	2000	2000	NA	NA	NA	NA	NA	ND (5)	ND	NA	NA

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Groundwater Contact Criteria [2]	Industrial Drinking Water Criteria [2]	Industrial Groundwater Volatilization to Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #								
				PFW-9 4/1/97 AOI # 16	MW-3 11/14/96 AOI # 18	MW-3 11/14/96 AOI # 18	MW-3 11/14/96 AOI # 18	PFW-4 4/1/97 AOI # 18	PFB-21 GW 4/25/97 AOI # 21	MW-10 11/13/96 None	MW-10 4/1/97 None	MW-6 11/13/96 None
Hexachlorobenzene	2	1	3000	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
Hexachlorobutadiene	200	45	3200	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
Hexachlorocyclopentadiene	1400	50	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
Hexachloroethane	1500	250	50000	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
Indeno(1,2,3-cd)pyrene	5	5	-	NA	NA	NA	NA	NA	ND (5)	NA	NA	ND (5.0)
Isophorone	1100000	3700	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
Naphthalene	31000	750	31000	NA	NA	NA	NA	NA	ND (5)	ND	NA	NA
Nitrobenzene	9600	10	2100000	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
N-Nitrosodi-n-propylamine	220	5	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
N-Nitrosodiphenylamine	30000	710	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
Pentachlorophenol	85	1	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (20)
Phenanthrene	1000	75	1000	NA	NA	NA	NA	NA	ND (5)	ND	NA	NA
Phenol	28000000	13000	-	NA	NA	NA	NA	NA	NA	NA	NA	ND (5.0)
Pyrene	140	140	140	NA	NA	NA	NA	NA	ND (5)	NA	NA	ND (5.0)
Volatiles (ug/L)												
1,1,1-Trichloroethane	220000	200	1300000	ND (1)	NA	ND (1)	ND	ND (1)	ND (1)	ND	ND (1)	NA
1,1,2,2-Tetrachloroethane	2100	17	77000	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
1,1,2-Trichloroethane	9500	5	110000	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
1,1-Dichloroethane	2100000	2500	5100000	ND (1)	NA	ND (1)	ND	ND (1)	ND (1)	ND	ND (1)	NA
1,1-Dichloroethene	9000	7	1300	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
1,2-Dichlorobenzene	160000	600	160000	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
1,2-Dichloroethane	11000	5	59000	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
1,2-Dichloropropane	7500	5	36000	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
1,3-Dichlorobenzene	110000	600	-	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
1,4-Dichlorobenzene	2800	75	74000	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
2-Butanone	240000000	38000	240000000	NA	ND (50)	NA	NA	NA	NA	NA	NA	NA
2-Chloroethyl vinyl ether	-	-	-	ND (10)	NA	NA	NA	ND (10)	ND (10)	NA	ND (10)	NA
2-Hexanone	4800000	2900	8800000	NA	ND (50)	NA	NA	NA	NA	NA	NA	NA
4-Methyl-2-pentanone	12000000	5200	20000000	NA	ND (50)	NA	NA	NA	NA	NA	NA	NA
Acetone	31000000	2100	1000000000	NA	ND (100)	NA	NA	NA	NA	NA	NA	NA
Benzene	9400	5	36000	ND (5)	ND (5.0)	NA	NA	ND (5)	ND (5)	NA	ND (5)	NA
Bromodichloromethane	11000	100	38000	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
Bromoform	100000	100	3100000	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
Bromomethane	65000	29	9000	ND (1)	NA	ND (1)	8.8	ND (1)	ND (1)	ND	ND (1)	NA
Carbon disulfide	1100000	2300	550000	NA	ND (50)	NA	NA	NA	NA	NA	NA	NA
Carbon tetrachloride	1600	5	2400	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
Chlorobenzene	68000	100	470000	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
Chloroethane	200000	910	5700000	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
Chloroform	96000	100	180000	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
Chloromethane	110000	270	52000	ND (1)	NA	ND (1)	ND	ND (1)	ND (1)	ND	ND (1)	NA

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Groundwater Contact Criteria (2)	Industrial Drinking Water Criteria (2)	Industrial Groundwater Volatilization to Inhalation Criteria (2)	PFW-9 4/1/97 AOI # 16	MW-3 11/14/96 AOI # 18	MW-3 11/14/96 AOI # 18	MW-3 11/14/96 AOI # 18	PFW-4 4/1/97 AOI # 18	PFB-21 GW 4/25/97 AOI # 21	MW-10 11/13/96 None	MW-10 4/1/97 None	MW-6 11/13/96 None
				Sample Location (depth ft. bgs)								
				Sample Date								
				AOI #								
cis-1,2-Dichloroethene	170000	70	3500000	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
cis-1,3-Dichloropropene	-	-	-	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
Dibromochloromethane	9500	100	110000	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
Dichlorodifluoromethane (CFC-12)	300000	4800	300000	ND (1)	NA	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
Ethylbenzene	170000	74	170000	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
m&p-Xylene	-	-	-	ND (3)	NA	NA	NA	ND (3)	ND (3)	NA	ND (3)	NA
Methylene chloride	110000	5	1400000	ND (5)	ND (5.0)	NA	NA	ND (5)	ND (5)	NA	ND (5)	NA
o-Xylene	-	-	-	ND (3)	NA	NA	NA	ND (3)	ND (3)	NA	ND (3)	NA
Styrene	3200	100	310000	NA	ND (1.0)	NA	NA	NA	NA	NA	NA	NA
Tetrachloroethene	5100	5	170000	ND (1)	NA	ND (1)	ND	ND (1)	ND (1)	0.6	ND (1)	NA
Toluene	530000	790	530000	ND (1)	NA	ND (1)	0.5	ND (1)	ND (1)	0.7	ND (1)	NA
trans-1,2-Dichloroethene	190000	100	6300000	ND (1)	NA	ND (1)	ND	ND (1)	ND (1)	ND	ND (1)	NA
trans-1,3-Dichloropropene	-	-	-	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
Trichloroethene	11000	5	97000	ND (1)	NA	ND (1)	3.8	ND (1)	ND (1)	4.6	ND (1)	NA
Trichlorofluoromethane (CFC-11)	1100000	7300	1100000	ND (1)	NA	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
Vinyl chloride	290	2	690	ND (1)	ND (1.0)	NA	NA	ND (1)	ND (1)	NA	ND (1)	NA
Xylene (total)	190000	280	190000	NA	ND (3.0)	NA	NA	NA	NA	NA	NA	NA

