

M67001.AR.006350
MCB CAMP LEJUENE
5090.3a

FINAL PRELIMINARY ASSESSMENT/SITE INSPECTION WORK PLAN FRENCH CREEK
ROAD EXTENSION MCB CAMP LEJEUNE NC
3/1/2009
CH2M HILL

Final

**Preliminary Assessment/Site Inspection Work Plan
French Creek Road Extension**

**Marine Corps Base Camp Lejeune
Jacksonville, North Carolina**

Contract Task Order 011

March 2009

Prepared for

**Department of the Navy
Naval Facilities Engineering Command
Atlantic**

Under the

**NAVFAC CLEAN 1000 Program
Contract N62470-08-D-1000**

Prepared by



CH2MHILL

Charlotte, North Carolina

Contents

Acronyms and Abbreviations	iii
1 Project Overview.....	1-1
1.1 Purpose of Preliminary Assessment/Site Inspection	1-1
1.2 Preliminary Assessment/Site Inspection Objective.....	1-1
1.3 Work Plan Organization	1-2
2 Site Background and Setting.....	2-1
2.1 French Creek Road Extension Location and Description.....	2-1
2.2 Site 28 History and Previous Investigations	2-1
2.2.1 Operational Use	2-1
2.2.2 Previous Investigations	2-1
3 Field Activities	3-1
3.1 Records Review	3-1
3.2 Buried Utility Locating.....	3-1
3.3 DPT Soil Sampling	3-2
3.4 Temporary Monitoring Well Installation	3-2
3.4.1 Basis for Proposed Well Locations and Depths.....	3-2
3.4.2 Installation Procedures.....	3-3
3.4.3 Monitoring Well Gauging.....	3-3
3.5 Groundwater Sampling	3-3
3.6 Geospatial Information	3-4
4 IDW Management	4-1
4.1 Waste Streams	4-1
4.2 Waste Management	4-1
4.2.1 Decontamination Fluids/Development Water.....	4-1
4.2.2 Soil Cuttings	4-1
4.2.3 PPE and Trash	4-2
5 Data Management and Evaluation.....	5-1
6 Preliminary Assessment/Site Inspection Report	6-1
7 Project Management.....	7-1
8 Schedule	8-1
9 References	9-1

Appendices

A	Remedial Investigation and Final Closeout Report Tables and Figures
B	Archive Records Search Report
C	Field Sampling Plan
D	UFP-QAPP Attachment

Table

3-1 Sample Analysis Summary

Figures

2-1 Location Map

2-2 Site Map

2-3 Topographic Map

2-4 Previous 1995 Soil Investigation

2-5 Previous 1995 Groundwater Investigation

2-6 Boundary of Land Use Controls

2-7 Boundary of Aquifer Use Controls

3-1 Utilities Map

3-2 Proposed PA/SI Soil Sample Locations

3-3 Proposed PA/SI Temporary Well Sample Locations

8-1 Proposed Schedule

Acronyms and Abbreviations

ASR	Archival Search Report
bgs	Below Ground Surface
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
COPC	Contaminants of Potential Concern
CS	Confirmation Study
DPT	Direct Push Technology
FID	Flame-ionization Detector
FS	Feasibility Study
FSP	Field Sampling Plan
ft/amsl	Feet above mean sea level
FTL	Field Team Leader
IAS	Initial Assessment Study
ID	Inner Diameter
IDW	Investigation Derived Waste
IR	Installation Restoration
LTM	Long-term monitoring
LUCIP	Land Use Controls Implementation Plan
MCB	Marine Corps Base
NCDENR	North Carolina Department of Environment and Natural Resources
NCGWQS	North Carolina Groundwater Quality Standards
NPL	National Priorities List
PA/SI	Preliminary Assessment/Site Assessment
PCB	Polychlorinated Biphenyl
PPE	Personal Protective Equipment
PVC	Polyvinyl Chloride
QAPP	Quality Assurance Project Plan
RACR	Remedial Action Close-out Report
RCRA	Resource Conservation and Recovery Act
RI	Remedial Investigation
ROD	Record of Decision
RSL	Regional Screening Level
SAP	Sampling and Analysis Plan
SI	Site Inspection
SOP	Standard Operating Procedures
SVOC	Semi-volatile Organic Compound

TAL	Target Analyte List
TCDD	2,3,7,8-tetrachlorodibenzo-p-dioxin
TCE	Trichloroethylene
UFP-SAP	Uniform Federal Policy for Sampling and Analysis Plans
USEPA	United States Environmental Protection Agency
UTM	Universal Transverse Mercator
VOC	Volatile Organic Compound

Project Overview

The United States Marine Corps plans to extend French Creek Road in the Hadnot Point area of Marine Corps Base (MCB) Camp Lejeune. A portion of the proposed extension is located within and adjacent to Installation Restoration (IR) Site 28, the former Hadnot Point Burn Dump. The MCB Camp Lejeune complex consists of six geographical locations under the jurisdiction of the Base command. These areas include Camp Geiger, Montford Point, Courthouse Bay, Mainside, the Greater Sandy Run Area, and the Rifle Range Area. The Hadnot Point development area is located within the Mainside area of MCB Camp Lejeune.

This Preliminary Assessment/Site Inspection (PA/SI) Work Plan has been prepared to evaluate the presence and nature of environmental impacts resulting from historical land use practices in the vicinity of the planned French Creek Road Extension area which may result in potential risks to construction workers during road construction. CH2M HILL has prepared this PA/SI Work Plan under contract N-62470-08-D-1000 Task Order 11. The following sections describe the objectives, scope, and schedule for the proposed assessment activities.

1.1 Purpose of Preliminary Assessment/Site Inspection

PA investigations collect readily available information about a site and its surrounding area. The PA is designed to distinguish, based on limited data, between sites that present little or no risk to human health and the environment and sites that may pose a risk and require further investigation. The PA also identifies sites requiring assessment for possible emergency response actions. If the PA results in a recommendation for further investigation, a SI is performed.

SI investigations typically involve the collection of environmental and waste samples to determine whether hazardous substances are present at a site; to determine if these substances are being released to the environment; and to assess if the substances have reached nearby receptors.

This work plan describes the proposed activities to be conducted during the PA/SI investigation of the proposed French Creek Road Extension.

1.2 Preliminary Assessment/Site Inspection Objective

The objective of the French Creek Road PA/SI activities is to assess the potential risk to human health and the environment under a construction worker scenario and determine whether additional environmental assessment is necessary.

1.3 Work Plan Organization

This Work Plan is organized as follows:

- **Introduction (Section 1)** – Presents an overview of the project, objective, and work plan organization.
- **Site Background and Setting (Section 2)** – Presents the site location and general description, environmental setting, operational history, and previous investigations.
- **Field Activities (Section 3)** – Presents an overview of proposed field activities including site reconnaissance, buried utility locating, and soil and groundwater sampling.
- **Investigation Derived Waste (IDW) Management (Section 4)** – Discusses how IDW generated during field activities will be managed and disposed.
- **Data Management (Section 5)** - Outlines how data will be managed, validated, and evaluated.
- **PA/SI Report (Section 6)** – Provides the reporting that will occur for the project.
- **Project Management (Section 7)** – Presents an overview of the project management and staffing.
- **Schedule (Section 8)** - Provides the project schedule.
- **References (Section 9)** - Provides the references used in this document.

Tables and figures accompanying the main text of this plan are included at the end of each section. Data collected during previous investigations are summarized by tables and figures in **Appendix A**. The Archival Search Report (ASR), the site-specific Field Sampling Plan (FSP), and Uniform Federal Policy for Quality Assurance Project Plan (UFP-QAPP) Attachment are included as **Appendix B**, **Appendix C**, and **Appendix D**, respectively. These documents are supported by the MCB Camp Lejeune Master Project Plans (CH2M HILL, 2008) (herein referred to as Master Project Plans), which are referenced throughout this document.

Site Background and Setting

General background information for the Base, including location, topography, geology, and Comprehensive Environmental Response, Compensation and Liability Act (CERCLA)-related history is presented in the Master Project Plans and is not repeated herein. Site-specific background information regarding IR Site 28 and the proposed French Creek Road Extension is presented below.

2.1 French Creek Road Extension Location and Description

Figure 2-1 illustrates the location of the French Creek Road Extension within MCB Camp Lejeune. The proposed road is currently planned to be constructed approximately 3,000 feet north of Julian C. Smith Road between O Street and Gonzalez Boulevard (**Figure 2-2**). The proposed road will bisect a portion of Site 28. Approximately 2,550 feet of the proposed road lies within an undeveloped, wooded area. The southern 450 feet of the proposed roadway extends into a cleared area within Site 28. The area surrounding the French Creek Road Extension is primarily used for recreation and physical training exercises.

Figure 2-3 shows that the ground surface within the western portion of the project area slopes from the central portion toward the New River and Cogdels Creek, while the topography within the eastern portion of the proposed construction area is generally flat with a slight slope toward Cogdels Creek.

2.2 Site 28 History and Previous Investigations

2.2.1 Operational Use

Site 28, located within Operable Unit (OU) 7, operated from 1946 to 1971 as a burn area for a variety of solid wastes generated on the Base. Industrial waste, trash, oil-based paint, and construction debris were reportedly burned and then covered with soil. In 1971, the burn dump ceased operations and was graded and seeded with grass. The total volume of fill within the dump is estimated to be between 185,000 and 375,000 cubic yards. This estimate was based on a surface area of 23 acres and a depth ranging from 5 to 10 feet.

2.2.2 Previous Investigations

Several phases of investigation have been conducted for Site 28 including an Initial Assessment Study (IAS), Confirmatory Study (CS), Remedial Investigation (RI), and Feasibility Study (FS). A Record of Decision (ROD) was issued in 1995 for OU7, specifying Long Term Monitoring (LTM) and Land Use Controls (LUCs) as the remedial action. Additional details on these activities are included in the following subsections. The information presented in these subsections focuses on groundwater and soil investigation due to the proposed road location. Please see the referenced reports for information on surface water, sediment, and bioassay samples.

Initial Assessment Study

Site 28 was first identified in the Base Wide IAS, conducted by the Naval Energy and Environmental Support Activity in 1983 as requiring additional investigation due the past disposal history and the potential to impact Cogdels Creek and the New River.

Confirmatory Study

A two part Confirmation Study (CS) was conducted by ESE from 1984 to 1987 to investigate potential contaminant source areas identified in the IAS report. Site 28 was evaluated during the CS and was determined to warrant further investigation. Groundwater samples were collected from four shallow monitoring wells in 1984 and 1986 (28GW01, 28GW02, 28GW03, and 28GW04). The samples were analyzed for VOCs, metals, pesticides, polychlorinated biphenyls (PCBs), tetrachlorodioxin, and oil and grease.

Table 11-2 in **Appendix A** summarizes the groundwater data collected during the CS. The sampling indicated the presence of VOCs, pesticides, and a number of metals.

Concentrations of metals in groundwater generally decreased from the 1984 sampling round to the 1986 sampling round. Lead was the only metal detected at concentrations exceeding regulatory limits in shallow groundwater samples collected during both the 1984 and 1986 investigations.

Concentrations of VOCs were detected in groundwater samples collected from monitoring well 28-MW01 during the 1984 and 1986 groundwater sampling events. Specifically, ground water sample 28-GW01 contained concentrations of trichloroethylene (TCE) and vinyl chloride that exceeded regulatory limits. VOCs were not detected in groundwater samples collected from the other three wells. None of the other analyzed compounds were detected above regulatory limits during either sampling event.

Additional Investigation

A third round of groundwater samples were collected by Baker in April 1993 to support RI scoping activities. The four existing shallow wells were sampled for TCL organics and TAL total metals.

Results of this sampling event indicated concentrations of vinyl chloride and metals (beryllium, cadmium, chromium, lead, and mercury) above the NCGWQS. **Table 11-4** in **Appendix A** summarizes the groundwater data collected during this sampling round.

Remedial Investigation

A Remedial Investigation (RI) was completed for Site 28 by Baker in 1995. As part of the RI, soil, groundwater, surface water, sediment, and aquatic investigations were conducted.

Based on the results of the RI, depth to groundwater near Site 28 ranges from approximately 3 to 6 feet below ground surface (bgs). Groundwater elevation contour maps were generated for the shallow and Castle Hayne aquifers, as shown on Figures 13-8 and 13-9 in **Appendix A**, respectively. Based on Figure 13-8, shallow groundwater flow appeared to be bi-directional at the site; flowing east-southeast toward Cogdels Creek from the western portion of the site and flowing west-southwest toward Cogdels Creek from the eastern portion of the site. The directions of shallow groundwater flow are shown on **Figure 2-5**.

The direction of groundwater flow in the deeper Castle Hayne aquifer appeared to be southwest toward the New River.

A summary of the analytical detections for surface soils, subsurface soils, and groundwater is provided in **Appendix A** on Figures 14-1 through 14-4. **Figure 2-4** depicts the locations of previous surface and subsurface soil samples with the detected compound classes highlighted. Similarly, **Figure 2-5** shows the locations of previous groundwater samples with the detected compound classes highlighted.

Potential non-carcinogenic and carcinogenic risks to the future residential child and adult receptors upon exposure to arsenic and manganese in shallow groundwater were identified. No potential risks were identified from exposure to surface and subsurface soil at the site for military and construction worker receptors.

Feasibility Study

A FS for Site 28 was completed in 1995 (Baker, 1995A), and evaluated two alternatives: No Action and Institutional Controls.

Record of Decision

The final ROD for OU 7 was signed in 1995. The primary objective of the remedial action at Site 28 was to address lead and manganese contamination in the shallow groundwater aquifer. The ROD identified long-term monitoring (LTM) and land use controls (LUCs) as the selected remedy for groundwater.

Long Term Monitoring Program

LTM (groundwater, surface water, and sediment sampling) at Site 28 was implemented in 1996. In 2001, one shallow monitoring well was installed in the area of the highest lead concentrations observed in soil found during the RI (IR28-MW09). Results from soil and groundwater sampling indicated lead concentrations in both media, but below the levels detected during the RI. Lead was found to be present at naturally high levels due to natural soil conditions.

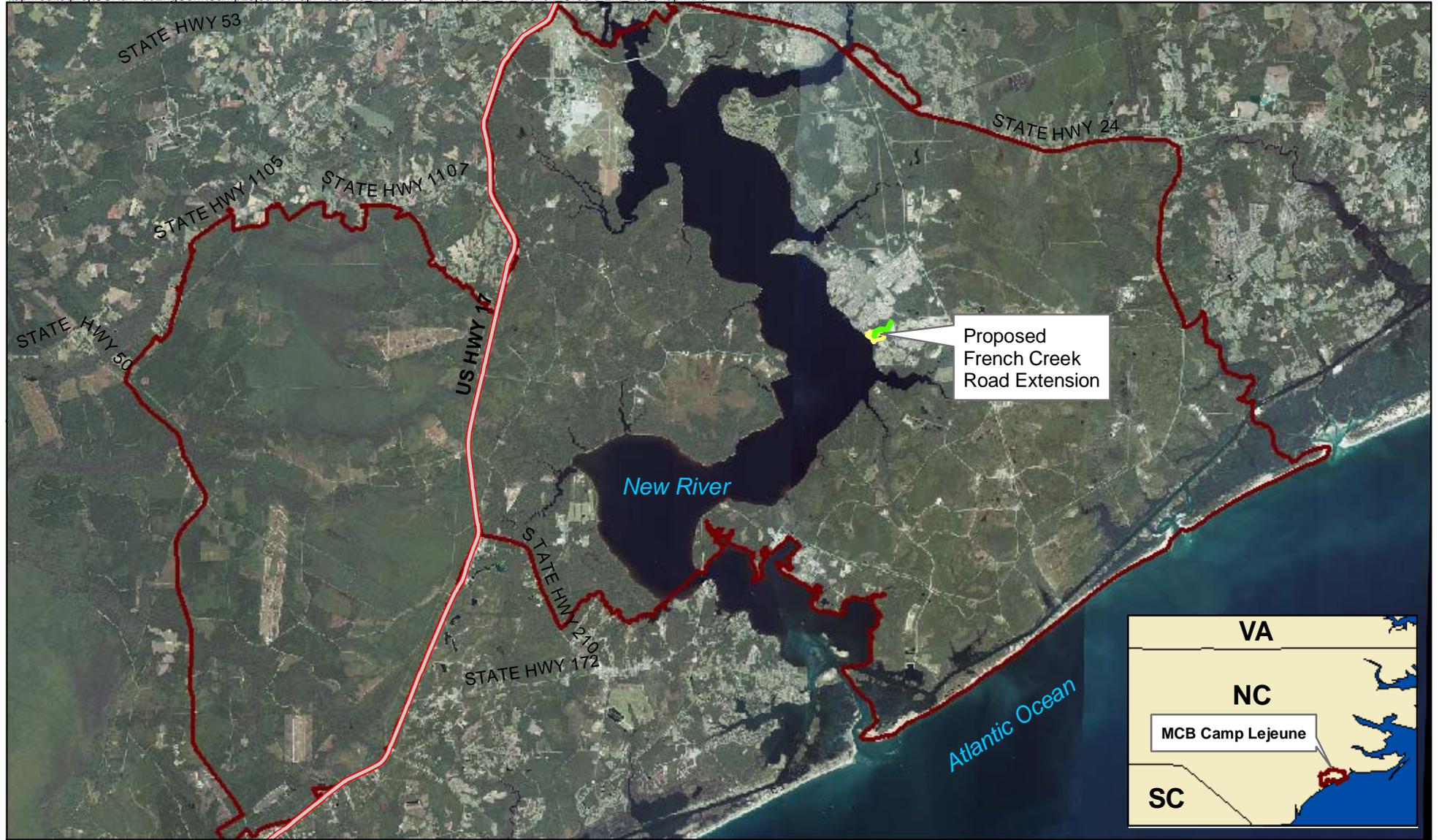
The LTM program at Site 28 was discontinued in October 2001 when site-wide concentrations decreased below the remedial action goals. A Final OU 7 Remedial Action Close-out Report (RACR) was completed in September 2002 (CH2M HILL-Baker, 2002) to document the completion of the remedial action.

Groundwater analytical results from samples collected during LTM are summarized on Table 28-2 included in **Appendix A**.

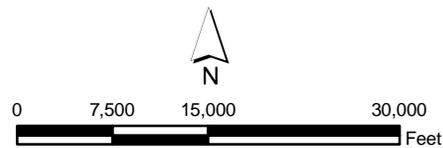
LUCIP

The Land Use Controls Implementation Plan (LUCIP), finalized for OU 7 (Site 1 and Site 28) in March 2000 and updated in July 2002, specified that the selected remedy for OU 7 include LUCs. Specifically, unless approved by both NCDENR and USEPA, land use for non-industrial purposes within these areas of concern is prohibited. These controls are to remain in effect until it can be demonstrated that contaminants no longer remain on site. **Figure 2-6** shows the boundary for non-industrial land use controls.

Additionally, the LUCIP specifies that all use of groundwater within a 1,000 ft buffer surrounding known areas of groundwater contamination at OU 7 is prohibited. Any activities which may impact the areas of known groundwater contamination are prohibited unless specifically approved by NCDENR and USEPA. These controls are to remain in effect until it can be demonstrated that contaminants no longer remain on site. However, because LTM was discontinued and the lead concentrations appear to be naturally occurring, these LUCs will be recommended for removal as part of the next five-year review. The boundaries for aquifer use controls can be found in **Figure 2-7**.