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Groundwater Contact Criteria [2]	Industrial Drinking Water Criteria [2]	Industrial Groundwater Volatilization to Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #								
				MW-6 11/13/96 None	MW-6 11/13/96 None	MW-6 11/13/96 None	MW-8 11/13/96 None	GP-7 8/30/96 None	MW-11 11/13/96 None	MW-12 11/13/96 None	PFW-1 4/1/97 None	MW-4 11/14/96 None
General Chemistry (mg/L)												
Diesel Fuel	-	-	-	NA	NA	NA	NA	ND (0.20)	NA	NA	NA	NA
Metals (ug/L)												
Arsenic (Dissolved)	4700	50	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Arsenic	4700	50	-	NA	NA	ND	NA	ND (5.0)	ND	ND	40	NA
Barium (Dissolved)	15000000	2000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Barium	15000000	2000	-	NA	NA	340	NA	710	260	270	550	240
Cadmium (Dissolved)	210000	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Cadmium	210000	5	-	NA	NA	NA	NA	2.2	NA	NA	ND (0.5)	ND (0.50)
Chromium (Dissolved)	1000000	30000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Chromium	1000000	30000	-	NA	NA	NA	NA	ND (50)	NA	NA	ND (50)	ND (50)
Copper (Dissolved)	8100000	1000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Copper	8100000	1000	-	NA	NA	ND	NA	32	ND	ND	ND (25)	27
Lead (Dissolved)	-	4	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Lead	-	4	-	NA	NA	ND	NA	ND (40)	22	ND	ND (3)	NA
Mercury (Dissolved)	56	2	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Mercury	56	2	-	NA	NA	NA	NA	ND (0.20)	NA	NA	ND (0.2)	ND (0.20)
Selenium (Dissolved)	1100000	50	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Selenium	1100000	50	-	NA	NA	ND	NA	ND (5.0)	ND	ND	ND (5)	NA
Silver (Dissolved)	1000000	98	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Silver	1000000	98	-	NA	NA	NA	NA	ND (0.50)	NA	NA	ND (0.5)	ND (0.50)
Zinc (Dissolved)	70000000	5000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Zinc	70000000	5000	-	NA	NA	ND	NA	91	ND	ND	ND (20)	20
PCBs (ug/L)												
Aroclor-1016	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Aroclor-1221	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Aroclor-1232	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Aroclor-1242	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Aroclor-1248	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Aroclor-1254	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Aroclor-1260	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Total PCBs	2.3	0.5	45	ND (.2)	NA	ND	NA	NA	ND	NA	NA	NA
Semi - Volatiles (ug/L)												
1,2,4-Trichlorobenzene	15000	70	300000	NA	NA	NA	NA	NA	NA	NA	NA	NA
2,4,5-Trichlorophenol	130000	2100	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2,4,6-Trichlorophenol	5500	320	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2,4-Dichlorophenol	40000	210	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2,4-Dimethylphenol	440000	1000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Groundwater Contact Criteria [2]	Industrial Drinking Water Criteria [2]	Industrial Groundwater Volatilization to Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #								
				MW-6 11/13/96 None	MW-6 11/13/96 None	MW-6 11/13/96 None	MW-8 11/13/96 None	GP-7 8/30/96 None	MW-11 11/13/96 None	MW-12 11/13/96 None	PFW-1 4/1/97 None	MW-4 11/14/96 None
2,4-Dinitrotoluene	1300	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2-Chloronaphthalene	6700	5200	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2-Chlorophenol	82000	130	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2-Methyl naphthalene	32000	750	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2-Methylphenol	710000	1000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2-Nitroaniline	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
2-Nitrophenol	72000	58	-	ND (5)	NA	ND	NA	NA	ND	ND	NA	NA
3,3-Dichlorobenzidine	270	8	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
3-Nitroaniline	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	8800	20	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
4-Chloro-3-methylphenol	62000	420	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
4-Chloroaniline	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
4-Methylphenol	75000	100	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
4-Nitroaniline	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
4-Nitrophenol	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Acenaphthene	4200	3800	4200	ND (5)	NA	ND	NA	NA	ND	ND	NA	NA
Acenaphthylene	3900	75	3900	NA	NA	NA	NA	NA	NA	NA	NA	NA
Anthracene	43	43	43	NA	NA	NA	NA	NA	NA	NA	NA	NA
Benzo(a)anthracene	5	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Benzo(a)pyrene	5	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Benzo(b)fluoranthene	5	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Benzo(g,h,i)perylene	5	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Benzo(k)fluoranthene	5	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroethoxy)methane	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroethyl)ether	2100	5	210000	NA	NA	NA	NA	NA	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	47	6	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Butyl benzylphthalate	2700	2700	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Carbazole	2900	170	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Chrysene	5	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Dibenzo(a,h)anthracene	5	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Dibenzofuran	-	-	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Diethyl phthalate	1100000	16000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Dimethyl phthalate	4200000	210000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Di-n-butylphthalate	11000	2500	-	ND (5)	NA	ND	NA	NA	ND	ND	NA	NA
Di-n-octyl phthalate	250	380	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Fluoranthene	210	210	210	NA	NA	NA	NA	NA	NA	NA	NA	NA
Fluorene	2000	2000	2000	ND (5)	NA	ND	NA	NA	ND	ND	NA	NA

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Groundwater Contact Criteria [2]	Industrial Drinking Water Criteria [2]	Industrial Groundwater Volatilization to Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI #								
				MW-6 11/13/96 None	MW-6 11/13/96 None	MW-6 11/13/96 None	MW-8 11/13/96 None	GP-7 8/30/96 None	MW-11 11/13/96 None	MW-12 11/13/96 None	PFW-1 4/1/97 None	MW-4 11/14/96 None
Hexachlorobenzene	2	1	3000	NA	NA	NA	NA	NA	NA	NA	NA	NA
Hexachlorobutadiene	200	45	3200	NA	NA	NA	NA	NA	NA	NA	NA	NA
Hexachlorocyclopentadiene	1400	50	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Hexachloroethane	1500	250	50000	NA	NA	NA	NA	NA	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	5	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Isophorone	1100000	3700	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Naphthalene	31000	750	31000	ND (5)	NA	ND	NA	NA	ND	ND	NA	NA
Nitrobenzene	9600	10	2100000	NA	NA	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	220	5	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
N-Nitrosodiphenylamine	30000	710	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Pentachlorophenol	85	1	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Phenanthrene	1000	75	1000	ND (5)	NA	ND	NA	NA	0.14	ND	NA	NA
Phenol	28000000	13000	-	NA	NA	NA	NA	NA	NA	NA	NA	NA
Pyrene	140	140	140	NA	NA	NA	NA	NA	NA	NA	NA	NA
Volatiles (ug/L)												
1,1,1-Trichloroethane	220000	200	1300000	ND (1)	NA	ND	ND	NA	ND	ND	ND (1)	NA
1,1,2,2-Tetrachloroethane	2100	17	77000	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
1,1,2-Trichloroethane	9500	5	110000	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
1,1-Dichloroethane	2100000	2500	5100000	ND (1)	NA	ND	ND	NA	ND	ND	ND (1)	NA
1,1-Dichloroethene	9000	7	1300	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
1,2-Dichlorobenzene	160000	600	160000	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
1,2-Dichloroethane	11000	5	59000	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
1,2-Dichloropropane	7500	5	36000	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
1,3-Dichlorobenzene	110000	600	-	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
1,4-Dichlorobenzene	2800	75	74000	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
2-Butanone	240000000	38000	240000000	NA	ND (50)	NA	NA	NA	NA	NA	NA	ND (50)
2-Chloroethyl vinyl ether	-	-	-	NA	NA	NA	NA	NA	NA	NA	ND (10)	NA
2-Hexanone	4800000	2900	8800000	NA	ND (50)	NA	NA	NA	NA	NA	NA	ND (50)
4-Methyl-2-pentanone	12000000	5200	20000000	NA	ND (50)	NA	NA	NA	NA	NA	NA	ND (50)
Acetone	31000000	2100	1000000000	NA	ND (100)	NA	NA	NA	NA	NA	NA	ND (100)
Benzene	9400	5	36000	NA	ND (5.0)	NA	NA	NA	NA	NA	ND (5)	ND (5.0)
Bromodichloromethane	11000	100	38000	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
Bromoform	100000	100	3100000	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
Bromomethane	65000	29	9000	ND (1)	NA	ND	ND	NA	ND	ND	ND (1)	NA
Carbon disulfide	1100000	2300	550000	NA	ND (50)	NA	NA	NA	NA	NA	NA	ND (50)
Carbon tetrachloride	1600	5	2400	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
Chlorobenzene	68000	100	470000	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
Chloroethane	200000	910	5700000	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
Chloroform	96000	100	180000	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
Chloromethane	110000	270	52000	ND (1)	NA	ND	ND	NA	ND	2.4	ND (1)	NA

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Groundwater Contact Criteria [2]	Industrial Drinking Water Criteria [2]	Industrial Groundwater Volatilization to Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI # MW-6 11/13/96 None	MW-6 11/13/96 None	MW-6 11/13/96 None	MW-8 11/13/96 None	GP-7 8/30/96 None	MW-11 11/13/96 None	MW-12 11/13/96 None	PFW-1 4/1/97 None	MW-4 11/14/96 None
cis-1,2-Dichloroethene	170000	70	3500000	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
cis-1,3-Dichloropropene	-	-	-	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
Dibromochloromethane	9500	100	110000	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
Dichlorodifluoromethane (CFC-12)	300000	4800	300000	NA	NA	NA	NA	NA	NA	NA	ND (1)	NA
Ethylbenzene	170000	74	170000	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
m&p-Xylene	-	-	-	NA	NA	NA	NA	NA	NA	NA	ND (3)	NA
Methylene chloride	110000	5	1400000	NA	ND (5.0)	NA	NA	NA	NA	NA	ND (5)	ND (5.0)
o-Xylene	-	-	-	NA	NA	NA	NA	NA	NA	NA	ND (3)	NA
Styrene	3200	100	310000	NA	ND (1.0)	NA	NA	NA	NA	NA	NA	ND (1.0)
Tetrachloroethene	5100	5	170000	ND (1)	NA	0.6	ND	NA	0.6	0.7	ND (1)	NA
Toluene	530000	790	530000	ND (1)	NA	0.7	1.4	NA	1.6	1.2	ND (1)	NA
trans-1,2-Dichloroethene	190000	100	6300000	ND (1)	NA	ND	ND	NA	ND	ND	ND (1)	NA
trans-1,3-Dichloropropene	-	-	-	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
Trichloroethene	11000	5	97000	ND (1)	NA	4.9	4.7	NA	6.2	7.1	ND (1)	NA
Trichlorofluoromethane (CFC-11)	1100000	7300	1100000	NA	NA	NA	NA	NA	NA	NA	ND (1)	NA
Vinyl chloride	290	2	690	NA	ND (1.0)	NA	NA	NA	NA	NA	ND (1)	ND (1.0)
Xylene (total)	190000	280	190000	NA	ND (3.0)	NA	NA	NA	NA	NA	NA	ND (3.0)

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Groundwater Contact Criteria [2]	Industrial Drinking Water Criteria [2]	Industrial Groundwater Volatilization to Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI # MW-4 11/14/96 None	MW-4 11/14/96 None	MW-10 11/13/96 None	MW-10 11/13/96 None
General Chemistry (mg/L)							
Diesel Fuel	-	-	-	NA	NA	NA	NA
Metals (ug/L)							
Arsenic (Dissolved)	4700	50	-	NA	NA	ND (5.0)	NA
Arsenic	4700	50	-	16	ND	NA	NA
Barium (Dissolved)	15000000	2000	-	NA	NA	NA	ND (200)
Barium	15000000	2000	-	NA	390	NA	NA
Cadmium (Dissolved)	210000	5	-	NA	NA	ND (0.50)	NA
Cadmium	210000	5	-	NA	NA	NA	NA
Chromium (Dissolved)	1000000	30000	-	NA	NA	ND (50)	NA
Chromium	1000000	30000	-	NA	NA	NA	NA
Copper (Dissolved)	8100000	1000	-	NA	NA	NA	ND (25)
Copper	8100000	1000	-	NA	ND	NA	NA
Lead (Dissolved)	-	4	-	NA	NA	ND (3.0)	NA
Lead	-	4	-	26	ND	NA	NA
Mercury (Dissolved)	56	2	-	NA	NA	ND (0.20)	NA
Mercury	56	2	-	NA	NA	NA	NA
Selenium (Dissolved)	1100000	50	-	NA	NA	5.4	NA
Selenium	1100000	50	-	12	ND	NA	NA
Silver (Dissolved)	1000000	98	-	NA	NA	ND (0.50)	NA
Silver	1000000	98	-	NA	NA	NA	NA
Zinc (Dissolved)	70000000	5000	-	NA	NA	NA	ND (20)
Zinc	70000000	5000	-	NA	ND	NA	NA
PCBs (ug/L)							
Aroclor-1016	-	-	-	NA	NA	NA	NA
Aroclor-1221	-	-	-	NA	NA	NA	NA
Aroclor-1232	-	-	-	NA	NA	NA	NA
Aroclor-1242	-	-	-	NA	NA	NA	NA
Aroclor-1248	-	-	-	NA	NA	NA	NA
Aroclor-1254	-	-	-	NA	NA	NA	NA
Aroclor-1260	-	-	-	NA	NA	NA	NA
Total PCBs	2.3	0.5	45	NA	NA	NA	ND (.2)
Semi - Volatiles (ug/L)							
1,2,4-Trichlorobenzene	15000	70	300000	NA	NA	NA	NA
2,4,5-Trichlorophenol	130000	2100	-	NA	NA	NA	NA
2,4,6-Trichlorophenol	5500	320	-	NA	NA	NA	NA
2,4-Dichlorophenol	40000	210	-	NA	NA	NA	NA
2,4-Dimethylphenol	440000	1000	-	NA	NA	NA	NA
2,4-Dinitrophenol	-	-	-	NA	NA	NA	NA