- Legend**
-  Highways
 -  Proposed French Creek Road Extension
 -  Site 28
 -  Installation Boundary



1 inch equals 15,000 feet

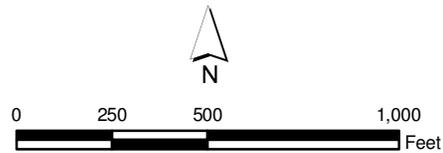
Figure 2-1
Location Map
French Creek Road Extension
MCB Camp Lejeune
North Carolina





Legend

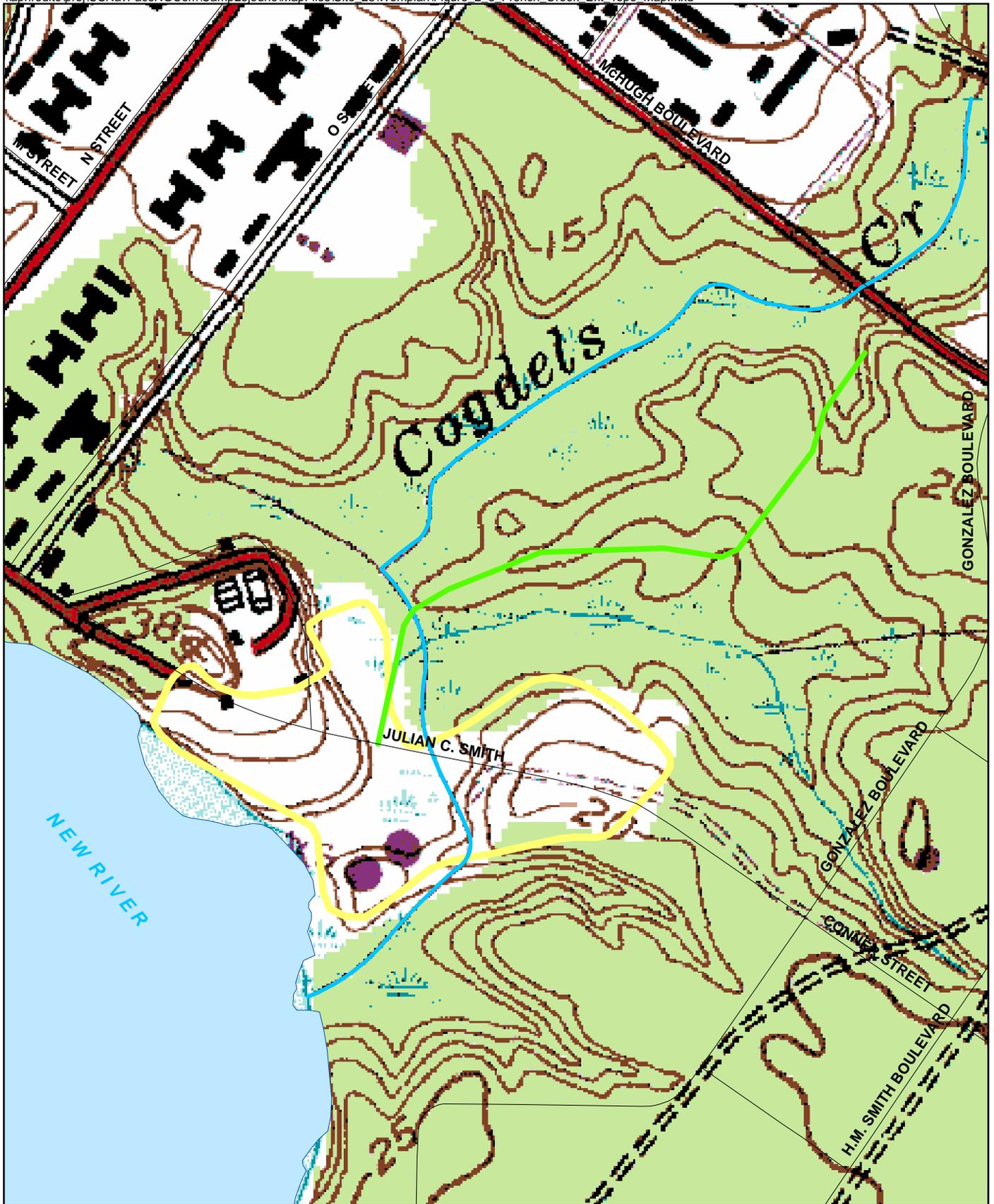
- Proposed French Creek Road Extension
- IR Site 28



1 inch equals 500 feet

Figure 2-2
Site Map
French Creek Road Extension
MCB Camp Lejeune
North Carolina





Legend

-  Proposed French Creek Road Extension
-  Surface Water Body
-  Road Centerline
-  Topographic Contour Lines
-  Site 28
-  Wetlands

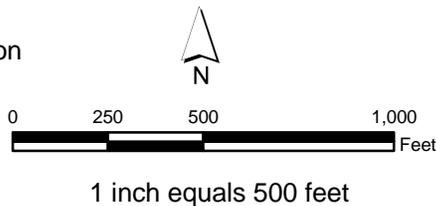


Figure 2-3
Topographic Map
French Creek Road Extension
MCB Camp Lejeune
North Carolina



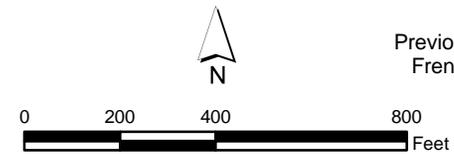


- Legend**
- Previous Soil Sample Locations**
- SVOC Detected
 - Pesticides Detected
 - SVOC and Pesticides Detected
 - VOC and Pesticides Detected
 - VOC, SVOC, and Pesticides Detected

- Proposed French Creek Road Extension
- IR Site 28

Color denotes type of compound previously detected

Note:
Previous soil sample locations and detections based on Baker Environmental's 1995 Remedial Investigation



1 inch equals 400 feet

Figure 2-4
Previous 1995 Soil Investigation
French Creek Road Extension
MCB Camp Lejeune
North Carolina





Groundwater Sample Locations and Compound Detections Based On Baker Environmental's 1995 Remedial Investigation

Legend

Previous Groundwater Sample Locations

- Volatiles Semivolatiles Pesticides/PCBs
- Semivolatiles Pesticides/PCBs
- Pesticides/PCBs

Color Denotes Type of Compound(s) Previously Detected

- Semivolatiles
- Not Sampled
- Non-Detect
- Groundwater Sample
- IR Site 28

- Proposed French Creek Road Extension
- Surface Water
- Estimated Direction of Shallow Groundwater Flow (Baker, 1995)

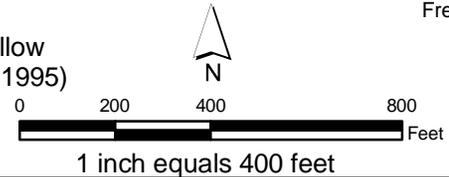


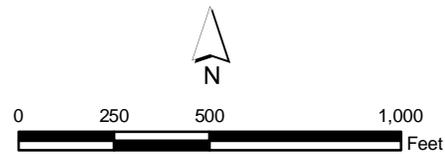
Figure 2-5
Previous 1995 Groundwater Investigation
French Creek Road Extension
MCB Camp Lejeune
North Carolina





Legend

- Proposed French Creek Road Extension
- Boundary of Non-Industrial Land Use Controls
- IR Site 28



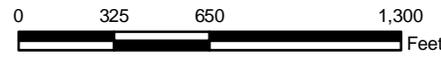
1 inch equals 500 feet

Figure 2-6
Boundary of Land Use Controls
French Creek Road Extension
MCB Camp Lejeune
North Carolina



Legend

- Proposed French Creek Road Extension
- Aquifer Use Control Boundary
- IR Site 28



1 inch equals 650 feet

Figure 2-7
Boundary of Aquifer Controls
French Creek Road Extension
MCB Camp Lejeune
North Carolina

Source: Land Use Controls Implementation Plan,
Operable Unit No. 7 (Sites 1 and 28), July 23, 2002.



Field Activities

Based upon the available information for the French Creek Road Extension, CH2M HILL has developed an approach to investigate the potential environmental impacts to soil and groundwater. Although no significant contamination was identified during previous investigations at Site 28, no samples were collected in the area of the proposed road extension. Due to Site 28's former use as a burn dump, soil and groundwater sampling is proposed in the construction area to evaluate the potential for construction workers to be exposed to unacceptable risks during road construction activities.

The location of the proposed road is based on information received from the Navy remediation project manager. If the location of the proposed road extension changes, the sampling approach may be modified to match the revised construction layout. The field activities for the French Creek Road Extension PA/SI investigation will include the following tasks:

- Underbrush clearing
- Buried utility locating
- Soil sampling using direct push technology (DPT)
- Temporary groundwater monitoring well installation
- Groundwater sampling

The following sections present a discussion of the proposed field activities. All field activities will be conducted in accordance with the Standard Operating Procedures (SOPs) provided in the Master Project Plans (CH2M HILL, 2008).

3.1 Records Review

In preparation for development of the PA/SI field investigation approach for the French Creek Road Extension, an ASR for the area was completed in September 2008 and can be found in **Appendix B**. The record review includes all available National Archive, Base, County, and/or City records. No information was found, outside of the Site 28 information presented in Section 2, identifying current or historical site operations, waste types and quantities, regulatory history, past environmental violations, and citizen complaints.

3.2 Buried Utility Locating

CH2M HILL will coordinate with the North Carolina One Call Center, Base personnel, and a professional underground utility locator to identify subsurface structures that may be impacted by intrusive activities implemented during the investigation of the French Creek Road Extension. **Figure 3-1** depicts known utilities near the Site.

3.3 DPT Soil Sampling

The investigation of the French Creek Road Extension will include soil sampling using DPT. Approximately 15 DPT soil borings will be advanced along the proposed French Creek Road Extension to evaluate site-specific lithology, presence of buried wastes, and assess potential impacts relating to historical land use practices.

Ten of the soil borings will be concentrated in the northern portion of Site 28, where environmental impacts are thought to be more likely, based on historical operations. Five soil borings will be advanced directly north of Site 28 in the densely wooded areas where construction activities are anticipated. The DPT sampling locations will be located along the proposed road extension at intervals of approximately 50 ft within and adjacent to Site 28 and intervals of approximately 500 feet outside of these areas, as shown on **Figure 3-2**. The proposed locations of the soil samples are more concentrated within the northern portion of Site 28 boundary, where environmental impacts are more likely to be present based on historical operations. The DPT soil sampling and lithologic characterization will be conducted as described in the Master Project Plans (CH2M HILL, 2008). In addition, any observed waste will be noted.

Continuous soil cores will be collected in disposable acetate sleeves using a DPT macro-core soil sampler. Soil collected from the borings will be screened for the presence of VOCs using a flame-ionization detector (FID), inspected by a CH2M HILL geologist, and described using the Unified Soil Classification System.

At each soil boring location, a surface soil sample will be collected from ground surface to 1 foot below ground surface (bgs). Subsurface soil samples will be collected from an interval between 1 foot bgs and the water table, which is anticipated to be between 3 and 6 feet bgs.

All soil samples submitted to the fixed base laboratory will be analyzed for VOCs, semi-volatile organic compounds (SVOCs), polychlorinated biphenyls (PCBs), pesticides, and target analyte list (TAL) metals (**Table 3-1**). Additionally, all soil samples collected within the boundary of Site 28 will be analyzed for 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD). All samples except the VOCs will be collected from a homogenized soil sample collected at each location and interval. A grab sample will be collected for VOC analysis prior to compositing at each soil sample location.

Once the target depth of each borehole has been reached and all soil samples have been collected, the boreholes will be abandoned per the Master Project Plans (CH2M HILL, 2008).

3.4 Temporary Monitoring Well Installation

Figure 3-2 depicts the proposed locations for ten temporary groundwater monitoring wells located along the proposed French Creek Road Extension.

3.4.1 Basis for Proposed Well Locations and Depths

The proposed temporary monitoring well locations are distributed along the French Creek Road Extension approximately 100 feet apart within the Site 28 boundary and approximately 500 feet apart north of the Site 28 boundary. Review of **Figure 2-5** suggests

that shallow groundwater flow is influenced by Cogdels Creek, and that shallow groundwater may discharge to the creek. As with the DPT soil borings, the proposed locations of the temporary monitoring wells are more concentrated within the northern portion of Site 28 boundary, where environmental impacts are considered to be more likely to be present based on historical operations. The screened interval of each temporary monitoring well will be set to bracket the water table; anticipated to be approximately 3 to 6 feet bgs.

3.4.2 Installation Procedures

Ten temporary groundwater monitoring wells (designated IR28-TW20 through IR28-TW29) will be installed, as shown on **Figure 3-3**. The temporary wells will be constructed using 1-inch inner diameter (ID) polyvinyl chloride (PVC) casing with 10 feet of 0.010-inch factory slotted well screen, and equipped with a pre-packed sand filter. A bentonite seal with a minimum thickness of 1.5 feet will be installed above the screened interval and associated filter pack to prevent surface water from entering the screened interval.

Due the temporary nature of the monitoring wells, a cement-bentonite grout annular seal will not be used to complete the well. If the temporary monitoring wells are to be left installed for longer than 2 weeks, the wells are to have a locking cap.

Each well will be developed by surging and pumping. Surging will be completed manually by running a disposable bailer across the screen in an up and down motion for 10 to 20 minutes to agitate and settle the filter pack material. The well will then be purged using a pump at an aggressive, but sustainable rate until water quality parameters are stabilized.

Within 5 days of construction, the temporary wells will be sampled and surveyed, then removed and the boreholes abandoned using a grout mixture with Portland cement in accordance with NCDENR guidelines.

3.4.3 Monitoring Well Gauging

Water level measurements will be collected from the ten temporary monitoring wells and existing Site 28 monitoring wells. These measurements will be used with the surveyed top-of-casing elevations to develop a site-wide potentiometric surface map.

3.5 Groundwater Sampling

Prior to sampling, the temporary groundwater monitoring wells will be allowed to equilibrate for 1 to 2 days. The wells will be purged and sampled using a peristaltic pump and the low-flow purging/sampling methods presented in the Master SAP, Section 3.11 “Groundwater Sample Collection” (CH2M HILL, 2008).

Groundwater samples will be collected from each monitoring well and analyzed for VOCs, SVOCs, pesticides/PCBs, and TAL total metals (**Table 3-1**).

3.6 Geospatial Information

A portable Global Positioning Satellite (GPS) system will be used to determine the horizontal coordinates for all soil borings and temporary monitoring wells in accordance with the Master SAP (CH2M HILL, 2008).

TABLE 3-1
Sample Analysis Summary - French Creek Road Extension
Preliminary Assessment / Site Inspection
MCB Camp Lejeune, Jacksonville, North Carolina

Sample Type	Boring/Monitoring Well Designation	Soil Samples						Groundwater Samples				
		VOCs (SW846-8260B)	SVOCs (SW846-8270C)	PCBs (SW846-8082)	Pesticides (SW846-8081A)	TAL Metals (SW846-6010B/7471A)	2,3,7,8-TCDD (SW846-8290)	VOCs (SW846-8260B)	SVOCs (SW846-8270C)	PCBs (SW846-8082)	Pesticides (SW846-8081A)	TAL Metals (SW846-6010A/7470A)
DPT Surficial (0-1 ft bgs)	IR28-SS01 through IR28-SS15	15	15	15	15	15	4					
DPT Subsurface (1-5 ft bgs)	IR28-IS01 through IR28-IS15	15	15	15	15	15	4					
Temporary Monitoring Wells	IR20-TW20 through IR28-TW29							10	10	10	10	10
Environmental Samples		30	30	30	30	30	8	10	10	10	10	10
Trip Blanks ⁽¹⁾		3	0	0	0	0	0	2	0	0	0	0
Duplicate Samples ⁽²⁾		3	3	3	3	3	1	1	1	1	1	1
Matrix Spike Samples (MS) ⁽³⁾		2	2	2	2	2	1	1	1	1	1	1
Matrix Spike Duplicate Samples (MSD) ⁽⁴⁾		2	2	2	2	2	1	1	1	1	1	1
Field Blanks ⁽⁵⁾		1	1	1	1	1	1	1	1	1	1	1
Equipment Rinsate Blanks ⁽⁶⁾		7	7	7	7	7	2	2	2	2	2	2
TOTAL SAMPLES		48	45	45	45	45	14	18	16	16	16	16

NOTES

*If applicable

⁽¹⁾ One per cooler containing VOCs

⁽²⁾ One per every 10 field samples

⁽³⁾ One per every group of 20 samples

⁽⁴⁾ One per every group of 20 samples

⁽⁵⁾ One per week

⁽⁶⁾ One per day



Legend

- Water Line
- Wastewater Line
- Storm Sewer Line
- Heat/Cool Line
- Electrical Cable Line
- Proposed French Creek Road Extension
- IR Site 28

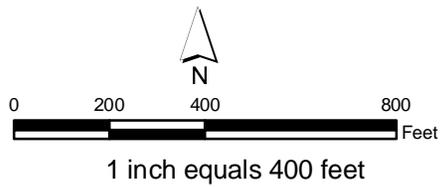


Figure 3-1
Utilities Map
French Creek Road Extension
MCB Camp Lejeune
North Carolina





Legend

-  Surface/Subsurface Soil Samples
-  Proposed French Creek Road Extension
-  IR Site 28

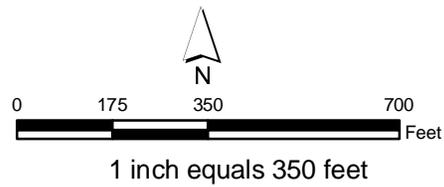


Figure 3-2
 Proposed PA/SI Soil Sample Locations
 French Creek Road Extension
 MCB Camp Lejeune
 North Carolina





- Legend**
- Temporary Wells
 - Proposed French Creek Road Extension
 - IR Site 28

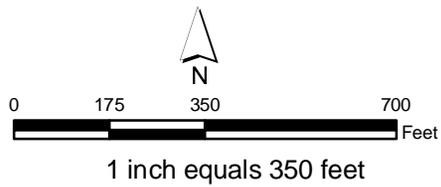


Figure 3-3
Proposed PA/SI Temporary Well Locations
French Creek Road Extension
MCB Camp Lejeune
North Carolina



IDW Management

Wastes generated during the investigation of potentially contaminated sites are classified as investigation derived wastes (IDW) and will be managed to protect the public and the environment. Section 3.17, "Investigation Derived Waste Handling" of the Master SAP provides general information for the characterization, handling, and disposal of contaminated wastes expected to be encountered or generated during this work (CH2M HILL, 2008).

4.1 Waste Streams

The waste streams associated with this scope of work may include:

- Soil cuttings from installation of monitoring wells and soil borings
- Decontamination fluids
- Development and purge water from the monitoring wells
- Personal Protective Equipment (PPE)
- Used sampling supplies
- Uncontaminated general construction debris

4.2 Waste Management

All IDW management actions will be documented in the field notes. Specific waste management procedures are documented in the IDW SOP. Based on historic analytical data, IDW is expected to be non-hazardous.

4.2.1 Decontamination Fluids/Development Water

Decontamination fluids and development water from the temporary monitoring wells will be contained either in drums or in bulk containers that will be transported on Base by the drilling subcontractor. The driller is responsible for transporting all IDW fluids to the wet well located at Lot 203 on Piney Green Road. The CH2M HILL Field Team Leader (FTL) will coordinate liquid disposal activities with the IDW subcontractor. A CH2M HILL representative will accompany the drillers and provide oversight when transferring IDW fluids to Lot 203. Adequate time will be allotted to allow for any solids to settle from the fluids prior to discharging to the wet well.

4.2.2 Soil Cuttings

Soil cuttings will be contained in DOT approved 55-gallon steel drums. If the soil is determined to be non-hazardous, the drilling subcontractor will move the drums to a temporary storage area located on Parachute Tower Road. The containers will be stored there until disposal. In accordance with the Investigation-Derived Waste Management Plan for Camp Lejeune, non-hazardous IDW will be stored for a maximum of 90 days.

Soil IDW is expected to be characterized as non-hazardous. However, if soil IDW is characterized as hazardous, then the drums will be marked with pre-printed hazardous waste labels that include the following information: accumulation start date, generator name, USEPA ID number, applicable waste codes, and the manifest number. The drums will be moved by a licensed hazardous waste transporter to the less-than-90-day storage facility located in Building S-962. Within 90 days from the accumulation start date, the soil will be transported offsite for disposal at a properly permitted RCRA Subtitle C treatment, storage, or disposal facility.

The FTL will coordinate and oversee placement of the IDW in accordance with the Waste Management Plan.

4.2.3 PPE and Trash

PPE associated with the generation of non-hazardous waste will be collected in black, non-translucent trash bags and disposed of in a dumpster aboard MCB Camp Lejeune. PPE associated with the generation of hazardous waste will be properly contained and disposed of at an offsite, permitted, Resource Conservation and Recovery Act (RCRA) Subtitle C treatment, storage, or disposal facility.

Data Management and Evaluation

It is anticipated that data management activities will consist primarily of entering field and laboratory data onto computerized spreadsheets using database software, and tabulating field and analytical results for preparation of the report.

An independent data validator will be subcontracted to validate laboratory analytical data. The analytical results will be evaluated to assess the technical adequacy and usability of the data. Data will be technically reviewed as described in the Master QAPP (CH2M HILL, 2008).

Once the data is received from the laboratory and is validated, an evaluation of the data will be completed. This task involves the evaluation of field-generated data including laboratory analytical data, water level measurements, boring log and well construction records, water quality measurements, and other field notes. Efforts under this task will include the tabulation of validated analytical data and field data, generation of well construction records, and generation of diagrams/figures/tables associated with field notes or data received from the laboratory (e.g., sampling location maps).

The laboratory analytical results will be compared to the North Carolina Groundwater Quality Standards, North Carolina Department of Environmental and Natural Resources (DENR) Soil to Groundwater screening criteria, United States Environmental Protection Agency (USEPA) Regional Screening Levels (RSLs) (adjusted), and base-wide background (metals only).

A human health risk screening will be conducted to assess potential risks to construction workers during road construction. If risks to construction workers are identified, additional delineation and/or soil removal may be required.

SECTION 6

Preliminary Assessment/Site Inspection Report

A PA/SI Report will be prepared following the general format as presented in USEPA's Guidance for Performing Preliminary Assessments Under CERCLA (1991) and USEPA's Guidance for Performing Site Inspections Under CERCLA, Interim Final (1992) and will include:

- A summary of the site-specific environmental setting, including topography, hydrology, geology, and hydrogeology.
- A description of the field investigation activities.
- Assessment of the significance, nature, and extent of hazardous substances.
- Human Health Risk Screening
- Conclusions and recommendations.

A draft report will be issued to allow for a comment period. Any comments received will be addressed in the final version.