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Groundwater Contact Criteria [2]	Industrial Drinking Water Criteria [2]	Industrial Groundwater Volatilization to Inhalation Criteria [2]	Sample Location (depth ft. bgs) / Sample Date / AOI # MW-4 11/14/96 None	MW-4 11/14/96 None	MW-10 11/13/96 None	MW-10 11/13/96 None
2,4-Dinitrotoluene	1300	5	-	NA	NA	NA	NA
2,6-Dinitrotoluene	-	-	-	NA	NA	NA	NA
2-Chloronaphthalene	6700	5200	-	NA	NA	NA	NA
2-Chlorophenol	82000	130	-	NA	NA	NA	NA
2-Methyl naphthalene	32000	750	-	NA	NA	NA	NA
2-Methylphenol	710000	1000	-	NA	NA	NA	NA
2-Nitroaniline	-	-	-	NA	NA	NA	NA
2-Nitrophenol	72000	58	-	NA	NA	NA	NA
3,3-Dichlorobenzidine	270	8	-	NA	NA	NA	NA
3-Nitroaniline	-	-	-	NA	NA	NA	NA
4,6-Dinitro-2-methylphenol	8800	20	-	NA	NA	NA	NA
4-Bromophenyl phenyl ether	-	-	-	NA	NA	NA	NA
4-Chloro-3-methylphenol	62000	420	-	NA	NA	NA	NA
4-Chloroaniline	-	-	-	NA	NA	NA	NA
4-Chlorophenyl phenyl ether	-	-	-	NA	NA	NA	NA
4-Methylphenol	75000	100	-	NA	NA	NA	NA
4-Nitroaniline	-	-	-	NA	NA	NA	NA
4-Nitrophenol	-	-	-	NA	NA	NA	NA
Acenaphthene	4200	3800	4200	NA	NA	NA	NA
Acenaphthylene	3900	75	3900	NA	NA	NA	NA
Anthracene	43	43	43	NA	NA	NA	NA
Benzo(a)anthracene	5	5	-	NA	NA	NA	NA
Benzo(a)pyrene	5	5	-	NA	NA	NA	NA
Benzo(b)fluoranthene	5	5	-	NA	NA	NA	NA
Benzo(g,h,i)perylene	5	5	-	NA	NA	NA	NA
Benzo(k)fluoranthene	5	5	-	NA	NA	NA	NA
bis(2-Chloroethoxy)methane	-	-	-	NA	NA	NA	NA
bis(2-Chloroethyl)ether	2100	5	210000	NA	NA	NA	NA
bis(2-Chloroisopropyl)ether	-	-	-	NA	NA	NA	NA
bis(2-Ethylhexyl)phthalate	47	6	-	NA	NA	NA	NA
Butyl benzylphthalate	2700	2700	-	NA	NA	NA	NA
Carbazole	2900	170	-	NA	NA	NA	NA
Chrysene	5	5	-	NA	NA	NA	NA
Dibenzo(a,h)anthracene	5	5	-	NA	NA	NA	NA
Dibenzofuran	-	-	-	NA	NA	NA	NA
Diethyl phthalate	1100000	16000	-	NA	NA	NA	NA
Dimethyl phthalate	4200000	210000	-	NA	NA	NA	NA
Di-n-butylphthalate	11000	2500	-	NA	NA	NA	NA
Di-n-octyl phthalate	250	380	-	NA	NA	NA	NA
Fluoranthene	210	210	210	NA	NA	NA	NA
Fluorene	2000	2000	2000	NA	NA	NA	NA

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	*Groundwater Contact Criteria* [2]	*Industrial Drinking Water Criteria* [2]	*Industrial Groundwater Volatilization to Inhalation Criteria* [2]	*Sample Location (depth ft. bgs)* / *Sample Date* / *AOI #* MW-4 11/14/96 None	MW-4 11/14/96 None	MW-10 11/13/96 None	MW-10 11/13/96 None
Hexachlorobenzene	2	1	3000	NA	NA	NA	NA
Hexachlorobutadiene	200	45	3200	NA	NA	NA	NA
Hexachlorocyclopentadiene	1400	50	-	NA	NA	NA	NA
Hexachloroethane	1500	250	50000	NA	NA	NA	NA
Indeno(1,2,3-cd)pyrene	5	5	-	NA	NA	NA	NA
Isophorone	1100000	3700	-	NA	NA	NA	NA
Naphthalene	31000	750	31000	NA	NA	NA	NA
Nitrobenzene	9600	10	2100000	NA	NA	NA	NA
N-Nitrosodi-n-propylamine	220	5	-	NA	NA	NA	NA
N-Nitrosodiphenylamine	30000	710	-	NA	NA	NA	NA
Pentachlorophenol	85	1	-	NA	NA	NA	NA
Phenanthrene	1000	75	1000	NA	NA	NA	NA
Phenol	28000000	13000	-	NA	NA	NA	NA
Pyrene	140	140	140	NA	NA	NA	NA
Volatiles (ug/L)							
1,1,1-Trichloroethane	220000	200	1300000	ND (1)	ND	ND (1.0)	NA
1,1,2,2-Tetrachloroethane	2100	17	77000	NA	NA	ND (1.0)	NA
1,1,2-Trichloroethane	9500	5	110000	NA	NA	ND (1.0)	NA
1,1-Dichloroethane	2100000	2500	5100000	ND (1)	ND	ND (1.0)	NA
1,1-Dichloroethene	9000	7	1300	NA	NA	ND (1.0)	NA
1,2-Dichlorobenzene	160000	600	160000	NA	NA	ND (1.0)	NA
1,2-Dichloroethane	11000	5	59000	NA	NA	ND (1.0)	NA
1,2-Dichloropropane	7500	5	36000	NA	NA	ND (1.0)	NA
1,3-Dichlorobenzene	110000	600	-	NA	NA	ND (1.0)	NA
1,4-Dichlorobenzene	2800	75	74000	NA	NA	ND (1.0)	NA
2-Butanone	240000000	38000	240000000	NA	NA	ND (50)	NA
2-Chloroethyl vinyl ether	-	-	-	NA	NA	NA	NA
2-Hexanone	4800000	2900	8800000	NA	NA	ND (50)	NA
4-Methyl-2-pentanone	12000000	5200	20000000	NA	NA	ND (50)	NA
Acetone	31000000	2100	1000000000	NA	NA	ND (100)	NA
Benzene	9400	5	36000	NA	NA	ND (5.0)	NA
Bromodichloromethane	11000	100	38000	NA	NA	ND (1.0)	NA
Bromoform	100000	100	3100000	NA	NA	ND (1.0)	NA
Bromomethane	65000	29	9000	ND (1)	ND	ND (1.0)	NA
Carbon disulfide	1100000	2300	550000	NA	NA	ND (50)	NA
Carbon tetrachloride	1600	5	2400	NA	NA	ND (1.0)	NA
Chlorobenzene	68000	100	470000	NA	NA	ND (1.0)	NA
Chloroethane	200000	910	5700000	NA	NA	ND (1.0)	NA
Chloroform	96000	100	180000	NA	NA	ND (1.0)	NA
Chloromethane	110000	270	52000	ND (1)	ND	ND (1.0)	NA

TABLE H.2

WATER ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	Groundwater Contact Criteria (2)	Industrial Drinking Water Criteria (2)	Industrial Groundwater Volatilization to Inhalation Criteria (2)	Sample Location (depth ft. bgs) / Sample Date / AOI # MW-4 11/14/96 None	MW-4 11/14/96 None	MW-10 11/13/96 None	MW-10 11/13/96 None
cis-1,2-Dichloroethene	170000	70	3500000	NA	NA	ND (1.0)	NA
cis-1,3-Dichloropropene	-	-	-	NA	NA	ND (1.0)	NA
Dibromochloromethane	9500	100	110000	NA	NA	ND (1.0)	NA
Dichlorodifluoromethane (CFC-12)	300000	4800	300000	NA	NA	NA	NA
Ethylbenzene	170000	74	170000	NA	NA	ND (1.0)	NA
m&p-Xylene	-	-	-	NA	NA	NA	NA
Methylene chloride	110000	5	1400000	NA	NA	ND (5.0)	NA
o-Xylene	-	-	-	NA	NA	NA	NA
Styrene	3200	100	310000	NA	NA	ND (1.0)	NA
Tetrachloroethene	5100	5	170000	ND (1)	0.7	ND (1.0)	NA
Toluene	530000	790	530000	ND (1)	0.8	ND (1.0)	NA
trans-1,2-Dichloroethene	190000	100	6300000	ND (1)	ND	ND (1.0)	NA
trans-1,3-Dichloropropene	-	-	-	NA	NA	ND (1.0)	NA
Trichloroethene	11000	5	97000	ND (1)	5.3	ND (1.0)	NA
Trichlorofluoromethane (CFC-11)	1100000	7300	1100000	NA	NA	NA	NA
Vinyl chloride	290	2	690	NA	NA	ND (1.0)	NA
Xylene (total)	190000	280	190000	NA	NA	ND (3.0)	NA

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14

TABLE H.3

TCLP ANALYTICAL RESULTS
FORMER PEREGRINE (US) INC.
COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

		Sample Location (depth ft. bgs)										
		Sample Date										
		AOI #										
Parameter	*Regulatory Levels* [1]	*Plater: South Box Tank* 4/4/97	*Plater: Tank 22 Vent Box* 4/4/97	*Plater: Tanks 19/20 Grate* 4/4/97	*Plater: South AST* 4/4/97	*Overhead Waste Pipe 1* 4/4/97	*Overhead Waste Pipe 2* 4/4/97	*Overhead Waste Pipe 3* 4/4/97	*Overhead Waste Pipe 4* 4/4/97	*Overhead Waste Pipe 5* 4/4/97	*AST No.6* 4/25/97	*Tool Room Air Wash Unit* 4/7/97
		AOI # 9	*AOI # 9*	*AOI # 9*	*AOI # 9*	*AOI # 10*	*AOI # 10*	*AOI # 10*	*AOI # 10*	*AOI # 10*	*AOI # 13*	*None*
Metals (ug/L)												
Arsenic	5000	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)	11
Barium	100000	ND (600)	700	ND (600)	ND (600)	ND (600)	1200	ND (600)	ND (600)	ND (600)	ND (600)	1200
Cadmium	1000	ND (10)	370	ND (10)	ND (10)	130	40	10	30	30	10	150
Chromium	5000	1200	2000	60	730	100	1400	ND (0.02)	40	ND (0.02)	60	8800
Copper	-	1600	30000	60	350	750	1700	60	50	10	ND (20)	11000
Lead	5000	650	3900	ND (80)	590	ND (80)	130	ND (80)	ND (80)	130	ND (80)	6100
Mercury	200	ND (1)	2	ND (1)	ND (1)	ND (1)	ND (1)	ND (1)	ND (1)	ND (1)	ND (2)	ND (1)
Selenium	1000	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)	ND (5)
Silver	5000	40	130	ND (20)	30	ND (20)	ND (20)	ND (20)	ND (20)	ND (20)	ND (20)	ND (20)
Zinc	-	4800000	620000	6800000	5100000	23000	270000	850000	3200000	2500	100	12000
Volatiles (ug/L)												
1,1-Dichloroethene	700	NA	NA	NA	NA	NA	NA	NA	NA	NA	ND (2000)	NA
1,2-Dichloroethane	500	NA	NA	NA	NA	NA	NA	NA	NA	NA	ND (200)	NA
1,4-Dichlorobenzene	7500	NA	NA	NA	NA	NA	NA	NA	NA	NA	ND (500)	NA
2-Butanone	-	NA	NA	NA	NA	NA	NA	NA	NA	NA	ND (1000)	NA
Benzene	500	NA	NA	NA	NA	NA	NA	NA	NA	NA	79000	NA
Carbon tetrachloride	500	NA	NA	NA	NA	NA	NA	NA	NA	NA	ND (200)	NA
Chlorobenzene	100000	NA	NA	NA	NA	NA	NA	NA	NA	NA	ND (500)	NA
Chloroform	6000	NA	NA	NA	NA	NA	NA	NA	NA	NA	ND (200)	NA
Tetrachloroethene	700	NA	NA	NA	NA	NA	NA	NA	NA	NA	ND (300)	NA
Trichloroethene	500	NA	NA	NA	NA	NA	NA	NA	NA	NA	ND (500)	NA
Vinyl chloride	200	NA	NA	NA	NA	NA	NA	NA	NA	NA	ND (300)	NA