SECTION 7

Project Management

CH2M HILL's primary participants for this project are as follows:

- Mr. Matt Louth - Activity Manager
- Ms. Keri Hallberg - Project Manager
- Ms. Monica Fulkerson - Task Manager
- Mr. Tegwyn Williams - Senior Reviewer

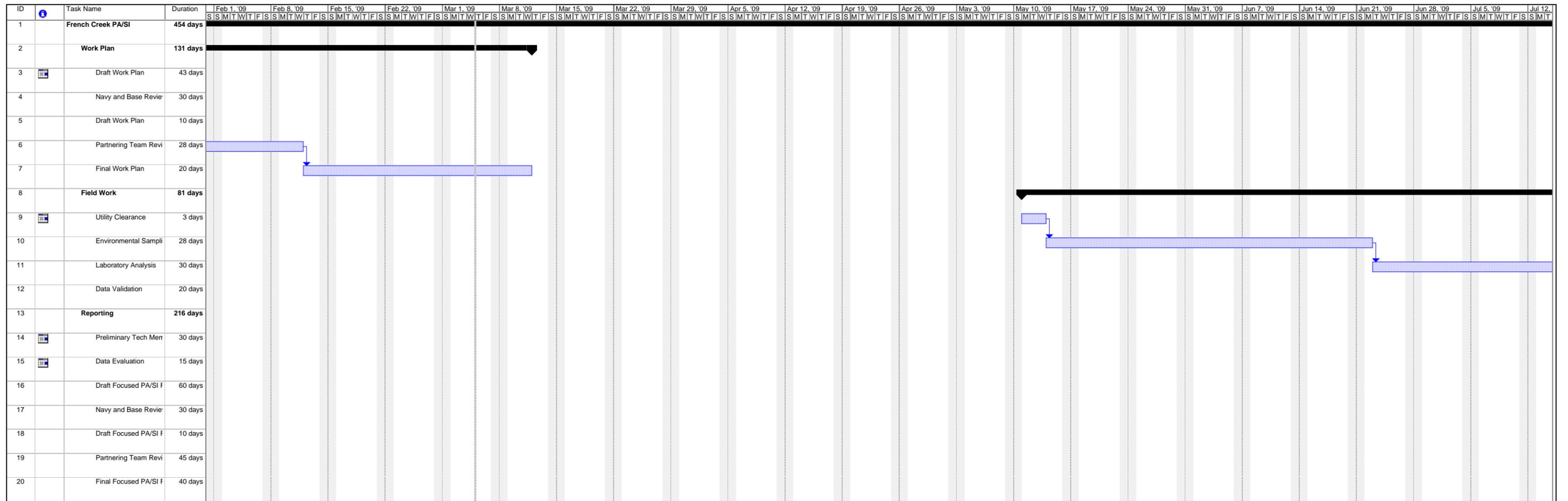
Ms. Hallberg and Ms. Fulkerson will have the overall responsibility for conducting the field activities and completing the reports associated with this PA/SI. Mr. Williams will review the technical aspects of the work from project scoping to project completion. They will be supported by geologists, engineers, scientists, and risk assessors, as needed. Ms. Fulkerson will report to Ms. Hallberg and Mr. Louth who will then relay pertinent issues and maintain close contact with NAVFAC Mid-Atlantic and the Base.

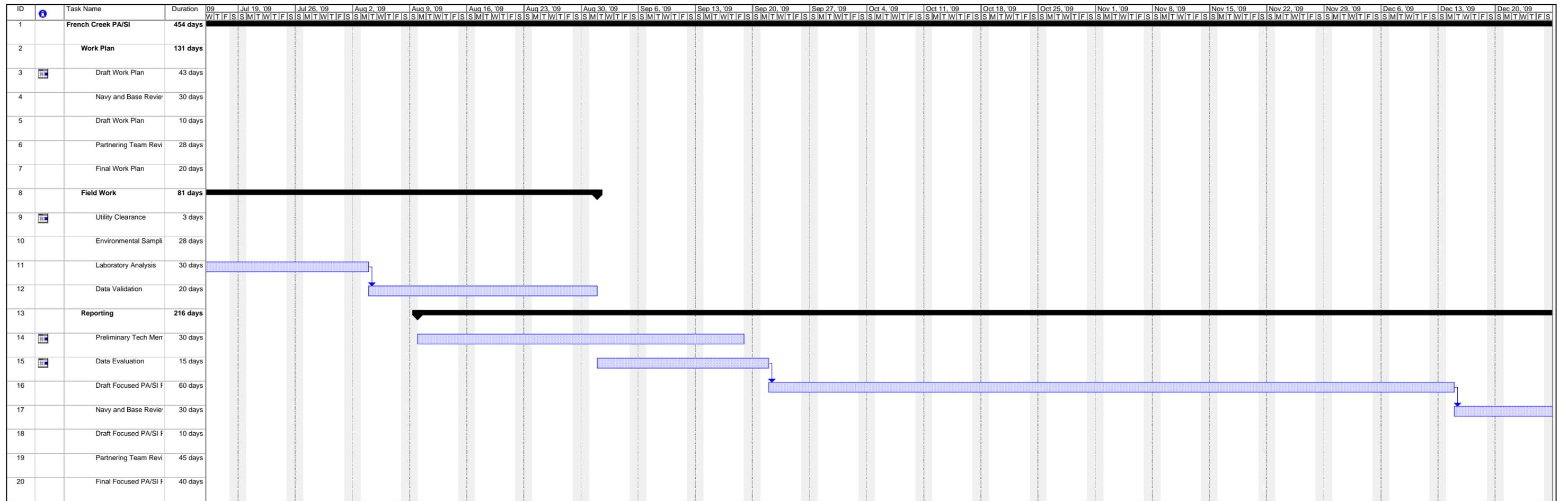
SECTION 8

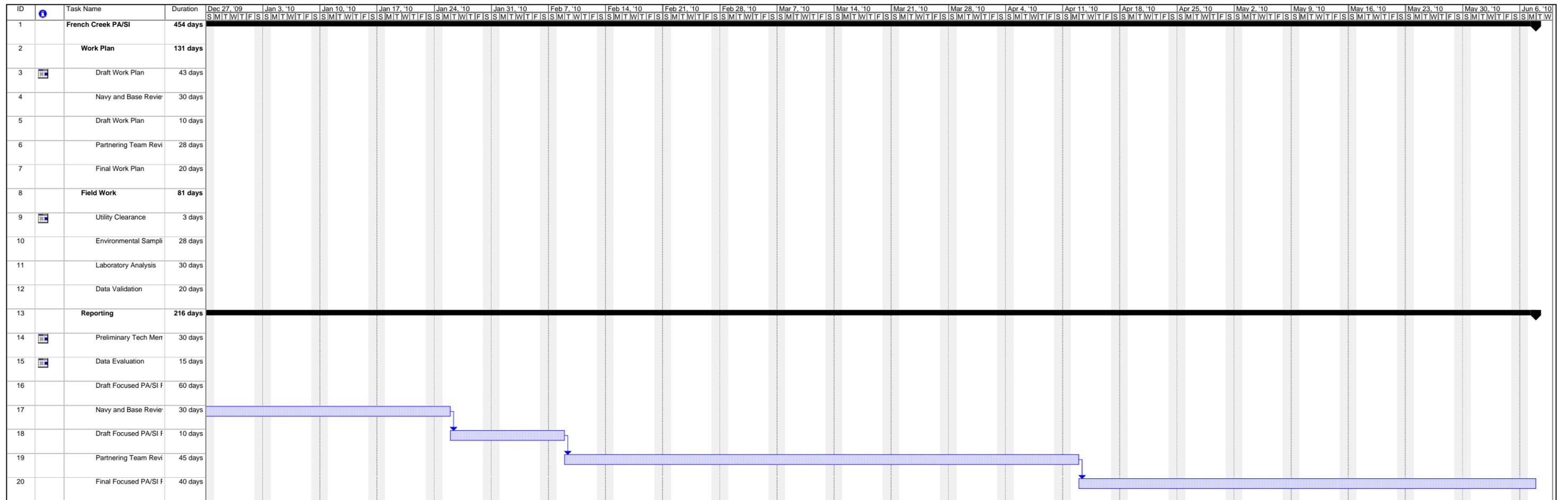
Schedule

The proposed schedule for conducting the PA/SI at French Creek is presented in **Figure 8-1**. The tasks presented in the PA/SI schedule corresponds to the tasks identified in this work plan.

ID	Task Name	Duration	Start	Finish	Predecessors
1	French Creek PA/SI	454 days	Wed 9/10/08	Mon 6/7/10	
2	Work Plan	131 days	Wed 9/10/08	Wed 3/11/09	
3	Draft Work Plan	43 days	Wed 9/10/08	Fri 11/7/08	
4	Navy and Base Review	30 days	Mon 11/10/08	Fri 12/19/08	3
5	Draft Work Plan	10 days	Mon 12/22/08	Fri 1/2/09	4
6	Partnering Team Review	28 days	Mon 1/5/09	Wed 2/11/09	5
7	Final Work Plan	20 days	Thu 2/12/09	Wed 3/11/09	6
8	Field Work	81 days	Mon 5/11/09	Mon 8/31/09	
9	Utility Clearance	3 days	Mon 5/11/09	Wed 5/13/09	
10	Environmental Sampling	28 days	Thu 5/14/09	Mon 6/22/09	9
11	Laboratory Analysis	30 days	Tue 6/23/09	Mon 8/3/09	10
12	Data Validation	20 days	Tue 8/4/09	Mon 8/31/09	11
13	Reporting	216 days	Mon 8/10/09	Mon 6/7/10	
14	Preliminary Technical Memorandum	30 days	Mon 8/10/09	Fri 9/18/09	
15	Data Evaluation	15 days	Tue 9/1/09	Mon 9/21/09	
16	Draft Focused PA/SI	60 days	Tue 9/22/09	Mon 12/14/09	15
17	Navy and Base Review	30 days	Tue 12/15/09	Mon 1/25/10	16
18	Draft Focused PA/SI	10 days	Tue 1/26/10	Mon 2/8/10	17
19	Partnering Team Review	45 days	Tue 2/9/10	Mon 4/12/10	18
20	Final Focused PA/SI	40 days	Tue 4/13/10	Mon 6/7/10	19







References

Baker Environmental, Inc. 1995. *Final Remedial Investigation Report, Operable Unit No. 7, Sites 1, 28, and 30, Marine Corps Base, Camp Lejeune, North Carolina*. Prepared for Department of the Navy, Atlantic Division, Naval Facilities Engineering Command.

Baker Environmental, Inc. 1995A. *Final Feasibility Study Report, Operable Unit No. 7, Site 28, Marine Corps Base, Camp Lejeune, North Carolina*. Prepared for Department of the Navy, Atlantic Division, Naval Facilities Engineering Command.

Baker Environmental, Inc. 2000. *Land Use Controls Implementation Plan (LUCIP), Operable Unit No. 7, Sites 1, 28, and 30, Marine Corps Base, Camp Lejeune, North Carolina*. Prepared for Department of the Navy, Atlantic Division, Naval Facilities Engineering Command.

Baker Environmental, Inc. 2002. *Land Use Controls Implementation Plan (LUCIP) Update, Operable Unit No. 7, Sites 1, 28, and 30, Marine Corps Base, Camp Lejeune, North Carolina*. Prepared for Department of the Navy, Atlantic Division, Naval Facilities Engineering Command.

CH2M HILL, 2008. *Final Master Project Plans, Marine Corps Base Camp Lejeune, Jacksonville, North Carolina, 2008*.

CH2M HILL-Baker. 2002. *Final Close Out Report, Operable Unit 7, Sites 1 and 28, Marine Corps Base Camp Lejeune, Jacksonville, NC, 2002*.

Environmental Science and Engineering (ESE). 1990. *Site Summary Report Final, Marine Corps Base, Camp Lejeune, North Carolina*. Prepared for the department of the Navy, Atlantic Division, Naval Facilities Engineering Command. ESE Project No.49-02036.

United States Environmental Protection Agency. 1991. *Guidance for Performing Preliminary Assessments Under CERCLA, 1991*.

United States Environmental Protection Agency. 1992. *Guidance for Performing Site Inspections Under CERCLA, Interim Final, 1992*.

United States Environmental Protection Agency. 1996. *Record of Decision, Camp Lejeune Military Reservation (US Navy)*.

Water and Air Research. 1983. Water and Air Research, Inc. *Initial Assessment Study of Marine Corps Base, Camp Lejeune, North Carolina*. Prepared for Naval Energy and Environmental Support Activity.

Appendix A
Remedial Investigation and Final Closeout
Report Tables and Figures

TABLE 11-2

**DETECTED TARGET CONTAMINANTS IN GROUNDWATER
CONFIRMATION STUDY
SITE 28, HADNOT POINT BURN DUMP
MCB, CAMP LEJEUNE, NORTH CAROLINA**

Parameter	Federal MCLs ⁽¹⁾	North Carolina WQS ⁽²⁾	Well No./Date							
			28-GW01 7/7/84	28-GW01 12/16/86	28-GW02 7/7/84	28-GW02 12/16/86	28-GW03 7/7/84	28-GW03 12/11/86	28-GW04 12/11/86	28-GW04 3/4/87
Trans-1,2-Dichloroethene	100	70	38	14	ND	ND	ND	ND	ND	ND
Trichloroethene	5	2.8	15	4.9	ND	ND	ND	ND	ND	ND
Vinyl Chloride	2	0.015	22	13	ND	ND	ND	ND	ND	ND
DDD, p-p'	None	None	0.12	ND	0.093	0.018	0.22	ND	ND	ND
DDE, p-p'	None	None	0.015	ND	0.028	ND	0.007	ND	ND	ND
Dieldrin	None	None	0.003	ND	ND	ND	ND	ND	ND	ND
Oil & Grease	None	None	5	8	2	0.4	0.8	ND	ND	9
Arsenic	50	50	18	9.5	ND	ND	21	INTF	INTF	12.1
Chromium (total)	100	50	ND	12	ND	ND	330	15.8	92.6	54
Chromium (+6)	None	None	NA	ND	NA	ND	NA	ND	46.4	ND
Lead	15 ⁽³⁾	15	ND	140	ND	38	336	ND	ND	ND
Mercury	2	1.1	0.3	0.2	ND	0.3	ND	0.8	0.7	0.5
Nickel	100	150	ND	ND	ND	ND	39	ND	43.1	16
Zinc	None	2,100	ND	58	ND	39	143	12.3	142	77

INTF = Interference

NA = Not Analyzed

ND = Not Detected

Values reported are concentrations in micrograms per liter ($\mu\text{g/L}$); this approximates parts per billion (ppb).

Source: ESE, 1992.

⁽¹⁾ Federal maximum contaminant levels (MCLs) established under the Safe Drinking Water Act of 1986.

⁽²⁾ NCWQS - North Carolina administrative code, Title 15A, NC DEHNR, Subchapter 2L, Section .0202 - Water Quality Standards (WQS) for groundwater, November 8, 1993. Class GA Standards.

⁽³⁾ Federal action level established under the Safe Drinking Water Act of 1986.

TABLE 11-4

**INORGANIC CONTAMINANTS IN GROUNDWATER
REMEDIAL INVESTIGATION SCOPING
SITE 28, HADNOT POINT BURN DUMP
MCB, CAMP LEJEUNE, NORTH CAROLINA**

Inorganics	Federal MCL ⁽¹⁾	North Carolina WQS ⁽²⁾	Sample I.D./Date Sampled			
			28-GW01 04/14/93	28-GW02 04/14/93	28-GW03 04/14/93	28-GW04 04/14/93
Aluminum	None	None	16,600	3,280	84,200	43,300
Antimony	6	None	22.0 R	22.0 R	22.0 R	22.0 R
Arsenic	50	50	13.0 J	5.4 J	7.2 J	7.4 J
Barium	2,000	2,000	78.8	556	494	576
Beryllium	4	None	1.2 J	1.0 UJ	1.8 J	9.3 J
Cadmium	5	5	3.0 UJ	17.3 J	3.0 UJ	3.3 J
Calcium	None	None	99,800	53,000	20,200	160,000
Chromium	100	50	39.1 J	9.0 J	140	122
Cobalt	None	None	3.0 U	3.0 U	3.0 U	29.3
Copper	1,300	1,000	19.8	75.4	18.8 J	20.7 J
Iron	None	3,000	15,200	16,000	65,200	35,300
Lead	15 ⁽³⁾	15	234J	197 J	20.3 J	22.4 J
Magnesium	None	None	11,900	26,300	6,020	11,500
Manganese	None	50	138	304	82.2	206
Mercury	2	1.1	0.71 U	1.4 J	0.84 U	0.58 U
Nickel	100	100	17.0 U	17.0 U	17.0 U	59.8
Potassium	None	None	17,800	44,900	5,790	4,810
Selenium	50	50	2.5 UJ	2.4 UJ	2.4 U	10.0 UJ
Silver	None	18	3.0 UJ	3.0 UJ	3.0 UJ	3.0 UJ
Sodium	None	None	33,600	74,400	9,480.0	37,300
Thallium	2	None	3.0 UJ	3.0 UJ	3.0 UJ	3.0 U
Vanadium	None	None	37.7	6.1	164.0	85.3
Zinc	None	2,100	122 U	423 U	40.2 U	390 U
Cyanide	200	154	10.0 U	10.0 U	10 U	10.0 U

Notes: J - Analyte present. Reported value may not be accurate or precise.

U - Not detected above the level reported in laboratory or field blanks.

UJ - The reported quantitation limits are estimated.

R - Unreliable result. Analyte may or may not be present in the sample.

Values reported are concentrations in micrograms per liter ($\mu\text{g/L}$); this approximates parts per billion (ppb).

TABLE 28 - 2
LEAD AND MANGANESE IN GROUNDWATER - SITE 28
OPERABLE UNIT NO. 7 - SITE 28
FINAL CLOSE OUT REPORT, CTO - 0120
MCB, CAMP LEJEUNE, NORTH CAROLINA

Monitoring Well ID	Lead Comparison Criteria			July 1996	Feb 1997	Aug 1997	Jan 1998	July 1998 (1)	Oct 1998	Jan 1999	April 1999	July 1999	Oct 1999 (2)	Jan 2000	April 2000	July 2000	Oct 2000	Jan 2001	April 2001	July 2001	Oct 2001			
	NCWQS	MCL	ROD																					
28-MW01	15	15	15	4.9	1.6	ND	ND	ND	2.9B	ND	2.6B	2.9B	NS	NS	NS	NS	NS	NS	NS	NS	NS			
28-MW02				4.9	ND	ND	ND	ND	ND	ND	ND	ND	1.6B	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	
28-MW07				12.4	6.8	30.6	ND	65	32.5	1B	6.6	34.2	17	4.5	8.7	4.5	41.7	ND	8.3	47.8	131			
28-MW09				NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	ND	ND

Monitoring Well ID	Manganese Comparison Criteria			July 1996	Feb 1997	Aug 1997	Jan 1998	July 1998 (1)	Oct 1998	Jan 1999	April 1999	July 1999	Oct 1999 (2)
	NCWQS	MCL	ROD										
28-MW01	50	NE	50	250	214	66.2	113	114	195	83.2	83.8	72.8	NA
28-MW02				174	185	196	197	204	181	184	169	179	NS
28-MW07				860	460	906	1270	798	787	497	706	740	NS

Notes:

All concentrations are presented in micrograms per liter (µg/L).

- (1) = Confirmatory Sampling Begins.
- (2) = Sampling was discontinued at monitoring wells 28-MW01 and 28-MW02, manganese was also discontinued for analyzation
- B = Reported value is < CRDL, but >IDL.
- NA = Not Applicable
- ND = Not Detected
- NS = Not Sampled
- NE = Not Established

NCWQS = North Carolina Water Quality Standard, 2L.

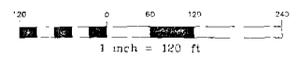
MCL = United States Environmental Protection Agency, Maximum Contaminant Level.

ROD = Record of Decision for Operable Unit No. 7 - Sites 1 and 28 (Baker, May 1996).

Shading indicates that a concentration is above the comparison criteria.



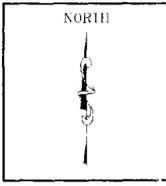
NOTE
 -BOTH ORDE POND AND THE TREATMENT PLANT LAGOON ARE LINED WITH A SEMI-IMPERMEABLE LAYER OF EITHER NATURAL OR SYNTHETIC MATERIAL.



LEGEND	
28GW1	EXISTING MONITORING WELL
28SG02	EXISTING STAFF GAUGE
1.63	GROUNDWATER ELEVATION(WELL SPECIFIC), FEET ABOVE MSL
-3.00	GROUNDWATER ELEVATION CONTOUR LINE, FEET ABOVE MSL
→	APPROXIMATE GROUNDWATER FLOW DIRECTION

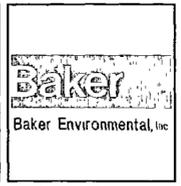
SOURCE: LANTDIV, FEBRUARY 1992

DATE	JANUARY 1995
SCALE	1" = 120'
DRAWN	REL
REVIEWED	TFT
S.O.#	62470-231-0000
CADD#	231126R1



REMEDIAL INVESTIGATION CTO-0231
 MARINE CORPS BASE, CAMP LEJEUNE
 NORTH CAROLINA

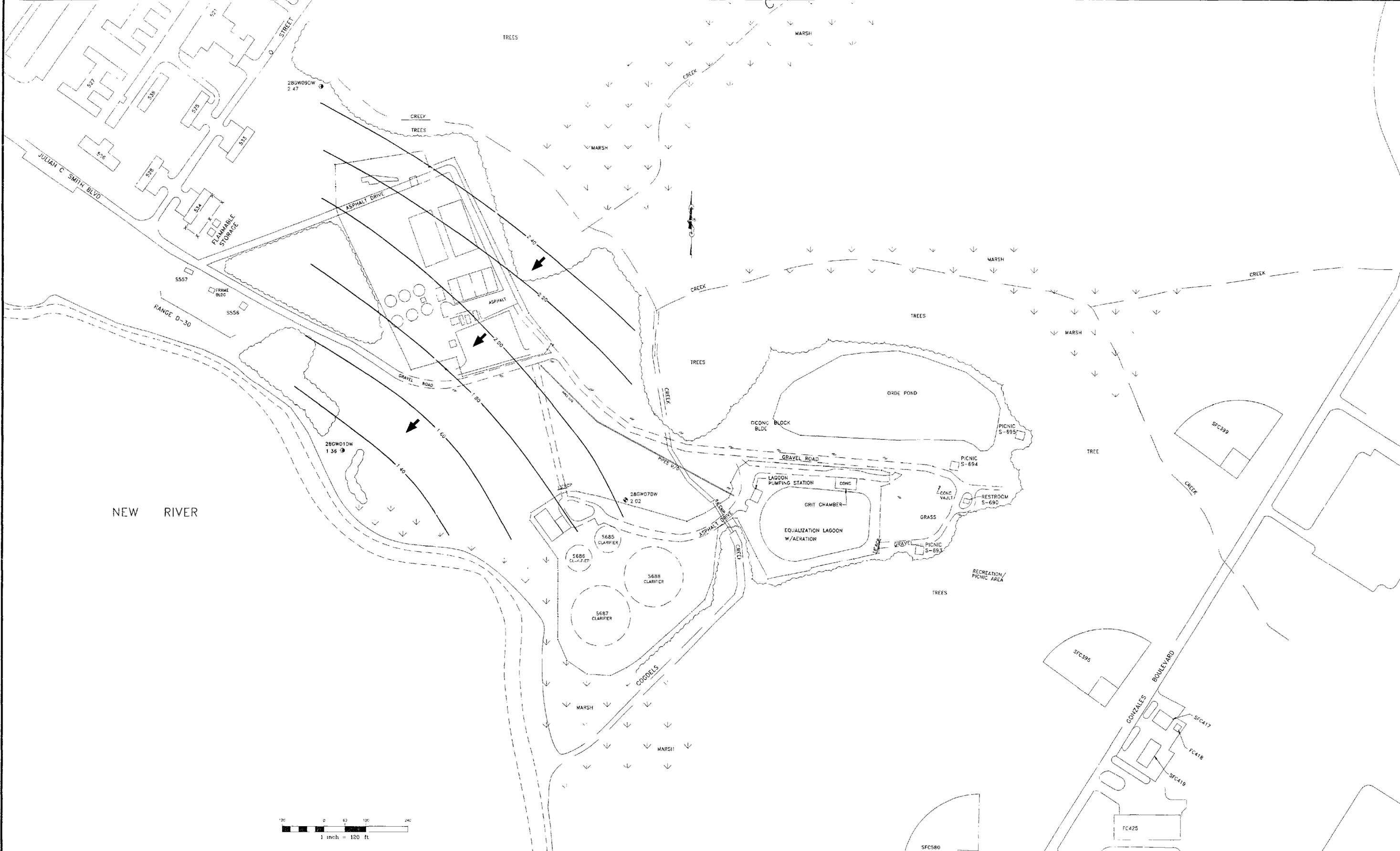
BAKER ENVIRONMENTAL, Inc
 Coraopolis, Pennsylvania



GROUNDWATER CONTOUR MAP FOR THE SURFICIAL AQUIFER - MAY 10, 1994 SITE 28 - HADNOT POINT BURN DUMP	
SCALE:	1" = 120'
DATE:	JANUARY 1995

FIGURE No
 13-8

01500008X



LEGEND

28GW01DW EXISTING MONITORING WELL

1.35 GROUNDWATER ELEVATION (WELL SPECIFIC), FEET ABOVE MSL

1.40 GROUNDWATER ELEVATION CONTOUR LINE, FEET ABOVE MSL

→ APPROXIMATE GROUNDWATER FLOW DIRECTION

SOURCE: LANTRIV, FEBRUARY 1992

DATE: JANUARY 1995

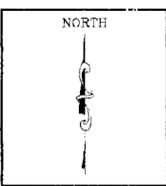
SCALE: 1" = 120'

DRAWN: REL

REVIEWED: TFT

S.O.#: 62470-231-0000

CADD#: 231127R1



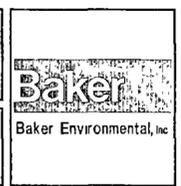
REMEDIAL INVESTIGATION CTO-0231

MARINE CORPS BASE, CAMP LEJEUNE

NORTH CAROLINA

BAKER ENVIRONMENTAL, Inc

Coraopolis, Pennsylvania



GROUNDWATER CONTOUR MAP FOR THE DEEP AQUIFER - MAY 10, 1994

SITE 28 - HADNOT POINT BURN DUMP

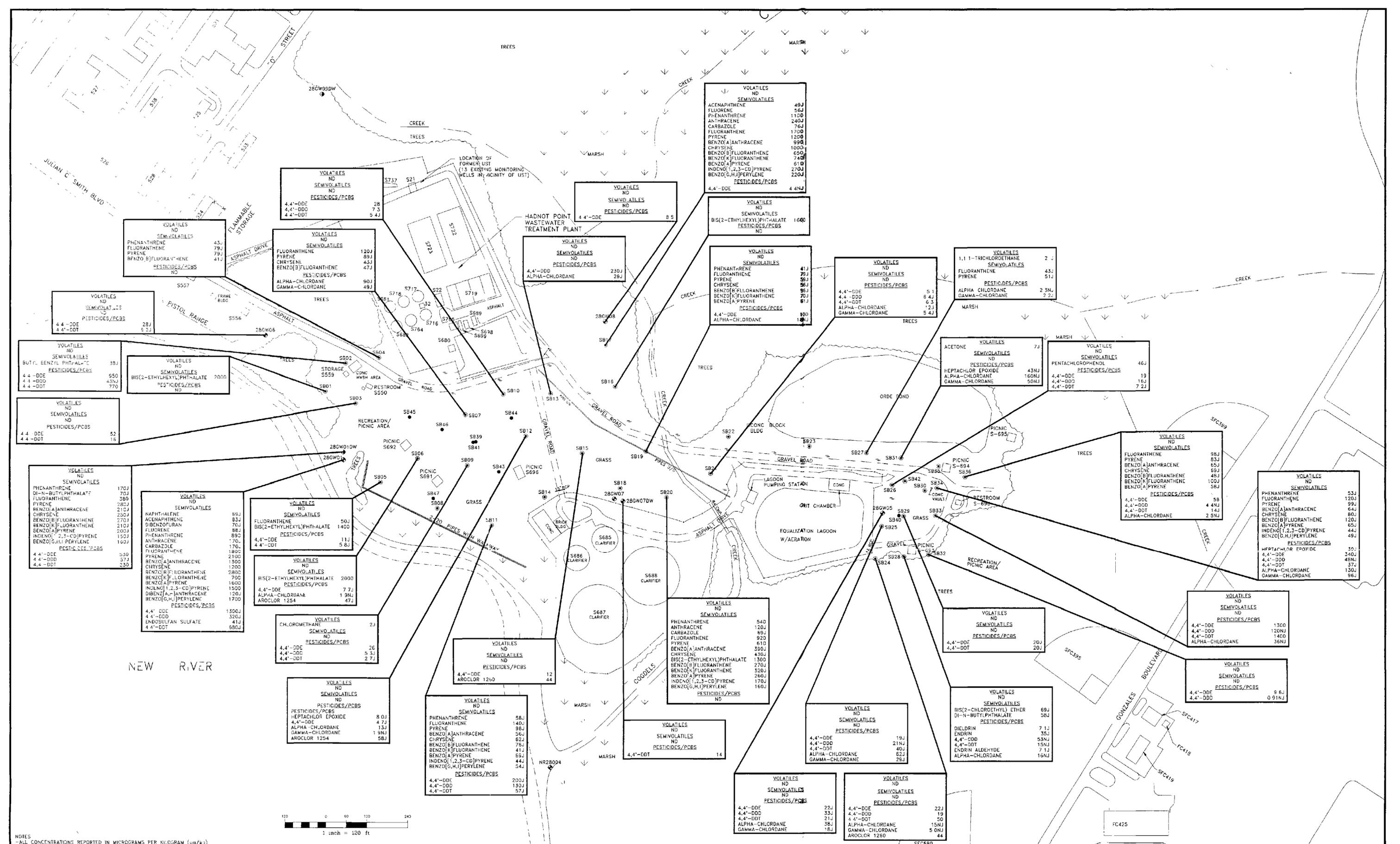
SCALE: 1" = 120'

DATE: JANUARY 1995

FIGURE No

13-9

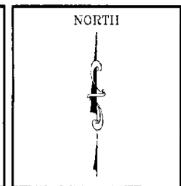
01505Q1EX



NOTES
 - ALL CONCENTRATIONS REPORTED IN MICROGRAMS PER KILOGRAM (ug/kg)
 - BORINGS SHOWN WITHOUT CONCENTRATIONS INDICATE NONDETECTABLE LEVELS
 - ENVIRONMENTAL SAMPLES WERE NOT COLLECTED FROM EXPLORATORY TEST BORINGS

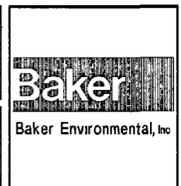
LEGEND	
28GW01	PILOT TEST BORING FOR SHALLOW MONITORING WELL
28GW02/04	PILOT TEST BORING FOR DEEP MONITORING WELL
SB1	SOIL BORING LOCATION
SB40	EXPLORATORY TEST BORING
○	VEGETATION
- x -	FENCE
→	CREEK/DRAINAGE
~	MARSH

DATE	JANUARY 1995
SCALE	1" = 120'
DRAWN	REL
REVIEWED	TFT
S O #	62470-231-0000
CADD#	231152R



REMEDIAL INVESTIGATION CTO-0231
 MARINE CORPS BASE, CAMP LEJEUNE
 NORTH CAROLINA

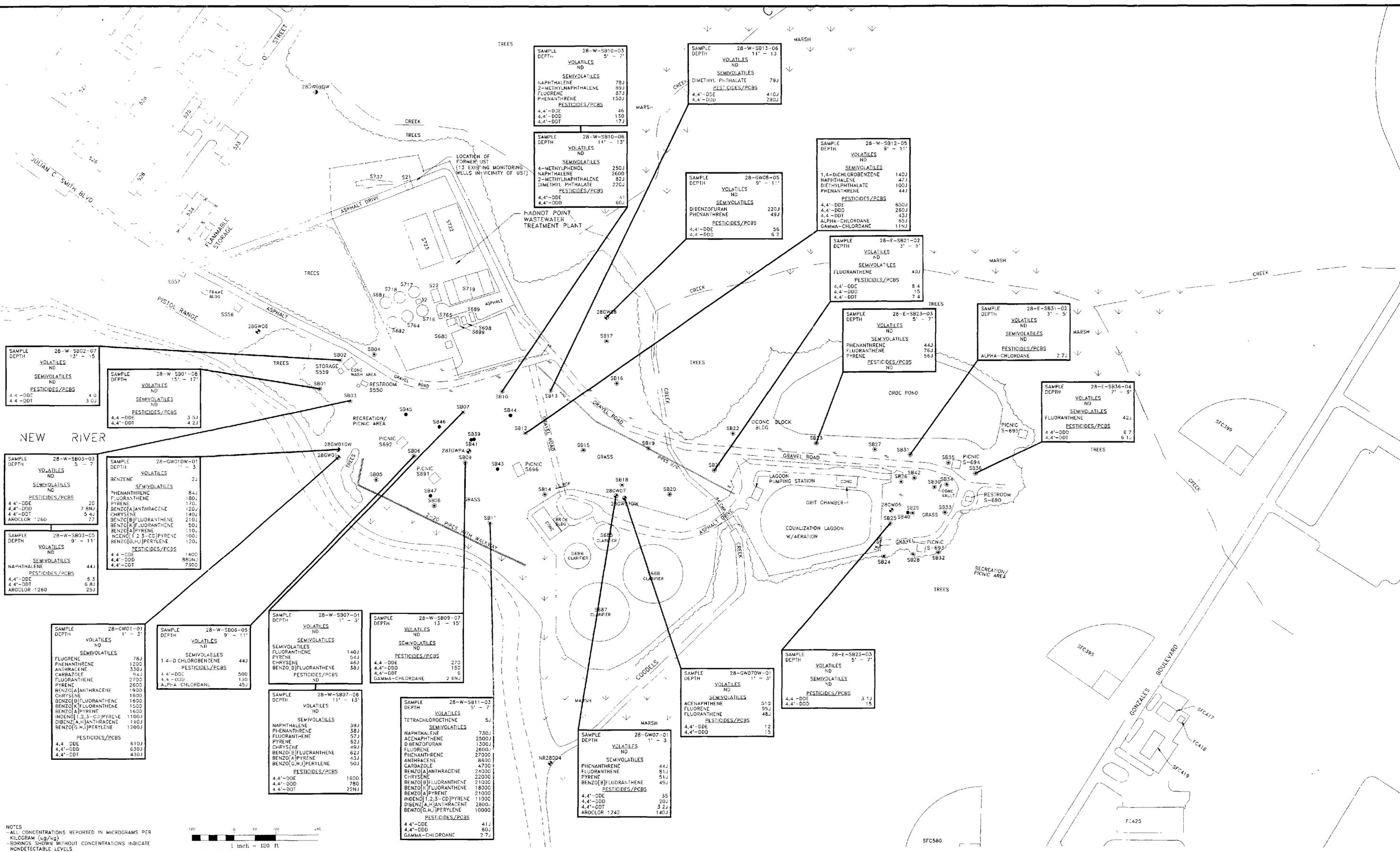
BAKER ENVIRONMENTAL, Inc
 Coraopolis, Pennsylvania



POSITIVE DETECTIONS OF ORGANIC
 COMPOUNDS IN SURFACE SOILS
 SITE 28 - HADNOT POINT BURN DUMP

SCALE 1" = 120'
 DATE JANUARY 1995

FIGURE No
 14-1



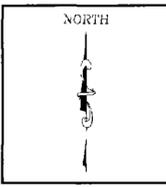
NOTES
 ALL CONCENTRATIONS REPORTED IN MICROGRAMS PER KILOGRAM (µg/kg)
 BORINGS SHOWN WITHOUT CONCENTRATIONS INDICATE NONDETECTABLE LEVELS
 ENVIRONMENTAL SAMPLES WERE NOT COLLECTED FROM EXPLORATORY TEST BORINGS

LEGEND

28GW01	PILOT TEST BORING FOR SHALLOW MONITORING WELL
28GW01DW	PILOT TEST BORING FOR DEEP MONITORING WELL
SB1	SOIL BORING LOCATION
SB40	EXPLORATORY TEST BORING
—	VEGETATION
-X-	FENCE
—	CREEK/DRAINAGE
—	MARSH

SOURCE: LANTDIV, FEBRUARY 1992

DATE	JANUARY 1995
SCALE	1" = 120'
DRAWN	REL
REVIEWED	TFT
S.O.#	62470-231-0000
CADD#	231153R1



REMEDIAL INVESTIGATION CTO-0231
 MARINE CORPS BASE, CAMP LEJEUNE
 NORTH CAROLINA

BAKER ENVIRONMENTAL, Inc
 Coraopolis, Pennsylvania

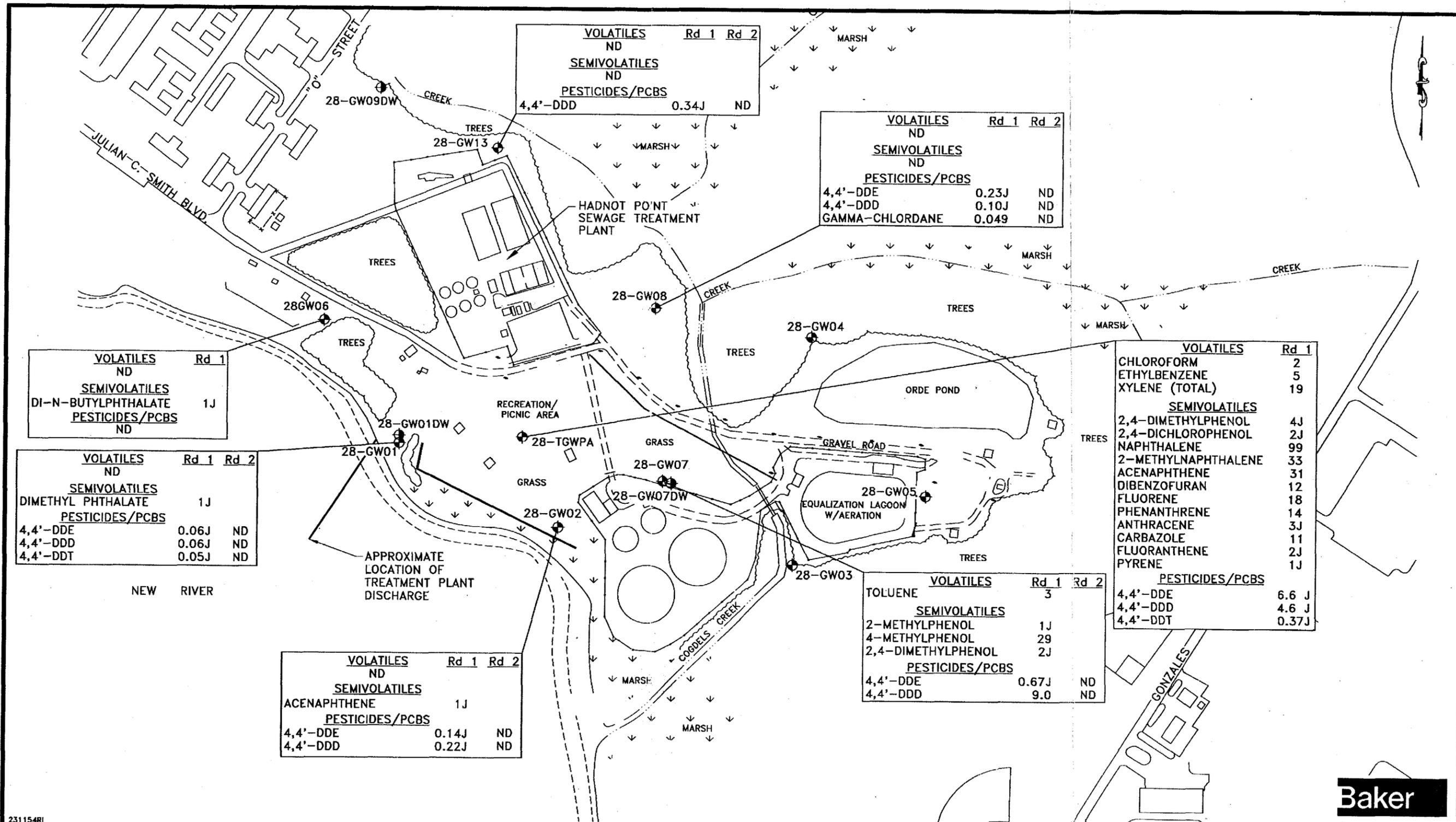


POSITIVE DETECTIONS OF ORGANIC COMPOUNDS IN SUBSURFACE SOILS
 SITE 28 - HADNOT POINT BURN DUMP

SCALE 1" = 120' DATE JANUARY 1995

FIGURE No
14-2

C1500Q17X



231154RI

LEGEND

28-GW01 SHALLOW MONITORING WELL
 28GW01DW DEEP MONITORING WELL

NOTES:
 -SHALLOW MONITORING WELL GW01 WAS ABANDONED AND REPLACED DURING THE RI FIELD INVESTIGATION.
 -ALL CONCENTRATIONS REPORTED IN MICROGRAMS PER LITER (ug/L).
 -WELLS SHOWN WITHOUT CONCENTRATIONS INDICATE NONDETECTABLE LEVELS.

SOURCE: LANTDIV, FEBRUARY 1992 AND W.K. DICKSON, JUNE 1994

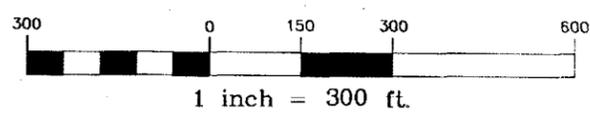
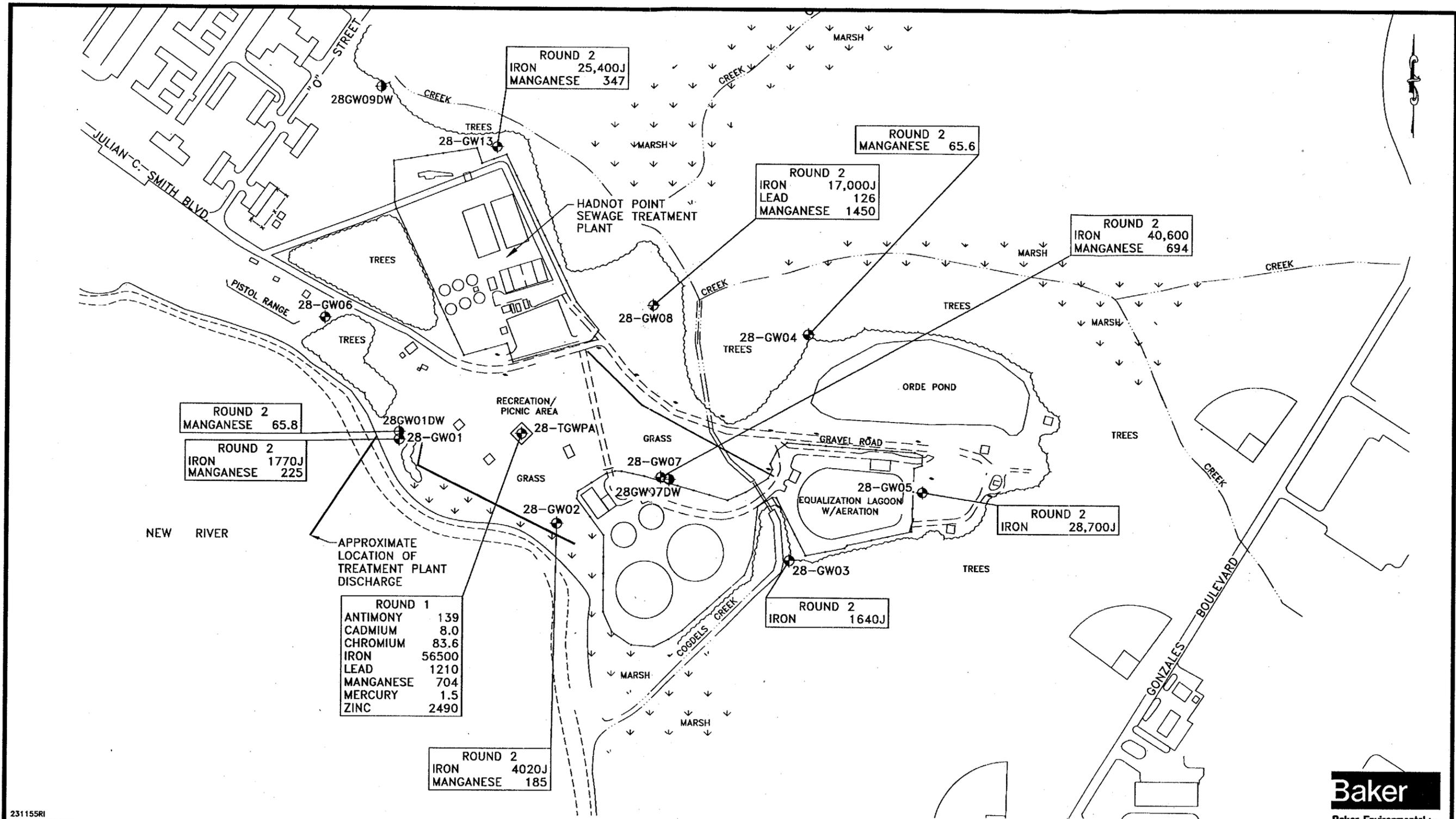


FIGURE 14-3
 POSITIVE DETECTIONS OF ORGANIC COMPOUNDS IN GROUNDWATER
 SITE 28 - HADNOT POINT BURN DUMP
 REMEDIAL INVESTIGATION CTO-0231
 MARINE CORPS BASE, CAMP LEJEUNE
 NORTH CAROLINA



01500Q182



Baker
Baker Environmental, Inc.