Notes:

_____ TCLP Criteria exceeded.

Metals - Michigan 10 Metals list

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(1) - Resource Conservation and Recovery Act (RCRA) (40 CFR 261.21-24).

TABLE H.4

PCB ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

		Sample Location (depth ft. bgs)						
		Sample Date						
		AOI #						
Parameter	*Regulatory Limit* [4]	*Wood Floor Blocks ML-1_5 4/4/97*	*Wood Floor Blocks H-19_2 4/4/97*	*Beneath Transformer 1A Wipe 4A 4/4/97*	*Beneath Transformer 1A Wipe 4B 4/4/97*	*Beneath Transformer 9 Wipe 3A 4/4/97*	*Beneath Transformer 9 Wipe 3B 4/4/97*	*C-7 11/7/96*
		AOI #19	*AOI #20*	*None*	*None*	*None*	*None*	*None*
General Chemistry (mg/Kg)								
Petroleum hydrocarbons	10	NA	NA	NA	NA	NA	NA	NA
Total Solids (%)	-	NA	NA	NA	NA	NA	NA	NA
PCBs (ug/100cm2)								
Aroclor-1016	10	NA	NA	NA	NA	NA	NA	ND (1.0)
Aroclor-1221	10	NA	NA	NA	NA	NA	NA	ND (1.0)
Aroclor-1232	10	NA	NA	NA	NA	NA	NA	ND (1.0)
Aroclor-1242	10	NA	NA	NA	NA	NA	NA	ND (1.0)
Aroclor-1248	10	NA	NA	NA	NA	NA	NA	ND (1.0)
Aroclor-1254	10	NA	NA	NA	NA	NA	NA	ND (1.0)
Aroclor-1260	10	NA	NA	NA	NA	NA	NA	ND (1.0)
Total PCBs	50000[3]	NA	NA	NA	NA	NA	NA	NA
Total PCBs	10	NA	NA	5.5	11	20	ND (5)	NA
Total PCBs (ug/Kg)	9900[2]	ND (1000)	ND (1000)	NA	NA	NA	NA	NA

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14.

(3) - Toxic Substances Control Act (40 CFR 761.60).

(4) - Toxic Substances Control Act (40 CFR 761.125).

TABLE H.4

PCB ANALYTICAL RESULTS
FORMER PEREGRINE (US), INC., COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

Parameter	*Regulatory Limit*[4]	*Galbestos* 4/4/97 None	*N-27* 11/7/96 None	*Pl Floor near C7spill Wipe 2A* 4/4/97 None	*Pl Floor near C7spill Wipe 2B* 4/4/97 None	*Pl Floor near C7spill Wipe 2C* 4/4/97 None	*Wood Floor Blocks D-3* 4/4/97 None	*Wood Floor Blocks F-2* 4/4/97 None	*Wood Floor Blocks HJ-11_7* 4/4/97 None
		Sample Location (depth ft. bgs) / *Sample Date* / *AOI #*							
General Chemistry (mg/Kg)									
Petroleum hydrocarbons	10	NA	56	NA	NA	NA	NA	NA	NA
Total Solids (%)	-	NA	87.6	NA	NA	NA	NA	NA	NA
PCBs (ug/100cm2)									
Aroclor-1016	10	NA	ND (1.0)	NA	NA	NA	NA	NA	NA
Aroclor-1221	10	NA	ND (1.0)	NA	NA	NA	NA	NA	NA
Aroclor-1232	10	NA	ND (1.0)	NA	NA	NA	NA	NA	NA
Aroclor-1242	10	NA	ND (1.0)	NA	NA	NA	NA	NA	NA
Aroclor-1248	10	NA	ND (1.0)	NA	NA	NA	NA	NA	NA
Aroclor-1254	10	NA	ND (1.0)	NA	NA	NA	NA	NA	NA
Aroclor-1260	10	NA	ND (1.0)	NA	NA	NA	NA	NA	NA
Total PCBs	50000[3]	1300	NA	NA	NA	NA	NA	NA	NA
Total PCBs	10	NA	NA	ND (5)	ND (5)	ND (5)	NA	NA	NA
Total PCBs (ug/Kg)	9900[2]	NA	NA	NA	NA	NA	ND (1000)	ND (1000)	ND (1000)

Notes:

PCBs - Polychlorinated Biphenyls.

TPH - Total Petroleum Hydrocarbons Diesel Range Organics (DRO) EPA Method 8015 modified.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

NA - Parameter not analyzed.

(2) - MDEQ/ERD Operational Memorandum No. 14.

(3) - Toxic Substances Control Act (40 CFR 761.60).

(4) - Toxic Substances Control Act (40 CFR 761.125).