231155RI

LEGEND

- 28-GW01 SHALLOW MONITORING WELL
- 28-TGWPA TEMPORARY SHALLOW MONITORING WELL
- 28GW01DW DEEP MONITORING WELL

NOTES:

- ALL CONCENTRATIONS REPORTED IN MICROGRAMS PER LITER (µg/L).
- SHALLOW MONITORING WELL GW01 WAS ABANDONED AND REPLACED DURING THE RI FIELD INVESTIGATION.

SOURCE: LANTDIV, FEBRUARY 1992 AND W.K. DICKSON, JUNE 1994

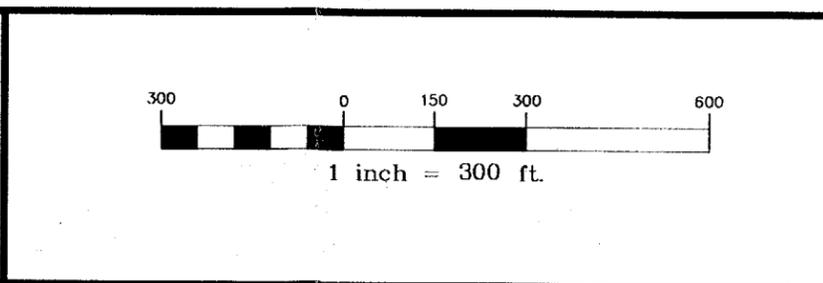


FIGURE 14-4
POSITIVE DETECTIONS OF TAL METALS ABOVE FEDERAL MCL AND/OR NCWQS IN GROUNDWATER
SITE 28 - HADNOT POINT BURN DUMP
REMEDIAL INVESTIGATION CTO-0231
MARINE CORPS BASE, CAMP LEJEUNE
NORTH CAROLINA

01500Q192

Appendix B
Archive Records Search Report

Final

Archival Records Search Report French Creek

Marine Corps Base Camp Lejeune
Jacksonville, North Carolina

Contract Task Order 011

March 2009

Prepared for

Department of the Navy
Naval Facilities Engineering Command
Atlantic

Under the

NAVFAC CLEAN 1000 Program
Contract N62470-08-D-1000

Prepared by



CH2MHILL

Charlotte, North Carolina

Contents

Acronyms and Abbreviations	B-v
B.1 Introduction, Purpose, and Scope.....	B-1-1
B.2 Site Information.....	B-2-1
A.2.1 Ownership and Operational History	B-2-1
A.2.1.1 MCB Camp Lejeune Ownership History	B-2-1
A.2.1.2 French Creek Road Extension Investigation Area	B-2-1
B.3 References	B-3-1

Attachment

1	Resource Review Summary
---	-------------------------

Acronyms and Abbreviations

ASR	Archival Search Report
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CTO	Contract Task Order
DGM	Digital Geophysical Mapping
MC	Munitions Constituents
MCB	Marine Corps Base
MEC	Munitions and Explosives of Concern
mm	millimeter
MRP	Munitions Response Program
NARA	National Archives and Records Administration
USACE	United States Army Corps of Engineers
UXO	Unexploded Ordnance
WWII	World War II

SECTION B.1

Introduction, Purpose, and Scope

Marine Corps Base (MCB) Camp Lejeune is planning an environmental investigation in support of the French Creek Road Extension construction project. The environmental investigation will be conducted under Contract N-62470-08-D-1000, Contract Task Order (CTO)-011.

The results of the environmental investigation will determine if any impacts to soil and groundwater resulting from Installation Restoration (IR) Site 28 or other historical activities will impact construction. To support site investigation effort, this archival records search report has been prepared to provide a narrative of the historical activities in the vicinity of the French Creek Road Extension investigation area that may have resulted in environmental contamination.

The archival records search is an investigative review of existing information about the site and its surrounding area, with an emphasis on obtaining information from personnel and historical resources that might indicate a potentially hazardous release to the environment. The scope of the report includes:

A review of existing information about the site (including MCB Camp Lejeune maps, drawings, and reports, and interviews with MCB Camp Lejeune personnel).

Collection of additional information about the site.

A complete listing of resources identified and investigated for this report is provided in **Attachment 1**. **Attachment 1** also includes details concerning the reviews of the historical information from the Marine Corps Library at Quantico, National Archives and Records Administration (NARA) map and text files, and MCB Camp Lejeune base files.

Site Information

2.1 Ownership and Operational History

2.1.1 MCB Camp Lejeune Ownership History

The history of the land now occupied by MCB Camp Lejeune is documented primarily through land records and maps. Following the start of World War II (WWII), the War Department began purchasing tracts of land in 1941 from local residents to meet the need for an East Coast amphibious training facility. Prior to the Marines occupation, the land had been occupied by white and African-American communities and farms since the Colonial era. The land contained plantation houses, cabins, farm buildings, tobacco barns, stores, and various cemeteries (Global Security Website, 2007).

The initial land transferred to the government was acquired in 14 different transactions between April and October 1941 and totaled 173.8 square miles or 111,155 acres, of which there were 85,155 land acres and about 26,000 acres under water (Loftfield, 1981, Louis Berger Group, 2002). The individual tracts of land were grouped into various 'Areas' for consolidation.

2.1.2 French Creek Road Extension Investigation Area

The French Creek Road Extension investigation area is located between Julian C. Smith Road and McHugh Boulevard in the Hadnot Point area of Camp Lejeune. IR Site 28, the Hadnot Point Burn Dump, is located within the French Creek investigation area along the eastern bank of the New River and is surrounded by the Hadnot Point Sewage Treatment Plant to the north, wooded areas to the east and south, and the New River to the west.

A remedial investigation (RI), feasibility study (FS), Proposed Remedial Action Plan (PRAP), Record of Decision (ROD) and Closeout Report have been completed for IR Site 28. In addition, Land Use Controls (LUCs) are in place. Section 2 of the Work Plan provides additional information regarding these investigations.

The Base Safety Specialist stated that he did not have any information pertaining to the French Creek Road Extension investigation area, but indicated that it was common to burn different types of oils, paints, wood products and other items as a means of disposal (Richardson, 2008).

SECTION B.3

References

Global Security Website, "Camp Lejeune", <http://www.globalsecurity.org/military/facility/camp-lejeune.htm>. Accessed May 29, 2007.

Loftfield, Thomas C., 1981. Principal Investigator, University of North Carolina, Wilmington, *Archeological and Historical Survey of USMC Base, Camp Lejeune*, Naval Facilities Engineering Command Norfolk, Coastal Zone Resource Corp, Vol II, Contract # N62470-79-C-4273, August 1981.

Louis Berger Group Inc., 2002. *Semper Fidelis: A Brief History of Onslow County, North Carolina and MCB, Camp Lejeune, 2002*, U.S.M.C., Lt. Col Lynn J. Kimball (USMC, Ret.), consulting historian.

Richardson, 2008. Personal Communication with Duane Richardson, Camp Lejeune Range Safety Specialist. October 3, 2008.

United States Army Corps of Engineers (USACE), 2001. St. Louis District. *Final Range Identification and Preliminary Range Assessment*, Marine Corps Base Camp Lejeune, Onslow, North Carolina, December 2001.

Attachment 1
Resource Summary Review

Resource Review Summary

The following table provides a summary of the specific references identified for review, interview, or contact for the archival report.

Resource	Actions Completed
Quantico, Virginia, Marine Corps Library, Gray Research Center	Reviewed all available file folders related to Camp Lejeune – Made copies of relevant historic maps. No files to copy.
US National Archives (NARA II) Historical Files	Reviewed text and drawing files from Text and Cartographic Divisions. Made copies of relevant files and maps.
Deborah Edge/National Archives Text File	See US National Archives Files Review
Camp Lejeune Technical Records files	Reviewed and copied all relevant documents related to historical land use for each site.

Camp Lejeune Personnel	
Linda Futrell/ Realty Specialist	Contacted and interviewed
Dennis Dunham/ Technical Records	Contacted and interviewed
Duane Richardson/ Base Range Safety Officer	Contacted and interviewed

Marine Corps Library Review

Text Division

Contact: Gregory Cina

Site Visit: October 7, 2008

File review at Marine Corps Base, Quantico, Virginia, Gray Research Center, Marine Corps Archives and Special Collections.

Several historic maps were digitally copied; however, no pertinent text documents were obtained from the file review.

List of Documents Obtained from Marine Corp Library

- “Camp Lejeune, New River, North Carolina”, August 1943.
- “Combat Training Chart, United States East Coast, North Carolina, Approaches to New River”, December 26, 1987.
- “New River”, 1972.
- “Jacksonville South Quadrangle”, NW/4 New River 15’ Quadrangle, USGS, 1952.
- “North Carolina, Approaches to New River”, November 1950.

National Archives and Records Administration Review

Text Division

Contact: Ms. Deborah Edge, 301-837-1687

Site visits on September 15 - 18, 2008

Reviewed 12 boxes of files associated with the Marine Corps, 1939-1950

- Record Group 127 (USMC), Office of the Commandant, General Correspondence, January 1939-June 1950, 1275/70-800 (10/45-1/47) to 1275/70-727 (1/44-12/47), Box 218.
- Record Group 127 (USMC), Office of the Commandant, General Correspondence, January 1939-June 1950, 1275/70-800 (10/44-1/45) to 1275/70-800 (7/45-9/45), Box 219.
- Record Group 127 (USMC), Office of the Commandant, General Correspondence, January 1939-June 1950, 1275/70-800 (10/44-1/45) to 1275/70-800 (7/45-9/45), Box 220.
- Record Group 127 (USMC), Office of the Commandant, General Correspondence, January 1939-June 1950, 2295-10 Brooklyn to 2285-10 Camp Lejeune, Box 1570.
- Record Group 127 (USMC), Office of the Commandant, General Correspondence, January 1939-June 1950, 2295-10 Camp Lejeune to 2285-10 Camp Lejeune, Box 1571.
- Record Group 127 (USMC), Office of the Commandant, General Correspondence, January 1939-June 1950, 2295-10 Camp Lejeune to 2285-10 Camp Lejeune, Box 1572.
- Record Group 127 (USMC), Quartermaster, General Correspondence, January 1940, 215-3, Box 144.
- Record Group 127 (USMC), Quartermaster, General Correspondence, January 1940, 215-3, Box 145.
- Record Group 127 (USMC), Quartermaster, General Correspondence, January 1940, 215-3, Box 146.
- Record Group 127 (USMC), Quartermaster, General Correspondence, January 1940, 215-3, Box 147.
- Record Group 127 (USMC), Quartermaster, General Correspondence, January 1940, 215-3, Box 148.
- Record Group 127 (USMC), Records of the USMC, Division of Public Information, General Correspondence, 1942- 1950, Box 1 of 1.

The boxes contained information primarily related to basic activities and events occurring at Camp Lejeune, as well as general ordnance orders and supply issues. Several historic maps were found showing the French Creek area.

List of Documents Obtained from National Archives

- "Camp Lejeune General Area Map", February 12, 1942.

- “Camp Lejeune General Area Map”, March 11, 1947.
- “Camp Lejeune, New River, North Carolina”, August 1943.
- “Camp Lejeune, North Carolina”, Vicinity Map, February 1947.
- “Division Training Area, Camp Lejeune, North Carolina”, June 30, 1946.
- “Division Training Area, Camp Lejeune, North Carolina”, June 30, 1947.
- “Fleet Marine Force, 2nd Marine Division, Shop Area, Location and Index Plan”, Vicinity Map, April, 28, 1950.
- “Index Sheet to Accompany Annual Report Maps, Camp Lejeune, North Carolina”, June 30, 1947.
- “Map of Magazine Area, Camp Lejeune, North Carolina”, June 30, 1947.

MCB Camp Lejeune Base Site Visit and Records Review

Base Contact: Ms. Linda Futrell, Public Works Division, 910-451-2818 x3257

File reviews of records in the base Technical Records office were conducted during the site visit. Additionally, interviews were conducted with Dennis Dunham/Technical Records, and Duane Richardson/EOD Base Range Safety Officer.

List of Documents Obtained from Camp Lejeune

Base Real Estate Office

- “Division Training Area, Camp Lejeune, North Carolina”, June 30, 1946.
- “Division Training Area, Camp Lejeune, North Carolina”, June 30, 1952.
- “Existing Conditions: Hadnot Point and French Creek”, Grid G8, December 20, 2004.
- “Existing Conditions: Hadnot Point and French Creek”, Grid H8, December 22, 2004.
- “Existing Conditions: Hadnot Point, French Creek, and Cogdels Creek”, Grid G8, September 2, 2008.
- “Existing Conditions: Hadnot Point, French Creek, and Cogdels Creek”, Grid H8, September 2, 2008.
- “French Creek Areas 300-400-500”, 1985.
- “Hadnot Point Regimental Areas 400-500, Division Shops Area 1800, and French Creek Area”, 1985.
- “Map of Magazine Area, Camp Lejeune, North Carolina”, June 30, 1949.

Base Library

- Louis Berger Group, Inc. Under USCOE, Wilmington District Contract DACWS4-99-C-0004, *Semper Fidelis: A Brief History of Onslow County, North Carolina and MCB, Camp*

Lejeune, 2002, United States Marine Corps, Lt. Col Lynn J. Kimball (USMC, Retired) Consulting Historian.

- Lotfield, Thomas, C. Principal Investigator. UNCW, August 1981. *Archeological and Historical Survey of USMC Base, Camp Lejeune; Naval Facilities Engineering Command Norfolk, Coastal Zone Resource Corp., Vol. II, Contract No. N62470-79-C-4273.*

Appendix C
Field Sampling Plan

Final

**Field Sampling Plan
French Creek Road Extension**

**Marine Corps Base Camp Lejeune
Jacksonville, North Carolina**

Contract Task Order 011

March 2009

Prepared for

**Department of the Navy
Naval Facilities Engineering Command
Atlantic**

Under the

**NAVFAC CLEAN 1000 Program
Contract N62470-08-D-1000**

Prepared by



Charlotte, North Carolina

Contents

C.1	Introduction.....	C-1-1
C.2	Field Activities.....	C-2-1
C.3	Sample Designation.....	C-3-1
C.4	Sample Handling and Analysis.....	C-4-1
	C.4.1 Sample Preservation and Handling.....	C-4-1
	C.4.2 Chain-of-Custody and Field Logbook.....	C-4-1

Tables

C-1	Summary of Samples
C-2	Sample Container, Preservation, and Holding Time

Acronyms and Abbreviations

DPT	Direct Push Technology
EPA	Environmental Protection Agency
FSP	Field Sampling Plan
HSP	Health and Safety Plan
IR	Installation Restoration
MCB	Marine Corps Base
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NAIP	Natural Attenuation Indicator Parameters
ORP	Oxidation/Reduction Potential
PA	Preliminary Assessment
PCB	Polychlorinated Biphenyl
PVC	Polyvinyl Chloride
QA	Quality Assurance
QC	Quality Control
SI	Site Inspection
SVOC	Semivolatile Organic Compound
TAL	Target Analyte List
TCDD	2,3,7,8-tetrachlorodibenzo-p-dioxin
VOC	Volatile Organic Compound

SECTION C.1

Introduction

This document has been prepared to serve as a Field Sampling Plan (FSP) for the Preliminary Assessment/Site Inspection (PA/SI) in the French Creek Road Extension area aboard Marine Corps Base (MCB) Camp Lejeune in Onslow County, North Carolina. This FSP sets forth site specific procedures for field activities and for the analysis of surface soil, subsurface soil, and groundwater samples.

All field activities will be conducted by CH2M HILL or subcontractors under the direct supervision of CH2M HILL. Field activities unless otherwise noted in the FSP will follow the procedures described in the Master Project Plans.

SECTION C.2

Field Activities

The PA/ SI for the French Creek Road Extension will include the following field activities:

- Site clearing
- Buried utility locating
- Surface and subsurface soil sampling using direct push technology (DPT)
- Installation and development of temporary groundwater monitoring wells
- Collection and analysis of groundwater samples
- Surveying

Each activity is described in detail in the French Creek Road Extension Work Plan and Master Project Plans. **Table C-1** summarizes the proposed field activities, including the number and type of samples that will be collected at each location. Proposed sampling locations are shown in **Figure 3-1** and **Figure 3-2** in the Work Plan.

TABLE C-1
Summary of Samples

Field Activity	No. of Samples	Media	Analysis
Surface Soil Sampling	15	Soil	<ul style="list-style-type: none"> - Lithology - Volatile Organic Compounds (VOCs), - Semivolatile Organic Compounds (SVOCs), - Polychlorinated biphenyls (PCBs), - Pesticides - Target analyte list (TAL) Metals, and - 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD), within Site 28 only
Subsurface Soil Sampling	15	Soil	<ul style="list-style-type: none"> - Lithology - VOCs, - SVOCs, - PCBs, - Pesticides - TAL Metals, and -- TCDD, within Site 28 only
Groundwater Sampling	10	GW	<ul style="list-style-type: none"> - VOCs, - SVOCs, - PCBs, - Pesticides, - TAL Metals, and - Field Parameters

Sample Designation

In order to identify and accurately track the various samples, all samples collected during this investigation, including quality assurance/quality control (QA/QC) samples, will be designated with a unique number. The number will serve to identify the investigation, the site, the sample media, sampling location, the depth (soil and groundwater collected from soil boring) or round (groundwater) of sample, and QA/QC qualifiers.

The sample designation format is as follows:

Site#-Media/Station# or QA/QC-Year/Round or Depth Interval

An explanation of each of these identifiers is given below.

Site#:	This investigation includes Site 28 under the Installation Restoration (IR) Program. Therefore, the prefix "IR28" will be used.
Media:	GW = Groundwater from temporary wells SS = Surface soil IS = Subsurface soil
Station#:	Each monitoring well will be identified with a unique identification number. Existing monitoring well numbers will be used.
QA/QC:	D = Duplicate Sample (following sample type/number) FB = Field Blank ER = Equipment Rinsate TB = Trip Blank

For QA/QC samples the date and year will be in a MMDDYY format, such as May 14, 2008 would be referred to as 051408.

All matrix spike/matrix spike duplicate (MS/MSD) samples will be entered in the same line as the field sample on the chain of custody. The total number of sample containers submitted will be entered on the chain of custody and "MS/MSD" will be indicated in the comments section.

Year/Round#: Year/Round indicators will be used for samples collected from monitoring wells. Each round of sampling will have a distinct identification number.

"08" = year 2008

"A" = sampling during the first quarter at the site

Depth Interval: Depth indicators will be used for soil samples collected using DPT. The number will reference the depth interval (in feet) of the sample.

2-3 = 2 to 3 feet bgs

Under this sample designation format, the sample designation IR28-GW01-08A refers to:

IR28-GW01-08B	IR Site 28
IR28-GW01-08B	Groundwater sample from temporary well IR28-TW01
IR28-GW01-08B	Sampled in the second quarter of 2008

The sample designation format for QA/QC IR28-TB105148 refers to:

IR28-TB1051408	IR Site 28
IR28-TB1051408	Trip Blank #1
IR28-TB1051408	Date sampled

This sample designation format will be followed throughout the project. Required deviations to this format in response to field conditions will be documented.

Sample Handling and Analysis

4.1 Sample Preservation and Handling

Sample preservation and handling are described in depth in Section 5 of the Master QAPP. QA/QC samples, with the exception of trip blanks, will be collected in the same containers with preservatives as the field samples. The preservative and holding time for analysis is shown in **Table C-2**.

TABLE C-2
Sample Containers, Preservation, and Holding Times
Field Sampling and Analysis Plan, French Creek Road Extension, MCB Camp Lejeune

Analysis	Matrix	Method	Container	Preservation	Maximum Hold Time
VOC	Aqueous	SW846 8260B	3 x 40 mL Glass vials with septa	Cool to 4°C, HCl to pH<2	14 days to analysis
VOC	Solid	SW846 8260B	3 x .5 g Encore	Cool to 4°C	48 hours to extraction / 14 to analysis
SVOC, Pesticides and PCBs	Aqueous	SW846 8270C, 8081A, 8082	2 x 1000 mL Glass amber for each parameter	Cool to 4°C	7 days to extraction / 40 to analysis
SVOC, Pesticides and PCBs	Solid	SW846 8270C, 8081A, 8082	1 x 8 oz Glass jar	Cool to 4°C	14 days to extraction / 40 to analysis
TAL Metals	Aqueous	SW846 6010A, 7470A	500 1000 mL Poly	Cool to 4°C, HNO ₃ to pH<2	6 months (28 days for Hg)
TAL Metals	Solid	SW846 6010B/ 7471A	100 g 4 oz. glass jar	Cool to 4°C	6 months (28 days for Hg)
TCDD	Solid	SW846 8290	1 x 4 oz amber jar	Cool to 4°C	30 days to extraction/45 days to analysis

4.2 Chain-of-Custody and Field Logbook

Chain of Custody and field logbook requirements are described in Section 6 of the Master QAPP.