TABLE H.5

WIPE ANALYTICAL RESULTS
FORMER PEREGRINE (US) INC.
COLDWATER ROAD FACILITY
GENESEE TOWNSHIP, MICHIGAN

		Sample Location (depth ft. bgs)						
		Sample Date						
		AOI #						
Parameter	*Regulatory Limits* [4]	*BeneathTransformer 1A Wipe 4A 4/4/97*	*BeneathTransformer 1A Wipe 4B 4/4/97*	*BeneathTransformer 9 Wipe 3A 4/4/97*	*BeneathTransformer 9 Wipe 3B 4/4/97*	*PI Floor near C7spill Wipe 2A 4/4/97*	*PI Floor near C7spill Wipe 2B 4/4/97*	*PI Floor near C7spill Wipe 2C 4/4/97*
		None	*None*	*None*	*None*	*None*	*None*	*None*
Total PCBs (ug/100cm2)	10	5.5	11	20	ND (5)	ND (5)	ND (5)	ND (5)

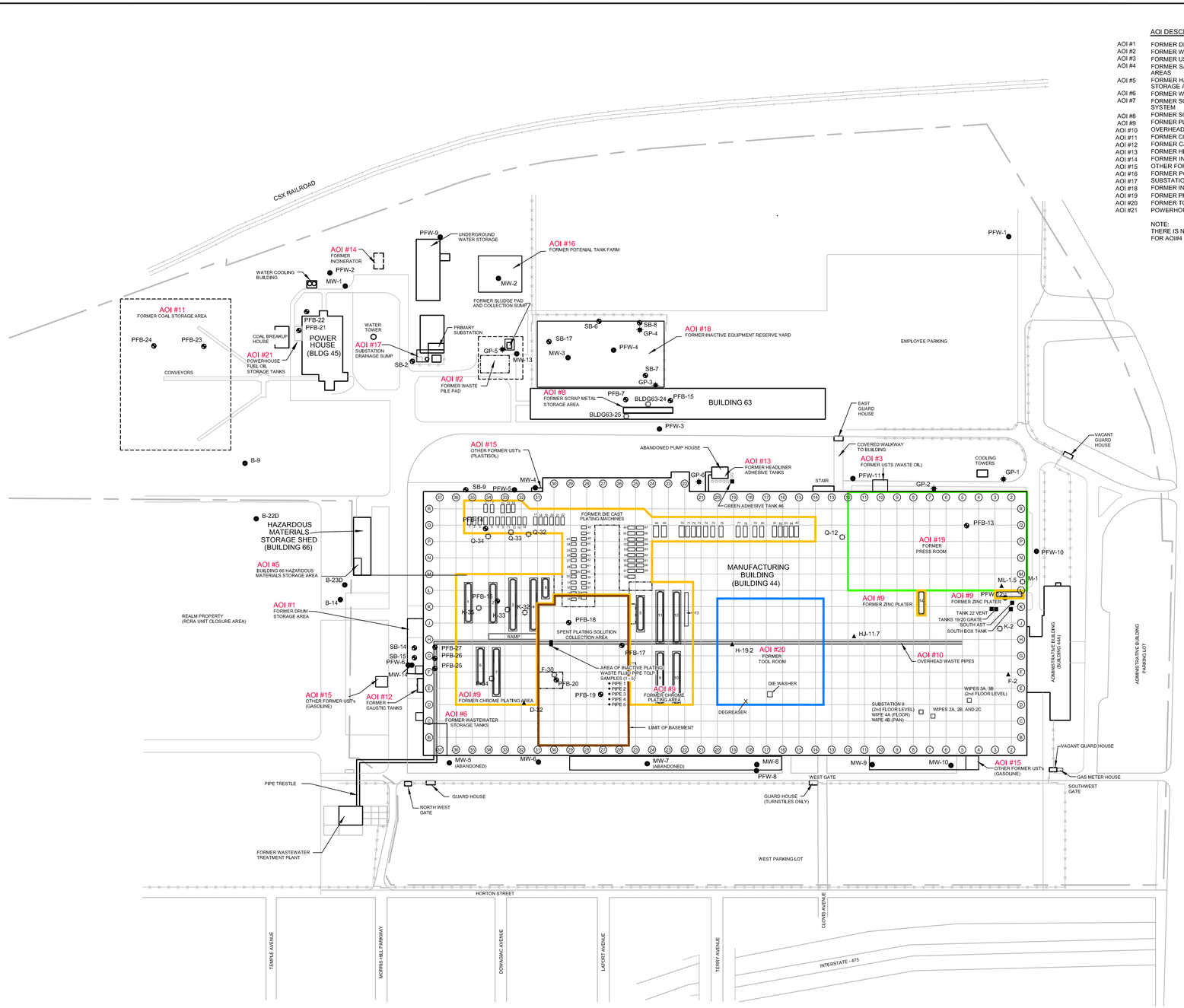
Notes:

_____ Regulatory Limits exceeded.

PCBs - Polychlorinated Biphenyls.

ND ( ) - Parameter not detected above quantitation limit in CRA sample quantitation limit stated in parentheses.

(4) - Toxic Substances Control Act (40 CFR 761.125).



AOI DESCRIPTION

AOI #1	FORMER DRUM STORAGE AREA
AOI #2	FORMER WASTE PILE PAD
AOI #3	FORMER USTs
AOI #4	FORMER SATELLITE ACCUMULATION AREAS
AOI #5	FORMER HAZARDOUS MATERIALS STORAGE AREA
AOI #6	FORMER WASTEWATER STORAGE TANKS
AOI #7	FORMER SCRAP METAL MANAGEMENT SYSTEM
AOI #8	FORMER SCRAP METAL STORAGE AREA
AOI #9	FORMER PLATING AREAS
AOI #10	OVERHEAD WASTE PIPES
AOI #11	FORMER COAL STORAGE AREA
AOI #12	FORMER CAUSTIC TANKS
AOI #13	FORMER HEADLINER ADHESIVE TANKS
AOI #14	FORMER INCINERATOR
AOI #15	OTHER FORMER USTs
AOI #16	FORMER POTENIAL TANK FARM
AOI #17	SUBSTATION DRAINAGE SUMP
AOI #18	FORMER INACTIVE EQUIPMENT RESERVE YARD
AOI #19	FORMER PRESS ROOM
AOI #20	FORMER TOOL ROOM
AOI #21	POWERHOUSE FUEL OIL STORAGE TANKS

NOTE:
THERE IS NO SPECIFIC LOCATION FOR AOI#4 AND AOI #7.