Appendix D
UFP-QAPP Attachment

Final

**Quality Assurance Project Plan
Preliminary Assessment/Site Inspection
French Creek Road Extension**

**Marine Corps Base Camp Lejeune
Jacksonville, North Carolina**

Task Order 011

March 2009

Prepared for

**Department of the Navy
Naval Facilities Engineering Command
Atlantic**

Under the

**NAVFAC CLEAN 1000 Program
Contract N62470-08-D1000**

Prepared by



CH2MHILL

Charlotte, North Carolina

Contents

Acronyms and Abbreviations	vii
SAP/QAPP Identifying Information.....	ix
Crosswalk to Related Information	xi
1. Project Management.....	1-1
1.1 Introduction	1-1
1.1.1 Distribution List and Signoff Sheet	1-1
1.1.2 Laboratory Work Group	1-2
1.1.3 Laboratory Communication	1-3
1.2 Quality Objectives and Criteria for Laboratory Measurement Data	1-6
1.2.1 Levels of Data Quality	1-6
1.2.2 Data Quality and Sampling Objectives.....	1-8
1.2.3 Method Performance Objectives.....	1-10
1.2.4 Quality of Data	1-10
1.2.5 Project Quality/Systematic Planning Process Statements	1-11
1.3 Laboratory Special Training, Requirements, and Certifications	1-13
1.4 Laboratory Documentation and Records	1-13
2. Laboratory Measurement and Data Acquisition	2-1
2.1 Laboratory Sample Handling and Custody Requirements	2-15
2.1.1 Sample Custody	2-15
2.1.2 Field Custody	2-15
2.1.3 Laboratory Sample Custody.....	2-16
2.1.4 Sample Packing and Shipping	2-17
2.2 Analytical Method Requirements.....	2-17
2.2.1 Analytical Methods for Volatile Organic Compounds.....	2-21
2.2.2 Analytical Laboratory	2-35
2.3 Quality Control Requirements.....	2-42
2.3.1 Field QC Blank Samples and Duplicate Field Samples	2-42
2.3.2 TBs.....	2-42
2.3.3 Equipment Rinsate Blank Samples.....	2-42
2.3.4 Field/Decontamination Source Water Blanks	2-42
2.3.5 Temperature Blanks.....	2-43
2.3.6 Duplicate Field Samples	2-43
2.3.7 Laboratory Method/Preparation Blanks.....	2-43
2.3.8 Matrix Spike/Matrix Spike Duplicate Samples.....	2-43
2.3.9 Surrogate Spikes.....	2-44
2.3.10 Laboratory Control Spike Samples.....	2-44
2.3.11 Interference Check Samples	2-44
2.3.12 Internal Standards.....	2-45
2.3.13 Retention Time Windows	2-45
2.3.14 Confirmation of Identification	2-45

2.3.15	Standard Materials	2-45
2.4	Laboratory Corrective Action.....	2-60
2.4.1	Field Corrective Action.....	2-61
2.4.2	Laboratory Corrective Action.....	2-61
2.4.3	Laboratory Equipment.....	2-62
2.5	Inspection and Acceptance Requirements for Supplies and Consumables	2-67
2.6	Data Acquisition Requirements	2-67
3.	Laboratory Assessment and Oversight	3-1
3.1	Assessments and Response Actions	3-1
3.1.1	Laboratory Performance and System Audits	3-1
3.2	Reports to Management	3-1
4.	Data Validation and Usability	4-1
4.1	Data Review, Validation, and Verification Requirements.....	4-1
4.2	Verification and Validation Methods	4-1
4.2.1	Data Verification.....	4-1
4.2.2	Data Validation.....	4-1
4.2.3	DQE Process.....	4-2
4.3	Reconciliation with Data Quality Objectives.....	4-3
4.4	Usability Assessment.....	4-3
5.	References.....	5-1

Attachment

A SOPs Laboratory

Tables

1-1	Distribution List	1-2
1-2	Communication Pathways.....	1-3
2-1	QA/QC Requirement Summary and Measurement Performance Criteria.....	2-2
2-1a	Measurement Performance Criteria Table – Field QC Samples Aqueous VOCs	2-2
2-1b	Measurement Performance Criteria Table – Field QC Samples Aqueous SVOCs	2-3
2-1c	Measurement Performance Criteria Table – Field QC Samples Aqueous Pesticides	2-5
2-1d	Measurement Performance Criteria Table – Field QC Samples Aqueous PCBs.....	2-6
2-1e	Measurement Performance Criteria Table – Field QC Samples Aqueous Metals	2-7
2-1f	Measurement Performance Criteria Table – Field QC Samples Soil VOCs.....	2-8
2-1g	Measurement Performance Criteria Table – Field QC Samples Soil SVOCs.....	2-9
2-1h	Measurement Performance Criteria Table – Field QC Samples Soil Pesticides.....	2-11
2-1i	Measurement Performance Criteria Table – Field QC Samples Soil PCBs.....	2-12
2-1j	Measurement Performance Criteria Table – Field QC Samples Soil Metals	2-13
2-2	Analytical Services.....	2-19
2-3	Required Analytical Method, Sample Containers, Preservation, and Holding Times	2-20
2-4	TAL Reporting Limits SW-846 Method 8260B Water	2-21
2-5	TAL Reporting Limits SW-846 Method 8260B Soil	2-23
2-6	TAL Reporting Limits SW-846 Method 8270C Water.....	2-25

2-7	TAL Reporting Limits SW-846 Method 8270C Soil	2-27
2-8	TAL Reporting Limits SW-846 Method 8081A Pesticides Water	2-29
2-9	TAL Reporting Limits SW-846 Method 8081A Pesticides Soil.....	2-30
2-10	TAL Reporting Limits SW-846 Method 8082 PCBs Water.....	2-31
2-11	Target Analyte List Reporting Limits SW-846 Method 8082 PCBs Soil.....	2-31
2-12	TAL List Reporting Limits SW-846 Method 6010B/7470A Metals Water.....	2-32
2-13	Target Analyte List Reporting Limits SW-846 Method 6010B/7470A Metals Soil ..	2-33
2-14	Analytical SOP References	2-37
2-15	Data Package Deliverables	2-41
2-16	Laboratory QC Samples.....	2-47
2-17	Analytical Instrument Calibration	2-63
2-18	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	2-66
4-1	Project Documents and Records.....	4-4

Acronyms and Abbreviations

A2LA	American Association of Laboratory Accreditation
AM	Activity Manager
CA	corrective action
CCV-RT	Continuing Calibration Verification - Retention Time
CD	compact disk
CLP	Contract Laboratory Program
COC	chain of custody
CQAPP	Comprehensive Quality Assurance Plan
CRQL	Contract Required Quantitation Limit
DO	dissolved oxygen
DoD QSM	Department of Defense Quality Systems Manual
DQE	data quality evaluation
DQO	data quality objectives
EICP	Enclosed Inductively Coupled Plasma
EMD	Environmental Management Division
ERB	equipment rinsate blank
FB	field blank
FSP	Field Sampling Plan
FTL	Field Team Leader
GC	gas chromatography
GC/MS	gas chromatograph/mass spectrometer
HPLC	high-performance liquid chromatography
ICP	inductively coupled plasma
ICS	interference check sample
IDL	instrument detection limit
IDW	investigation-derived waste
IS	internal standard
LCS	laboratory control sample
MCB	Marine Corps Base
MCL	maximum contaminant level
MDL	method detection limit
MS	mass spectroscopy
MS/MSD	matrix spike/matrix spike duplicate
NAVFAC	Naval Facilities Engineering Command
NC2LGW	North Carolina 2L Groundwater
NCDENR	North Carolina Department of the Environment and Natural Resources
NCGWQS	North Carolina 2L Groundwater Quality Standards
NIST	National Institute of Standards and Technology

NTR	Navy Technical Representative
ORP	oxidation reduction potential
PAL	project action limit
PARCC	precision, accuracy, representativeness, completeness, and comparability
PC	Project Chemist
PCB	polychlorinated biphenyls
PM	Project Manager
POC	point of contact
PQO	project quality objective
QA	quality assurance
QAM	Quality Assurance Manual
QAPP	Quality Assurance Project Plan
QC	quality control
QL	quantitation limit
RF	response factor
RL	reporting limit
RPD	relative percent difference
RPM	Remedial Project Manager
RSD	relative standard deviation
RSL	regional screening level
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SOP	standard operating procedure
SQL	sample quantitation limit
SVOC	semivolatile organic compound
TAL	target analyte list
TB	trip blank
TCL	target compound list
UFP-QAPP	Uniform Federal Policy-Quality Assurance Project Plans
USEPA	U.S. Environmental Protection Agency
VOC	volatile organic compound
WP	Work Plan

SAP/QAPP Identifying Information

Site Name/Number: French Creek Road Extension/ Installation Restoration Site 28

Operable Unit: 7

Contractor Name: CH2M HILL

Contract Number: N62470-08-D-1000

Contract Title: NAVFAC CLEAN 1000 Contract Task Order 011

Work Assignment Number (optional):

1. This Sampling and Analysis Plan (SAP) was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)* (U.S. Environmental Protection Agency [USEPA], 2005) and *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, QAMS* (USEPA, 2002) .

2. Identify regulatory program: Comprehensive Environmental Response, Compensation, and Liability Act

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
Naval Facilities Engineering Command (NAVFAC) Request for Proposal	6/11/08
CH2M HILL Implementation Plan/Fee Proposal	7/24/08

5. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation.

Title	Date
N/A	

6. List organizational partners (stakeholders) and connection with lead organization:
USEPA Region IV, North Carolina Department of the Environment and Natural Resources, NAVFAC Mid-Atlantic, Marine Corps Base Camp Lejeune

7. Lead organization NAVFAC Mid-Atlantic

8. If any required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:

Worksheet #9 – Not applicable. Scope was issued by NAVFAC.

Worksheet #13 – Not applicable. No secondary data used in developing this SAP.

Crosswalk to Related Information

In an effort to comply with the requirements of the Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP) (U.S. Environmental Protection Agency [USEPA], 2005), the following crosswalk table has been provided. Because this project is Activity-Funded, this QAPP does not follow the UFP-QAPP 37 worksheet format; however, this Crosswalk Table identifies where the key elements of the UFP-QAPP are located within the enclosed QAPP and/or attached Work Plan (WP).

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
A. Project Management		
Documentation		
1	Title and Approval Page	QAPP Title Pages
2	Table of Contents SAP Identifying Information	QAPP Identifying Information Page
3	Distribution List	QAPP Table 1-1
4	Project Personnel Sign-Off Sheet	QAPP Table 1-1
Project Organization		
5	Project Organizational Chart	Figure 3-1 of Final Master QAPP QAPP Figure 1-1
6	Communication Pathways	Section 7 of WP Section 3.2 of Final Master QAPP
7	Personnel Responsibilities and Qualifications Table	Section 7 of WP Section 3.1 of Final Master QAPP
8	Special Personnel Training Requirements Table	Field personnel will meet requirements specified in Section 4.1 of Final Master HSP
Project Planning/ Problem Definition		
9	Project Planning Session Documentation (including Data Needs tables) Project Scoping Session Participants Sheet	Not applicable
10	Problem Definition, Site History, and Background. Site Maps (historical and present)	Sections 1 and 2 of WP Site Map – Figure 2-2 of WP
11	Site-Specific Project Quality Objectives	Section 1.2 of WP Section 4 of Final Master QAPP
12	Measurement Performance Criteria Table	QAPP Table 2-1

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
13	Sources of Secondary Data and Information Secondary Data Criteria and Limitations Table	Not applicable
14	Summary of Project Tasks	Sections 1.1 and 3 of WP
15	Reference Limits and Evaluation Table	Section 4 of Final Master QAPP QAPP Tables 2-4 to 2-13
16	Project Schedule/Timeline Table	Figure 8-1 of WP
B. Measurement Data Acquisition		
Sampling Tasks		
17	Sampling Design and Rationale	Sections 3.3 through 3.5 of WP
18	Sampling Locations and Methods/ Standard Operating Procedure (SOP) Requirements Table Sample Location Map(s)	Table 3-1 of WP, Figures 3-2 and 3-3 of WP Sections 3.3 through 3.5 of WP Section 3 of Final Master SAP
19	Analytical Methods/SOP Requirements Table	Table 3-1 of WP, Tables C-1 and C-2 of FSP (Appendix C) QAPP Table 2-14
20	Field Quality Control Sample Summary Table	Table 3-1 of WP
21	Project Sampling SOP References Table Sampling SOPs	Section 3 of WP Section C.4 of FSP Section 3 of Final Master SAP
22	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	Sections 7 and 12 of Final Master QAPP
Analytical Tasks		
23	Analytical SOPs Analytical SOP References Table	QAPP Table 2-6
24	Analytical Instrument Calibration Table	QAPP Table 2-17
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	QAPP Table 2-18
Sample Collection		
26	Sample Handling System, Documentation Collection, Tracking, Archiving and Disposal Sample Handling Flow Diagram	Section 6 of the Final Master QAPP Section C.4 of Field Sampling Plan (FSP) Section 3-5 – Work Plan
27	Sample Custody Requirements, Procedures/ SOPs Sample Container Identification Example Chain-of-Custody Form and Seal	Section C.3 of FSP Section 6 of Final Master QAPP Section 4.2 of Final Master SAP Attachment 6 of Final Master SAP

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
Quality Control Samples		
28	Quality Control Samples Table Screening/Confirmatory Analysis Decision Tree	Table 3-1 of WP QAPP Section 2.3
Data Management Tasks		
29	Project Documents and Records Table	Section 6 of Work Plan Sections 6 and 9 of Final Master QAPP
30	Analytical Services Table Analytical and Data Management SOPs	QAPP Section 2.2
C. Assessment Oversight		
31	Planned Project Assessments Table Audit Checklists	Section 11 of Final Master QAPP
32	Assessment Findings and Corrective Action Responses Table	Section 14 of Final Master QAPP
33	Quality Assurance Management Reports Table	Section 11 of Final Master QAPP Data will be validated externally and reviewed by the chemist prior to loading to database.
D. Data Review		
34	Verification (Step I) Process Table	Section 5 of Work Plan Section 9 of Final Master QAPP
35	Validation (Steps IIa and IIb) Process Table	Section 5 of Work Plan Section 9 of Final Master QAPP
36	Validation (Steps IIa and IIb) Summary Table	Section 5 of Work Plan Section 9 of Final Master QAPP
37	Usability Assessment	Section 5 of Work Plan Section 9 of Final Master QAPP

Project Management

1.1 Introduction

This site-specific Quality Assurance Project Plan (QAPP) is meant to serve in conjunction with the Marine Corps Base (MCB) Camp Lejeune Master Project QAPP (CH2M HILL, 2008). Site-specific information is contained within this QAPP. This document applies only to the French Creek Road Extension Preliminary Assessment /Site Investigation and is a component of the Work Plan (WP). The QAPP describes the data quality objectives (DQOs), specific quality assurance (QA) and quality control (QC) activities, and laboratory activities necessary to achieve the DQOs of the project. Subcontractors will be required to review this site-specific QAPP. Subcontractors will be expected to adhere to the procedures specified in this document.

The requirements of this document apply to contractors and subcontractors. Deviations from these procedures will be documented.

This section provides an overview of the following topics:

- Laboratory organization roles and responsibilities
- Project quality objectives (PQOs) and criteria for measurement data
- Laboratory personnel special training requirements and certifications

Section 2 describes the measurement and data acquisition procedures and the analytical methods to be performed in support of this monitoring. It addresses the following laboratory aspects of measurement and data acquisition:

- Sampling method requirements
- Sample handling and custody requirements
- Analytical method requirements
- QC requirements
- Instrument and equipment testing, inspection, and maintenance requirements
- Instrument calibration and frequency
- Inspection and acceptance requirements for supplies and consumables
- Data acquisition requirements

Section 3 describes the laboratory assessment and oversight activities that will be followed to determine whether the QC identified this QAPP is being implemented and documented as required.

Section 4 presents the data review, validation, and evaluation requirements.

1.1.1 Distribution List and Signoff Sheet

Table 1-1 identifies all recipients of the Sampling and Analysis Plan (SAP)/QAPP and is not exclusively for CH2M HILL personnel.

TABLE 1-1
Distribution List

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-mail Address or Mailing Address	Signature	SAP/QAPP Section Reviewed	Date SAP/QAPP Read
Bryan K. Beck	Navy Technical Representative (NTR)	Naval Facilities Engineering Command (NAVFAC) Mid-Atlantic	(757) 322-4734	bryan.beck@usmc.mil			
Bob Lowder	Environmental Engineer	MCB Camp Lejeune-Environmental Management Division (EMD)	(910) 451-9607	robert.a.lowder@usmc.mil			
Gena Townsend	Remedial Project Manager (RPM)	U.S. Environmental Protection Agency Region 4	(404) 562-8538	towsend.gena@epa.gov			
Randy McElveen	RPM	<u>North Carolina Department of the Environment and Natural Resources (NCDENR)</u>	(919) 508-8467	randy.mcelveen@ncmail.net			
Matt Louth	Activity Manager (AM)	CH2M HILL	(757) 671-6240	matt.louth@ch2m.com			
Keri Hallberg	Project Manager (PM)	CH2M HILL	(704) 543-3260	Keri.hallberg@ch2m.com			

1.1.2 Laboratory Work Group

The selected laboratory is responsible for analyzing samples collected during field activities, in accordance with the SAP and the laboratory comprehensive quality assurance project plan (CQAPP). The laboratory PM or client service manager acts as a liaison between the Project Chemist (PC) and the field and laboratory operations and is responsible for the following:

- Receipt of sample custody from the field team members, verification of sample integrity, and transfer of sample fractions to the appropriate analytical departments
- Coordination of sample analyses to meet project objectives
- Preparation of analytical reports
- Review of laboratory data for compliance with method requirements
- Review of any QC deficiencies reported by the analytical department manager
- Coordination of any data changes resulting from review by the project QA supervisor or the PM

- Completion of data package deliverables
- Communication with the PC pertaining to analytical and QC issues
- Response to questions from the project team during the data quality evaluation (DQE) process

1.1.3 Laboratory Communication

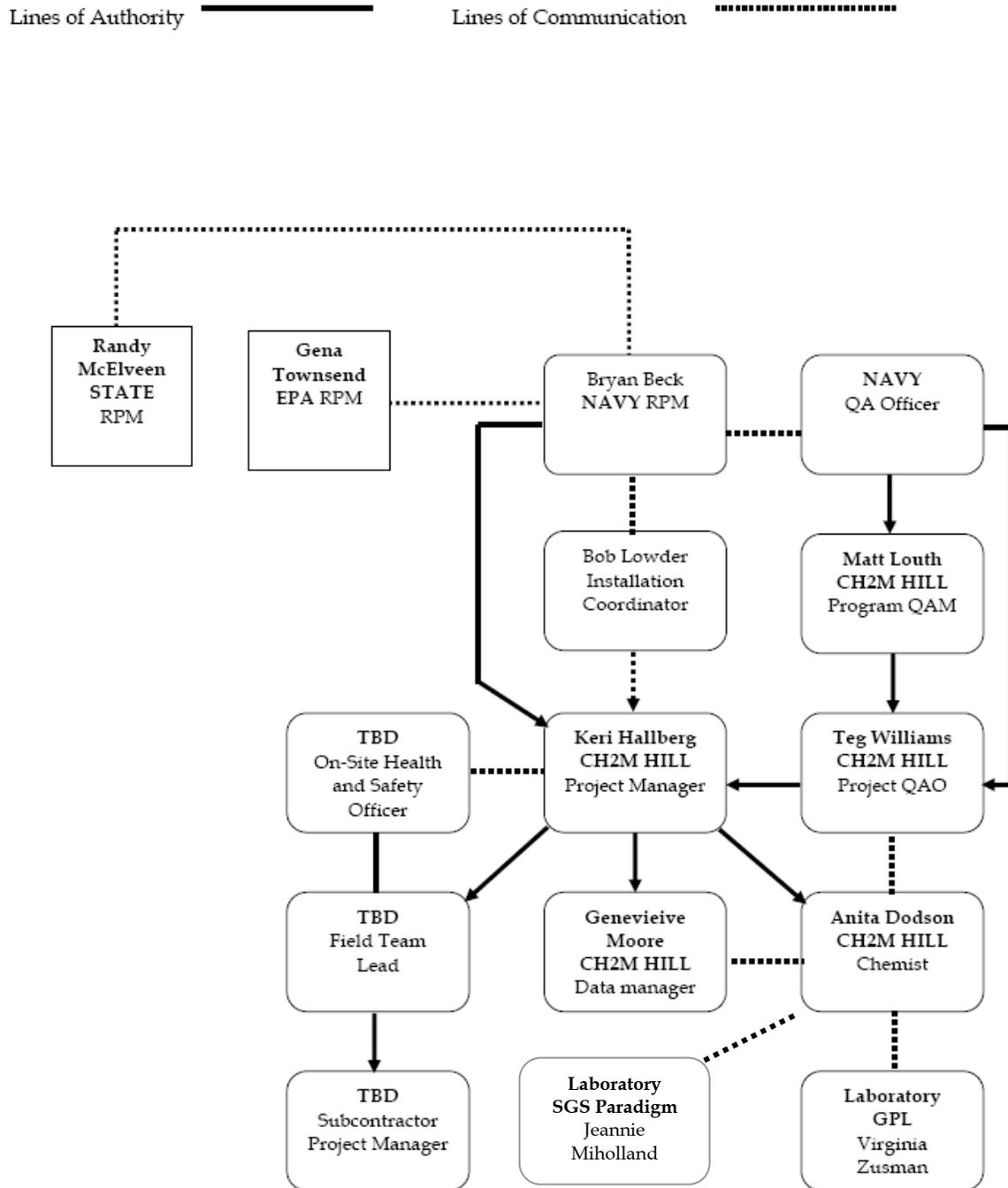
TABLE 1-2
Communication Pathways

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure, Pathway, etc.
Communication with Navy (lead agency)	NTR/ RPM	Bryan Beck	bryan.beck@usmc.mil (757) 322-4734	Primary point of contact (POC) for Navy; can delegate communication to other internal or external points of contact. RPM will notify USEPA and NCDENR via email or telephone call within 24hrs for field changes effecting the scope or implementation of the design occur. Navy will have 30 days for Work Plan (WP) review. All sampling data will be presented and discussed during partnering meetings.
Communication with USEPA Region 4	USEPA Region 4 RPM	Gena Townsend	townsend.gena@epa.gov (404) 562-8538	Primary POC for USEPA; can delegate communication to other internal or external points of contact. Upon notification of field changes, USEPA will have 24hrs to approve or comment on the field changes. All data results will be presented and discussed during partnering meetings
Communication with NCDENR	NCDENR RPM	Randy McElveen	randy.mcelveen@ncmail.net (919) 508-8467	Primary POC for NCDENR; can delegate communication to other internal or external points of contact. Upon notification of field changes, NCDENR will have 24hrs to approve or comment on the field changes.
Communication regarding overall project status and implementation and primary POC with Navy RPM, USEPA, and NCDENR	CH2M HILL AM	Matt Louth	matt.louth@ch2m.com (757) 671-6240	Oversees project and will be informed of project status by the PM. If field changes occur AM will work with the Navy RPM to communicate in field changes to the team via email within 24hrs. All data results will be communicated to the project team during the first partnering meeting following data receipt.
Technical communications for project implementation, and data interpretation	CH2M HILL Senior Consultants	Teg Williams	tegwyn.williams@ch2m.com (704) 543-3297	Contact senior consultant regarding questions/issues encountered in the field, input on data interpretation, as needed. Sr. Consultants will have 24 hrs to respond to technical field questions as necessary. Additionally, Sr. consultants will review of the data as necessary prior to partnering team discussion and reporting review.

TABLE 1-2
Communication Pathways

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure, Pathway, etc.
Communications regarding project management and implementation	PM	Keri Hallberg	keri.hallberg@ch2m.com (704) 543-3260	All information and materials about the project will be forwarded to the Navy, Activity Mangers and Senior Consultants as necessary. POC for field sampling team.
Health & Safety	CH2M HILL Site Safety Coordinator	TBD		Responsible for the adherence of team members to the site safety requirements described in the HASP. Will report health and safety incidents and near losses to PM.
WP changes in field	Field Team Leader (FTL)	TBD		Documentation of deviations from the WP will be made in the field logbook and the PM will be notified immediately. Deviations will be made only with approval from the PM.
QAPP Field Changes/ Field Progress Reports	FTL	TBD		Documentation of field activities and WP deviations (made with the approval of AM and/or Quality Assurance Officer) in field logbooks; provide daily progress reports to PM.
Data tracking from field collection to database upload	Environmental Information Specialist	Genevieve Moore	genevieve.moore@ch2m.com (757) 671-6284	Tracking data from sample collection through database upload.
Reporting Lab Data Quality Issues	Laboratory QA officer	Virginia Zusman/GPL Laboratories	zusman@gplab.com (301) 694-5310	All QA/ QC issues with project field samples will be reported within 2 days to the project chemist by the laboratory.
Reporting Lab Data Quality Issues (Dioxin)	Assistant Director	Jeannie Milholland/SGS Paradigm	jeannie.milholland@sgs.com (910) 350-1903	All QA/ QC issues with project field samples will be reported within 2 days to the project chemist by the laboratory.
Field and analytical corrective actions (CAs)	Project Chemist (PC)	Anita Dodson	anita.dodson@ch2m.com (757) 671-6218	Any CAs for field and analytical issues will be determined by the FTL and/or the PC and reported to the PM within 4hrs.
Release of Analytical Data	PC	Anita Dodson	anita.dodson@ch2m.com (757) 671-6218	No analytical data can be released until validation of the data is completed and has been approved by the PC. The PC will review analytical results within 7 days of receipt for release to the project team.
Field corrective actions	FTL and PM	TBD/Keri Hallberg	keri.hallberg@ch2m.com (704) 543-3260	Field and analytical issues requiring corrective action will be determined by the FTL and/or PM; the PM will ensure QAPP requirements are met by field staff

FIGURE 1-1
Project Organization



1.2 Quality Objectives and Criteria for Laboratory Measurement Data

This section defines the levels of data and briefly outlines the DQO development process for this project. The level of data quality is dependent on the objective use of the results supported by the data. This subsection also provides the quantitative quality objectives and measurement performance criteria for the analytical data.

1.2.1 Levels of Data Quality

The data use determines the required levels of data quality. The two categories of data quality established by the USEPA, *screening* and *definitive*, are defined as follows:

Screening data are generated by rapid methods of analysis with less rigorous sample preparation, calibration and/or QC requirements as compared to the requirements for producing definitive data. Sample preparation steps commonly are restricted to simple procedures such as dilution with a solvent, instead of elaborate extraction/digestion and cleanup. Screening data may provide analyte identification and quantitation, although the quantitation may be relatively imprecise, unless USEPA reference methods are used. Physical test methods such as dissolved oxygen (DO) measurements, temperature and pH measurements, moisture content, turbidity, conductance, etc., have been designated by definition as screening techniques.

Depending on the DQOs, screening methods may require confirmation samples that generate definitive data. Confirmation samples will be selected to include both detected and non-detected results from the screening technique.

Definitive data are generated using rigorous analytical methods such as approved USEPA reference methods. Data are analyte-specific, and both identification and quantitation are confirmed. These methods have standardized QC and documentation requirements. Definitive data are not restricted in their use unless quality problems require data qualification.

Four levels of data reporting may be performed as part of this field effort, with each level having different supporting QA/QC documentation. The four levels correspond to QC Levels I, II, III, and IV. Level I data reporting includes field monitoring activities such as measurements of pH, temperature, conductivity, DO, oxidation reduction potential (ORP), and turbidity. Level II data reporting may include screening activities, which are indicative of the nature of contamination, whereas Level III data reporting provides definitive or confirmation data. Level IV data reporting includes the highest level of QC with significant additional documentation.

Level IV data packages will be requested for this project.

Level I-Field Surveys

Level I encompasses field monitoring or screening activities and does not require formal data package deliverables. Level I activities are focused on easily measured bulk characteristics of a sample such as pH, conductivity, ORP, and DO. Monitoring results, as

well as pertinent data concerning the sampling event, will be documented in the bound field book. Level I documentation will consist of the following:

- Instrument identification
- Calibration information (standards used and results)
- Date and time of calibration and field measurements
- Field measurement results

The logbooks will be reviewed daily by the FTL for completeness and correctness. No additional documentation or DQE is required.

Level II—Screening Activities, Physical Parameters, and Investigation-derived Waste Analyses

Level II includes the analyses submitted to the laboratories for screening, physical parameter testing, and analyses associated with the characterization of the investigation-derived waste (IDW) samples. Samples submitted for analysis under Level II will require the delivery of an analytical data package. Level II documentation will consist of the following:

- Case narrative
- Sample results
- Selected QC information such as surrogate recovery
- Associated blank results
- Completed chain-of-custody and any sample receipt information

Level III—Laboratory Analysis

The purposes of Level III data include the following:

- To further define the nature and extent of contamination at MCB, Camp Lejeune;
- To define the risk associated with contamination;
- To define the fate and transport mechanisms of site-related contaminants; and
- To monitor contaminant levels at the Base.

The list of methods and the corresponding target compound lists (TCLs) will be designed to meet the needs of the specific project. In general, samples will be analyzed using USEPA-approved methods from the current edition of *SW-846, Test Methods for Evaluating Solid Waste*. Specific methods are included in the site-specific QAPPs.

Level IV—Laboratory Analysis

The requirements for Level IV documentation also are described in **Table 2-15**. This level provides the most stringent level of documentation, and allows the data reviewer or data validator to recreate the analytical sequence and evaluate raw data such as quantitation reports generated from the instrumentation used in the analyses. The list of methods (presented in **Table 2-3**) and the corresponding target analytes have been designed to evaluate the potential for contamination at the site. Samples will be analyzed using USEPA-approved methods, including methods from the following documents:

- SW-846—Test Methods for Evaluating Solid Waste (USEPA, 1998)

A Level IV data package will be provided by the laboratory for all methods in this project, excluding IDW.

1.2.2 Data Quality and Sampling Objectives

The data quality objectives (DQOs) for the project were established based upon the *Guidance for the Data Quality Objectives Process* (USEPA, 2000). The DQOs are the basis for the design of the data collection plan and, as such, specify the type, quality, and quantity of data to be collected and how the data are to be used to make the appropriate decisions for the project.

DQOs are qualitative and quantitative statements, developed using the USEPA DQO process, that clarify study objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as a basis for establishing the quality and quantity of data needed to support decisions. DQOs define the performance criteria that limit the probabilities of making decision errors by considering the purpose of collecting data, defining the appropriate type of data needed, and specifying tolerable probabilities of making decision errors. The seven-step DQO process is as follows:

- Step 1 – State the Problem
- Step 2 – Identify the Decision
- Step 3 – Identify the Inputs to the Decision
- Step 4 – Define the Boundaries of the Work
- Step 5 – Develop a Decision Rule
- Step 6 – Specify Tolerable Limits on Decision Errors
- Step 7 – Optimize the Design for Obtaining Data

The following sections present the seven-step DQO process developed for the French Creek Road Extension at this time. As the monitoring program develops, DQOs will be re-evaluated and refined as appropriate.

Step 1 – State the Problem

The U.S. Marine Corps plans to extend French Creek Road in the Hadnot Point area of MCB Camp Lejeune. A portion of the proposed extension is located within Installation Restoration Site 28, the former Hadnot Point Burn Dump. A Preliminary Assessment/Site Inspection WP has been prepared to evaluate the presence and nature of environmental impacts resulting from historical land use practices in the vicinity of the planned French Creek Road Extension area, which may result in potential risks to construction workers during road construction.

Step 2 – Identify the Decision

The principal study questions identified at this time are:

- Is there a potential risk to human health and/or the environment during French Creek Road extension construction activities?
- Is additional environmental assessment necessary?

Step 3 – Identify the Inputs to the Decision

Inputs that are readily available consist of:

- North Carolina 2L Groundwater Quality Standards (NCGWQS)
- NCDENR Soil to Groundwater screening criteria
- USEPA Regional Screening Levels (RSLs) (adjusted)
- Camp Lejeune Base-wide background concentrations (metals only)
- Well construction diagrams, water elevation, and water quality data from previous investigations conducted at Site 28

Inputs requiring additional data collection:

- Groundwater data of sufficient quantity and quality from temporary wells installed in the uppermost aquifer located around along the proposed French Creek Road Extension
- Soil data of sufficient quantity and quality from DPT borings located along the proposed French Creek Road Extension

Step 4 – Define the Boundaries of the Work

Groundwater and soil samples will be collected along the proposed French Creek Road Extension as shown on **Figure 3-2** in the SAP. Groundwater samples will be collected from an estimated depth of 3 to 6 feet. Surface soil samples will be collected from 0 to 1 foot below ground surface and subsurface soil samples will be collected from 1 to 5 ft below ground surface.

Step 5 – Develop a Decision Rule

The decision rule developed for the proposed French Creek Road Extension at this time is as follows:

- If concentrations of contaminants, as established via statistically significant trend analysis of analytical data for each individual sample, exceed any of the screening criteria, then the contaminant will be considered a site-specific contaminant of potential concern for the risk assessments and potential additional environmental investigation.

Step 6 – Specify Limits on Decision Errors

In the absence of environmental data, specification of tolerable limits on the decision errors will not be performed at this time. Potential decision errors include:

- Method detection limits (MDLs) insufficient for comparison to screening criteria
- Insufficient sample numbers to provide statistically significant data
- Absence of regulatory criteria for comparison
- Spatial variability between samples

Step 7 – Optimize the Design for Obtaining Data

The following study design elements will be implemented to ensure data of sufficient quantity and quality are collected to address the study question:

- Collect surface and subsurface soil samples, groundwater samples, water quality data, and water levels along the proposed French Creek Road Extension.
- Collect lithologic and hydrogeologic data to support characterization of aquifer properties.

1.2.3 Method Performance Objectives

The sampling approach and rationale are based on the DQOs, which are presented in the SAP. One activity associated with developing the sampling approach and rationale is developing a list of samples to be collected, sample types, sampling intervals, analytical parameters, and required detection and quantification limits for each required parameter.

Once the number and type of samples and analytical parameters are determined, the method performance requirements are developed. The method performance requirements focus on determining the level of QA/QC and the data package deliverable requirements for all analyses.

1.2.4 Quality of Data

Analytical performance requirements are expressed in terms of precision, accuracy, representativeness, comparability, and completeness (PARCC). Summarized below are brief definitions for each PARCC parameter, and calculation equations as appropriate.

Precision

Precision is a measure of the agreement or repeatability of a set of replicate results obtained from duplicate analyses made under identical conditions. Precision can be estimated by comparing duplicate matrix spike concentrations and field duplicate sample results. The precision of a duplicate determination can be expressed as the relative percent difference (RPD), calculated as:

$$RPD = \left\{ \frac{|X_1 - X_2|}{\left(\frac{X_1 + X_2}{2} \right)} \right\} \times 100$$

where X_1 is the result from the native sample, and X_2 is the result from the duplicate sample.

Accuracy

Accuracy is a measure of the agreement between an experimental determination and the true value of the parameter being measured. Accuracy is estimated through the use of known reference materials and matrix spikes. It is calculated from analytical data and is not measured directly. Spiking of reference materials into a sample matrix provides a measure of the matrix effects on analytical accuracy. Spiking of reference materials into a “non-

matrix”, such as deionized water or Ottawa sand, provides a measure of the accuracy of the analytical method itself. Accuracy, defined as percent recovery (P), is calculated as:

$$P = \left[\frac{(SSR - SR)}{SA} \right] \times 100$$

where SSR is the spiked sample result, SR is the sample result (native), and SA is the spike concentration added to the spiked sample.

Representativeness

Representativeness is a measure of the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is a qualitative parameter that is most concerned with the proper design of the sampling program. Representativeness is demonstrated by providing full descriptions in the project planning documents of the sampling techniques and by making certain that the sampling locations are selected and the number of samples collected such that the accuracy and precision criteria are met.

Comparability

Comparability is another qualitative measure designed to express the confidence with which one data set may be compared to another. Sample collection and handling techniques, sample matrix type, and analytical method all affect comparability. Comparability is limited by the other PARCC parameters because data sets can be compared with confidence only when precision and accuracy are known. Data from one phase of an investigation can be compared to others when similar methods are used and similar data packages are obtained.

Completeness

Completeness is defined as the percentage of measurements judged to be valid, compared to the total number of measurements made for a specific sample matrix and analysis.

Completeness is calculated using the formula:

$$Completeness = \frac{Valid\ Measurements}{Total\ Measurements} \times 100$$

Experience on similar projects has shown that laboratories typically achieve approximately 95 percent completeness. All validated data will be used. During the data validation process, an assessment will be made of whether the valid data are sufficient to meet project objectives. If sufficient valid data are not obtained, the PM will initiate corrective action.

1.2.5 Project Quality/Systematic Planning Process Statements

Who will use the data?

The data will be used by NAVFAC, MCB Camp Lejeune, and CH2M HILL. Within each organization the data will be used by staff scientists/engineers and project managers.

What are the Project Action Limits (PALs)?

- Concentrations of soil and groundwater contaminants detected above the applicable screening criteria including Camp Lejeune Base-wide background concentrations (metals only), NCGWQS, NCDENR Soil to Groundwater screening criteria, and USEPA RSLs.

What will the data be used for?

- Data will be used to determine if contamination exists in groundwater and/or soil along the proposed French Creek Road Extension, to identify potential risk to human health and/or the environment, and to evaluate if additional environmental investigation is necessary.

What types of data are needed?

- The chemistry data for the site for possible contaminants in groundwater and soil: volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), polychlorinated biphenyls (PCBs), pesticides, and target analyte list (TAL) metals.

How “good” must the data be to support the environmental decision?

- Contaminates should achieve reporting limits at or lower than NCGWQS, NCDENR Soil to Groundwater screening criteria, USEPA RSLs, and Base-wide background concentrations.

How much data should be collected (number of samples for each analytical group, matrix, and concentration)? Where, when, and how should the data be collected/generated?

See the WP for specifics.

- Ten (10) temporary wells will be sampled, see **Figure 3-2** of the WP.
- Fifteen (15) DPT borings will be advanced along the proposed French Creek Road Extension, see **Figure 3-2** of the WP.
- Numbers of QA/QC samples for each chemical analysis are discussed in **Table 3-1** of the WP.
- Soil IDW samples will be collected in order to characterize the waste.
- The data will be collected following the standard operating procedures (SOPs) presented in the WP.

Who will collect and generate the data? How will the data be reported?

- A CH2M HILL field team will collect the chemical data.
- Chemical analyses have been subcontracted to GPL Laboratories, LLLP. GPL has been evaluated by NFESC to perform the methods in this project.
- Once generated, all chemical analytical data will be submitted to a yet-to-be selected, Navy-approved third party data validator for validation using the analytical methods and laboratory SOPs as necessary.

- CH2M HILL will receive validated data and upload the data into a centralized electronic database used for Navy projects (EnDat) by the project team(s).
- All chemical data will be reported in a Preliminary Assessment/Site Investigation report following data collection and data validation.

How will the data be archived?

All analytical data will be uploaded into a centralized database developed and maintained by CH2M HILL (EnDat) and used for Navy projects. At the end of the project, paper copies of archived laboratory data and validation reports will be returned to the Navy.

PQOs listed in the form of if/then qualitative and quantitative statements.

Use of Reference Area Data PQOs

- If screening criteria are exceeded and potential human health and/or environmental risks are identified, then it will be determined that additional environmental assessment is necessary.
- If screening criteria are exceeded and no potential human health and/or environmental risks are identified, then it will be determined if additional environmental assessment is necessary.
- If screening criteria are not exceeded, then no further environmental assessment will be necessary.

1.3 Laboratory Special Training, Requirements, and Certifications

Environmental laboratories performing services in support of this project must possess any required state or host nation certification and/or be accredited for each applicable test method by a nationally recognized laboratory accreditation body (e.g. NELAP) and the laboratory must declare conformance to the latest version of the Department of Defense, Quality Systems Manual (DoD QSM).

1.4 Laboratory Documentation and Records

Document	Where Maintained
Chain-of-Custody Records	Electronic .pdf copies in the project file. Hardcopy in the project file. Archived at project closeout.
Corrective Action Forms	Electronic .pdf copies in the project file. Hardcopy in the project file. Archived at project closeout.
Sample Receipt, Custody, and Tracking Records	Electronic .pdf copies in the project file. Hardcopy in the full data package.
Standard Traceability Logs	Hardcopy in the full data package. Archived at project closeout.
Equipment Calibration Logs	Hardcopy in the full data package. Archived at project closeout.
Sample Prep Logs	Hardcopy in the full data package. Archived at project closeout.

Document	Where Maintained
Run Logs	Hardcopy in the full data package. Archived at project closeout.
Equipment Maintenance, Testing, and Inspection Logs	Hardcopy in the full data package. Archived at project closeout.
Reported Field Sample Results	Electronic .pdf copies in the project file. Hardcopy in the data package. Archived at project closeout.
Reported Results for Standards, QC Checks, and QC Samples	Hardcopy in the full data package. Archived at project closeout.
Instrument Printouts (raw data) for Field Samples, Standards, QC Checks, and QC Samples	Hardcopy in the full data package. Archived at project closeout.
Data Package Completeness Checklists	Hardcopy in the data validation report. Archived at project closeout.
Sample Disposal Records	Maintained by the laboratory.
Extraction/Clean-up Records	Maintained by the laboratory.
Raw Data	Hardcopy in the full data package. Archived at project closeout.
Fixed Laboratory Audit Checklists	If completed, hardcopy in the project file. Archived at project closeout.
Data Validation Reports	Electronic .pdf copies in the project file. Hardcopy stored with the data package. Archived at project closeout.

SECTION 2

Laboratory Measurement and Data Acquisition

This section describes the procedures for measurement and data acquisition activities performed in support of the project:

- Method requirements
- Sample handling and custody requirements
- Analytical method requirements
- QC requirements
- Instrument and equipment testing, inspection, and maintenance requirements
- Instrument calibration and frequency
- Inspection and acceptance requirements for supplies and consumables
- Data acquisition requirements

The frequency of field QC sample collection and their measurement performance criteria are specified below in **Table 2-1**.

TABLE 2-1
QA/QC Requirement Summary and Measurement Performance Criteria

Table 2-1a: Measurement Performance Criteria Table – Field QC Samples Aqueous VOCs

QC Sample	Analytical Group ¹	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	VOCs - Water	Once every tune (12 hrs)	Once every tune (12 hrs)	Recovery < 1/2 Contract Required Quantitation Limit (CRQL) except Methylene Chloride & Cyclohexane < 2.5x CRQL, and Acetone & 2-Butanone < 5x CRQL	A
Field Blank (FB), Equipment Blank, TB (TB)		One per week per media, One per day of sampling per type of sampling equipment, One per cooler to the laboratory	One per week per media, One per day of sampling per type of sampling equipment, One per cooler to the laboratory	Recovery < 1/2 CRQL, except Methylene Chloride & Cyclohexane < 2.5x CRQL, and Acetone & 2-Butanone < 5x CRQL	S&A
Field Duplicate		One per 10 field samples	One per 10 field samples	Values >5X CRQL: ± 25%	S&A
Matrix Spike/Matrix Spike Duplicate (MS/MSD)		Once each Sample Delivery Group (SDG), matrix within an SDG, or group of samples of similar concentration level (whichever is most frequent).	Once each SDG, matrix within an SDG, or group of samples of similar concentration level (whichever is most frequent).	see attached recovery list (with allowable marginal exceedances)	A
Laboratory Control Sample (LCS)		once per batch of 20	once per batch of 20	see attached recovery list (with allowable marginal exceedances)	A
Internal Standards (ISs)(Bromochloromethane, 1,4-Difluorobenzene, Chlorobenzene-d5)	VOCs- Water	Spiked in all analyses	Spiked in all analyses	Peak area must be within -50 to +100% of most recent CCV; Peak retention time must be within 30 seconds of most recent Continuing Calibration Verification-Retention Time (CCV RT).	A
Surrogates/ System monitoring compounds		Spiked in all analyses	Spiked in all analyses	Percent Recoveries: 1,2-Dichloroethane-d4 70-120% 4-Bromofluorobenzene 75-120% Toluene-d8 80-122% 1,2-dichlorobenzene-d4 64-132%	A

¹If information varies within an analytical group, separate by individual analyte.