№	Revision	Date	Initial

0 50 150ft

LEGEND

- PROPERTY BOUNDARY
- FENCE LINE
- MW-1 — STUDY 2 MONITORING WELL LOCATION AND NUMBER
- PFW-1 — STUDY 4 MONITORING WELL LOCATION AND NUMBER
- B-9 — GENERAL MOTORS RCRA UNIT CLOSURE MONITORING WELL LOCATION AND NUMBER
- PFB-7 — STUDY 4 SOIL BORING LOCATION AND NUMBER
- SB-2 — STUDY 2 SOIL BORING LOCATION AND NUMBER
- GR-1 — STUDY 1 GEOPROBE LOCATION AND NUMBER
- Q-34 — STUDY 2 GEOPROBE LOCATION AND NUMBER
- H-192 — WOOD BLOCK SAMPLE LOCATION
- WIPE 3A — WIPE SAMPLE LOCATION
- TANK 22 VENT — TCLP SAMPLE LOCATION

NOTES

1. THIS DRAWING IS FOR REFERENCE ONLY AND IS NEITHER COMPLETE NOR TO SCALE.
2. DRAWING No. 1795W BUICK MOTOR DIVISION SHEET No. 1M WAS USED AS A REFERENCE DRAWING.
3. LOCATIONS OF GENERAL MOTORS RCRA UNIT CLOSURE MONITORING WELLS (i.e. B-WELLS) ARE APPROXIMATE

SCALE VERIFICATION

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

Approved

DRAWING STATUS

Status	Date	Initial

FORMER PEREGRINE (US), INC.
COLDWATER ROAD FACILITY

REALM

SITE PLAN

CRA CONESTOGA-ROVERS & ASSOCIATES

Source Reference:
RMT, INC.

Project Manager:	Reviewed By:	Date:	
G.T.	C.R.H.	MARCH 2000	
Scale: 1"=150"	Project N º: 12636-40	Report N º: 011	Drawing N º: 1

12636-40(011)GN-WA029 JUN 22/2000

K-35		
LEAD	SOIL	1,280,000 ug/kg
CADMIUM	SOIL	6,050 ug/kg
CHROMIUM	SOIL	1,160,000 ug/kg

SAMPLE LOCATION
CONCENTRATION (ppm)
MEDIA
COMPOUND
VALUE EXCEEDS DIRECT CONTACT VALUE
VALUE EXCEEDS DRINKING WATER CRITERIA

PFW-9		
LEAD	WATER	9 ug/L

MW-13		
TCE	WATER	8.4 ug/L

MW-3		
LEAD	WATER	12 ug/L

PFB-7		
ARSENIC	SOIL	32,000 ug/kg

PFW-10		
BARIUM	WATER	2,600 ug/L

MW-4		
TCE	WATER	5.3 ug/L
LEAD	WATER	20 ug/L

K-35		
LEAD	SOIL	1,280,000 ug/kg
CADMIUM	SOIL	6,050 ug/kg
CHROMIUM	SOIL	1,160,000 ug/kg

PFW-6		
TCE	SOIL	1,600 ug/kg

MW-14		
TCE	SOIL	2,210 ug/kg
TCE	WATER	21.3 ug/L / 6 ug/L

WWT-3		
TCE	SOIL	139 ug/kg

MW-11		
TCE	WATER	6.2 ug/L
LEAD	WATER	22 ug/L

MW-12		
CYANIDE	SOIL	88,300 ug/kg
TCE	WATER	7.1 ug/L

K-32		
LEAD	SOIL	42,800 ug/kg

F-30		
CYANIDE	SOIL	31,800 ug/kg

MW-6		
LEAD	SOIL	103,000 ug/kg

MW-10		
LEAD	WATER	14 ug/L

AOI DESCRIPTION

AOI #1	FORMER DRUM STORAGE AREA
AOI #2	FORMER WASTE PILE PAD
AOI #3	FORMER USTs
AOI #4	FORMER SATELLITE ACCUMULATION AREAS
AOI #5	FORMER HAZARDOUS MATERIALS STORAGE AREA
AOI #6	FORMER WASTEWATER STORAGE TANKS
AOI #7	FORMER SCRAP METAL MANAGEMENT SYSTEM
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AOI #11	FORMER COAL STORAGE AREA
AOI #12	FORMER CAUSTIC TANKS
AOI #13	FORMER CAUSTIC TANKS
AOI #14	FORMER HEADLINER ADHESIVE TANKS
AOI #15	FORMER INCINERATOR
AOI #16	OTHER FORMER USTs
AOI #17	FORMER POTENTIAL TANK FARM
AOI #18	SUBSTATION DRAINAGE SUMP
AOI #19	FORMER INACTIVE EQUIPMENT RESERVE YARD
AOI #20	FORMER PRESS ROOM
AOI #21	FORMER TOOL ROOM
	POWERHOUSE FUEL OIL STORAGE TANKS

NOTE:
THERE IS NO SPECIFIC LOCATION FOR AOI#4 AND AOI #7.

No	Revision	Date	Initial

0 50 150ft

LEGEND

- PROPERTY BOUNDARY
- FENCE LINE
- MW-1 — STUDY 2 MONITORING WELL LOCATION AND NUMBER
- PFW-1 — STUDY 4 MONITORING WELL LOCATION AND NUMBER
- B-9 — GENERAL MOTORS RCRA UNIT CLOSURE MONITORING WELL LOCATION AND NUMBER
- PFB-7 — STUDY 4 SOIL BORING LOCATION AND NUMBER
- SB-2 — STUDY 2 SOIL BORING LOCATION AND NUMBER
- GP-1 — STUDY 1 GEOPROBE LOCATION AND NUMBER
- Q-34 — STUDY 2 GEOPROBE LOCATION AND NUMBER
- H-192 — WOOD BLOCK SAMPLE LOCATION
- WIPE 3A — WIPE SAMPLE LOCATION
- TANK 22 VENT — TCLP SAMPLE LOCATION
- BH-9-2-00 — PROPOSED BOREHOLE SAMPLE
- BK-2-00 — BACKGROUND BOREHOLE SAMPLE
- MW-10 — RESAMPLE EXISTING WELL

BOREHOLE 2000
BH-9-2-00
AOI # LOCATION #

NOTES
1. THIS DRAWING IS FOR REFERENCE ONLY AND IS NEITHER COMPLETE NOR TO SCALE.
2. DRAWING No. 1795W BUICK MOTOR DIVISION SHEET No. 1M WAS USED AS A REFERENCE DRAWING.
3. LOCATIONS OF GENERAL MOTORS RCRA UNIT CLOSURE MONITORING WELLS (i.e. B-WELLS) ARE APPROXIMATE

SCALE VERIFICATION

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

Approved

DRAWING STATUS

Status	Date	Initial

FORMER PEREGRINE (US), INC.
COLDWATER ROAD FACILITY

REALM

PROPOSED RFI SAMPLING LOCATIONS

CRA CONESTOGA-ROVERS & ASSOCIATES

Source Reference: RMT, INC.			
Project Manager: G.T.	Reviewed By: C.R.H.	Date: MARCH 2000	
Scale: 1"=150"	Project N º: 12636-40	Report N º: 011	Drawing N º: 2

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