Table 2-1b: Measurement Performance Criteria Table – Field QC Samples Aqueous SVOCs

QC Sample	Analytical Group ¹	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	SVOCs - Water	Each extraction batch	contamination, bias	All target analytes < CRQL, except the phthalate esters, which must be < 5x the CRQL	A
FB, Equipment Blank		One per week per media, One per day of sampling per type of sampling equipment	contamination, bias	All target analytes < CRQL, except the phthalate esters, which must be < 5x the CRQL	S&A
Field Duplicate		One per 10 field samples	precision and accuracy	Values >5X CRQL: ± 25%	S&A
LCS		Each extraction batch	extraction efficiency, accuracy, bias	All ISs with +/- 30seconds of the corresponding CCV RT. All surrogates within +/- 0.06 RRT units of the corresponding CCV. Analyte recoveries should meet the attached recovery limits (with allowable marginal exceedances)	A
MS / MSD		One set per 20 field samples extracted	Precision and Accuracy	All ISs with +/- 30seconds of the corresponding CCV RT. All surrogates within +/- 0.06 RRT units of the corresponding CCV. Analyte recoveries should meet the attached recovery limits (with allowable marginal exceedances)	A
Surrogate spike	SVOCs - Water	All field and QC samples	Extraction efficiency	Relative retention time of each surrogate must be within 0.06 RRT unite of the corresponding CCV RT. No Surrogate recovery <10% and no more than one Acid surrogate and one Base/Neutral Surrogate outside the following limits. Nitrobenzene-d5(B/N): 20-137 2-Fluorobiphenyl(B/N): 23-110 p-Terphenyl-d14(B/N): 20-138 Phenol-d5(A): 29-133 2-Fluorophenol(A): 20-118 2,4,6-Tribromophenol(A): 24-177	A

Table 2-1b: Measurement Performance Criteria Table – Field QC Samples Aqueous SVOCs

QC Sample	Analytical Group ¹	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
IS spike	SVOCs - Water	All field and QC samples	Instrument performance	RT of each IS must be within +/- 30 seconds of the corresponding CCV RT. The EICP area of each IS must be within -50% to +100% of the corresponding CCV IS area.	A

¹If information varies within an analytical group, separate by individual analyte.

Table 2-1c: Measurement Performance Criteria Table – Field QC Samples Aqueous Pesticides

QC Sample	Analytical Group ¹	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	PEST - Water	Each extraction batch	contamination, bias	All target analytes < CRQL, surrogate recoveries must be within 30-150%	A
FB, Equipment Blank		One per week per media, One per day of sampling per type of sampling equipment	contamination, bias	All target analytes < CRQL	S&A
Field Duplicate		One per 10 field samples	precision and accuracy	Values >5X CRQL: ± 25%	S&A
LCS		Each extraction batch	extraction efficiency, accuracy, bias	Surrogates must be within established RT windows. Target analytes should meet the attached recovery limits	A
MS / MSD		One set per 20 field samples	precision and accuracy	Surrogates must be within established RT windows. Target analytes should meet the attached recovery limits.	A
Surrogate Spike	PEST - Water	All field and QC samples	extraction efficiency	Surrogates must fall within established RT windows. Surrogate recoveries should meet recovery limits: decachlorobiphenyl 20-112%, tetrachloro-m-xylene 43-113%	A

¹If information varies within an analytical group, separate by individual analyte.

Table 2-1d: Measurement Performance Criteria Table – Field QC Samples Aqueous PCBs

QC Sample	Analytical Group ¹	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	PCB - Water	Each extraction batch	contamination, bias	All target analytes < CRQL, surrogate recoveries must be within 30-150%	A
FB, Equipment Blank		One per week per media, One per day of sampling per type of sampling equipment	contamination, bias	All target analytes < CRQL	S&A
Field Duplicate		One per 10 field samples	precision and accuracy	Values >5X CRQL: ± 25%	S&A
LCS		Each extraction batch	extraction efficiency, accuracy, bias	Surrogates must be within established RT windows. Target analytes should meet the following advisory recovery limits [Analyte : lower-upper]: Ar-1016 : 35-145 Ar-1260 : 30-145	A
MS / MSD	PCB - Water	One set per 20 field samples	precision and accuracy	Surrogates must be within established RT windows. Target analytes should meet the following advisory recovery and RPD limits [Analyte : lower-upper, RPD]: Ar-1016 : 35-145, 20 Ar-1260 : 30-145, 20	A
Surrogate Spike		All field and QC samples	extraction efficiency	Surrogates must fall within established RT windows. Surrogate recoveries should meet recovery limits: Decachlorobiphenyl 16-148%, Tetrachloro-m-xylene 21-129%	A

¹If information varies within an analytical group, separate by individual analyte.

Table 2-1e: Measurement Performance Criteria Table – Field QC Samples Aqueous Metals

QC Sample	Analytical Group ¹	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
LCS	Total Metals including mercury - Water	1 per batch of 20	accuracy, bias	80-120 %Recovery	A
Method Blank		1 per batch of 20	contamination, bias	concentration < CRQL	A
FB, Equipment Blank		One per week per media, One per day of sampling per type of sampling equipment	contamination, bias	concentration < CRQL; with the exception of common field/ laboratory contaminants (Na, K, Ca and Mg)	S&A
Field Duplicate		One per 10 field samples	precision and accuracy	Values >5X CRQL: ± 25%	S&A
MS		1 per batch of 20	accuracy, precision	75-125 %Recovery	A
MSD		1 per batch of 20	accuracy, precision	RPD < 20%	A
Serial Dilution		1 per batch of 20	accuracy, precision	RPD < 10% from undiluted if element concentration is >50x the CRQL	A

¹If information varies within an analytical group, separate by individual analyte.

Table 2-1f: Measurement Performance Criteria Table – Field QC Samples Soil VOCs

QC Sample	Analytical Group ¹	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	VOCs - Soil	Once every tune (12 hrs)	contamination, bias	Recovery < 1/2 CRQL, except Methylene Chloride & Cyclohexane < 2.5x CRQL, and Acetone & 2-Butanone < 5x CRQL	A
FB, Equipment Blank, TB		One per week per media, One per day of sampling per type of sampling equipment, One per cooler to the laboratory	contamination, bias	Recovery < 1/2 CRQL, except Methylene Chloride & Cyclohexane < 2.5x CRQL, and Acetone & 2-Butanone < 5x CRQL	S&A
Field Duplicate		One per 10 field samples	precision and accuracy	Values >5X CRQL: ± 30%	S&A
MS/ MSD		Once each SDG, matrix within an SDG, or group of samples of similar concentration level (whichever is most frequent).	accuracy, precision	see attached recovery list (with allowable marginal exceedances)	A
LCS		once per batch of 20	accuracy	see attached recovery list (with allowable marginal exceedances)	A
ISs (Bromochloromethane, 1,4-Difluorobenzene, Chlorobenzene-d5)	VOCs - Soil	Spiked in all analyses	system performance	Peak area must be within -50 to +100% of most recent CCV; Peak retention time must be within 30 seconds of most recent CCV RT.	A
System Monitoring Compounds		Spiked in all analyses	accuracy	Percent recoveries: 1,2-Dichloroethane-d4 65-125% 4-Bromofluorobenzene 85-120% Toluene-d8 85-115% 1,2-dichlorobenzene-d4 65-123%	A

¹If information varies within an analytical group, separate by individual analyte.

Table 2-1g: Measurement Performance Criteria Table – Field QC Samples Soil SVOCs

QC Sample	Analytical Group ¹	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	SVOCs - Soil	Each extraction batch	contamination, bias	All target analytes < CRQL, except the phthalate esters, which must be < 5x the CRQL	A
FB, Equipment Blank		One per week per media, One per day of sampling per type of sampling equipment	contamination, bias	All target analytes < CRQL, except the phthalate esters, which must be < 5x the CRQL	S&A
Field Duplicate		One per 10 field samples	precision and accuracy	Values >5X QL: ± 30%	S&A
LCS		Each extraction batch	extraction efficiency, accuracy, bias	All ISs with +/- 30seconds of the corresponding CCV RT. All surrogates within +/- 0.06 RRT units of the corresponding CCV. Analyte recoveries should meet the attached recovery limits (with allowable marginal exceedances)	A
MS / MSD	SVOCs - Soil	One set per 20 field samples extracted	Precision and Accuracy	All ISs with +/- 30seconds of the corresponding CCV RT. All surrogates within +/- 0.06 RRT units of the corresponding CCV. Analyte recoveries should meet the attached recovery limits (with allowable marginal exceedances)	A
Surrogate spike		All field and QC samples	Extraction efficiency	Relative retention time of each surrogate must be within 0.06 RRT unite of the corresponding CCV RT. No Surrogate recovery <10% and no more than one Acid surrogate and one Base/Neutral Surrogate outside the following limits. Nitrobenzene-d5(B/N): 34-113 2-Fluorobiphenyl(B/N): 24-120 p-Terphenyl-d14(B/N): 20-152 Phenol-d5(A): 30-119 2-Fluorophenol(A): 33-110 2,4,6-Tribromophenol(A): 23-160	A

Table 2-1g: Measurement Performance Criteria Table – Field QC Samples Soil SVOCs

QC Sample	Analytical Group ¹	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
IS spike	SVOCs - Soil	All field and QC samples	Instrument performance	RT of each IS must be within +/- 30 seconds of the corresponding CCV RT. The EICP area of each IS must be within -50% to +100% of the corresponding CCV IS area.	A

¹If information varies within an analytical group, separate by individual analyte.

Table 2-1h: Measurement Performance Criteria Table – Field QC Samples Soil Pesticides

QC Sample	Analytical Group ¹	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	PEST - soil	Each extraction batch	contamination, bias	All target analytes < CRQL, surrogate recoveries must be within 30-150%	A
FB, Equipment Blank		One per week per media, One per day of sampling per type of sampling equipment	contamination, bias	All target analytes < 1/2 CRQL	S&A
Field Duplicate		One per 10 field samples	precision and accuracy	Values >5X CRQL: ± 30%	S&A
LCS		Each extraction batch	extraction efficiency, accuracy, bias	Surrogates must be within established RT windows. Target analytes should meet the attached recovery limits.	A
MS / MSD		One set per 20 field samples	precision and accuracy	Surrogates must be within established RT windows. Target analytes should meet the attached recovery limits.	A
Surrogate Spike	PEST - soil	All field and QC samples	extraction efficiency	Surrogates must fall within established RT windows. Surrogate recoveries should meet recovery limits: decachlorobiphenyl 36-120%, tetrachloro-m-xylene 36-120%	A

¹If information varies within an analytical group, separate by individual analyte.

Table 2-1i: Measurement Performance Criteria Table – Field QC Samples Soil PCBs

QC Sample	Analytical Group ¹	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	PCB - soil	Each extraction batch	contamination, bias	All target analytes < CRQL, surrogate recoveries must be within 30-150%	A
FB, Equipment Blank		One per week per media, One per day of sampling per type of sampling equipment	contamination, bias	All target analytes < 1/2 CRQL	S&A
Field Duplicate		One per 10 field samples	precision and accuracy	Values >5X CRQL: ± 30%	S&A
LCS		Each extraction batch	extraction efficiency, accuracy, bias	Surrogates must be within established RT windows. Target analytes should meet the following advisory recovery limits [Analyte : lower-upper]: Ar-1016 : 40-140 Ar-1260 : 60-130	A
MS / MSD	PCB - soil	One set per 20 field samples	precision and accuracy	Surrogates must be within established RT windows. Target analytes should meet the following advisory recovery and RPD limits [Analyte : lower-upper, RPD]: Ar-1016 : 40-140, 20 Ar-1260 : 60-130, 15	A
Surrogate Spike	PCB - soil	All field and QC samples	extraction efficiency	Surrogates must fall within established RT windows. Surrogate recoveries should meet recovery limits: Decachlorobiphenyl 30-144%, Tetrachloro-m-xylene 49-133%	A

¹If information varies within an analytical group, separate by individual analyte.

Table 2-1j: Measurement Performance Criteria Table – Field QC Samples Soil Metals

QC Sample	Analytical Group ¹	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Method Blank	Total Metals including mercury - Soil	1 per batch of 20	contamination, bias	concentration < CRQL	A
LCS		1 per batch of 20	accuracy, bias	80-120% Recovery	A
FB, Equipment Blank		One per week per media, One per day of sampling per type of sampling equipment	contamination, bias	All target analytes < 1/2 CRQL; with the exception of common field/laboratory contaminants (Na, K, Ca and Mg)	S&A
Field Duplicate		One per 10 field samples	precision and accuracy	Values >5X CRQL: ± 30%	S&A
Lab Duplicate		1 per batch of 20	accuracy, precision	RPD<20%	A
MS		1 per batch of 20		75-125% recovery	A

¹If information varies within an analytical group, separate by individual analyte.

Table 2-1k: Measurement Performance Criteria Table – Field QC Samples Soil Dioxin

QC Sample	Analytical Group ¹	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A), or both (S&A)
Field Blank, Equipment Blank	Dioxins	One per week per media, One per day of sampling per type of sampling equipment	contamination, bias	All target analytes < ½ QL	S&A
Field Duplicate		One per 10 field samples	precision and accuracy	Values >5X QL: ± 25%	S&A
MS/ MSD		1 per 20 samples	precision and accuracy	± 30%, 20% difference	A

¹If information varies within an analytical group, separate by individual analyte.

2.1 Laboratory Sample Handling and Custody Requirements

2.1.1 Sample Custody

The sample custody and documentation procedures described in this subsection will be followed throughout all sample collection activities. Components of sample custody procedures include the use of field logbooks, sample labels, custody seals, and chain of custody (COC) forms. Each person involved with sample handling must be trained in COC procedures before the start of the field project. The COC form must accompany the samples during shipment from the field to the laboratory.

A sample is under custody under the following conditions:

- It is in one's actual possession.
- It is in one's view, after being in one's physical possession.
- It was in one's physical possession and that person locks it up to prevent tampering.
- It is in a designated and identified secure area.

2.1.2 Field Custody

The following procedures must be used to document, establish, and maintain custody of field samples:

- Sample labels must be completed for each sample with waterproof ink, ensuring that the labels are legible and affixed firmly on the sample container.
- All sample-related information must be recorded in the project logbook.
- The field sampler must retain custody of samples until they are transferred or properly dispatched.
- One individual from the field sampling team should be designated as the individual responsible for all sample transfer activities. This field investigator will be responsible for the care and custody of samples until they are properly transferred to another person or facility.
- All samples will be accompanied by a COC record. This record documents the transfer of custody of samples from the field investigator to another person, to the laboratory, or to other organizational entities. Each change of possession must be accompanied by an authorized signature for relinquishment and receipt of the samples.
- Completed COC forms will be enclosed in a "sealed" plastic "Zip-Lock ®" baggie (or its equivalent) and placed inside the shipping container used for sample transport from the field to the laboratory.
- When samples are relinquished to a shipping company for transport, the tracking number from the shipping bill or receipt will be recorded on the COC form.
- Custody seals must be affixed on shipping containers when samples are shipped to the laboratory to prevent sample tampering during transportation. If seals are numbered, record the numbers on the COC and in the field log-book.

2.1.3 Laboratory Sample Custody

Each laboratory receiving samples must comply with the laboratory sample custody requirements outlined in the subcontract document and its own CQAPP. The FTL or PC will notify the laboratory of upcoming field sampling activities and the subsequent transfer of samples to the laboratory. This notification will include information concerning the number and type of samples to be shipped and the expected date of arrival.

The following procedures will be used by the laboratory sample custodian, once the samples have arrived at the laboratory:

- The laboratory will designate a sample custodian who will be responsible for maintaining custody of the samples and for maintaining all associated records documenting that custody.
- Upon receipt of the samples, the custodian will check the original chain of custody and request-for-analysis documents and compare them with the labeled contents of each sample container for corrections and traceability. The sample custodian will sign the chain of custody and record the date and time received. The sample custodian also will assign a unique laboratory sample number to each sample.
- Cooler temperature (temperature blank) will be checked and recorded.
- Care will be exercised to annotate any labeling or descriptive errors. If discrepancies occur in the documentation, the laboratory will immediately contact the FTL as part of the corrective action process. A qualitative assessment of each sample container will be performed to note anomalies, such as broken or leaking bottles. This assessment will be recorded as part of the incoming chain of custody procedure.
- If all data and samples are correct and there has been no tampering with the custody seals, the "Received by Laboratory" box will be signed and dated.
- Samples will be stored in a secured area and at a temperature of approximately 4 degrees Centigrade (°C), if necessary, until analyses are to begin.
- The laboratory will send a sample acknowledgment letter to the PC as a record that the shipment arrived and noting the conditions of the containers upon arrival. Any discrepancy will be identified and corrective actions performed. These remarks will be documented on a "sample receipt checklist" or its equivalent. The PC may need to be contacted to provide guidance concerning additional corrective actions or guidance. The PM and PC will retain copies of the sample acknowledgment with the chain-of-custody.
- All samples will be accompanied by a chain-of-custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the field sampler to another person or to the laboratory. Overnight carriers will be treated as a single entity, and a single signature will be required when samples are delivered to the laboratory.
- A laboratory chain of custody form will accompany the sample or sample fraction through final analysis for control.

- Copies of the chain-of-custody and request-for-analysis forms will accompany the laboratory report and will become a permanent part of the project records.
- Samples must be properly packaged for shipment and dispatched to the appropriate laboratory for analysis with a separate signed chain of custody form enclosed in each sample box or cooler.
- All packages must be accompanied by a chain of custody form identifying the contents. The original record must accompany the shipment, and the FTL must retain a copy. Additional details about laboratory sample custody will be included in the CQAPP.

2.1.4 Sample Packing and Shipping

Samples will be delivered to the designated laboratories by local courier or by a common carrier such as Federal Express. Hard plastic ice chests or coolers with similar durability will be used for shipping samples. The coolers must be able to withstand a 4-foot drop onto solid concrete in the position most likely to cause damage. The samples must be cushioned to cause the least amount of damage if such a fall occurs.

After packing is complete, the cooler will be taped with chain of custody seals affixed across the top and bottom joints. Each container will be clearly marked with a sticker containing the originator's address.

The following procedures must be used when transferring samples for shipment:

- All sample coolers and packages must be accompanied by a chain of custody form identifying the contents. When transferring possession of samples, the individuals relinquishing and receiving the sample must sign, date, and note the time on the record. This record documents the transfer of custody of samples from the field sampler to another person or to the laboratory. The original chain of custody record must accompany the shipment, and the FTL must retain a copy.
- Samples must be properly packaged for shipment and dispatched to the appropriate laboratory for analysis with a separate signed chain of custody form enclosed in each sample box or cooler.

2.2 Analytical Method Requirements

This subsection summarizes the target analytes, analytical methods, reporting limits (RLs), and data package deliverables.

Samples will be analyzed using USEPA-approved methods or other recognized standard methods. The principal sources for analytical methods are as follows:

- *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (USEPA SW-846, Third Edition, and its updates, 1998)

Table 2-2 presents the analytical services to be performed during sampling.

TABLE 2-2
Analytical Services

Matrix	Analytical Group	Sample Locations/ ID Number	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
Groundwater	VOC	Refer to WP	low-level SW846 8260B	28 Calendar day TAT	GPL Laboratories, LLLP 7210A Corporate Court Frederick, MD 21703 Garth Herdrich (301) 694-5310	GPL Laboratories, TN 71 Wilson Ave. Johnson City, TN 37604 (423)926-6385
	SVOC	Refer to WP	low-level SW846 8270C			
	Pesticide	Refer to WP	SW846 8081B			
	PCBs	Refer to WP	SW846 8082A			
	Metals	Refer to WP	SW846 6010B/7470A			
Surface/ Subsurface Soil	VOC	Refer to WP	SW846 8260B	28 Calendar day TAT	SGS Environmental 5500 Business Dr. Wilmington, NC 28405 910-350-1903	TBD
	SVOC	Refer to WP	low-level SW846 8270C			
	Pesticides	Refer to WP	SW846 8081B			
	PCBs	Refer to WP	SW846 8082A			
	Metals	Refer to WP	SW846 6010B/7470A			
	Dioxin	Refer to WP	SW-846 8290	28 Calendar day TAT		

Table 2-3 presents the analytical methods, preservatives, containers, and holding times to be used for the analysis of the target compounds.

TABLE 2-3
Required Analytical Method, Sample Containers, Preservation, and Holding Times

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference¹	Containers (number, size, and type)	Sample Volume (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time² (preparation / analysis)
Groundwater	TCL Volatile Organics	SW846 8260B/GPL SOP M.5	3, 40ml, glass	40ml	HCl, cool to 4 ± 2° C	14 days
	TCL Semi Volatile Organics	SW846 8270C/GPL SOPs N.11, P.5	2, 1L, amber glass	1L	cool to 4 ± 2° C, light protected	7 days/40 days
	TCL Pesticides	SW846 8081A/GPL SOPs N.6, Q.6	2, 1L, amber glass	1L	cool to 4 ± 2° C, light protected	7 days/40 days
	TCL PCBs	SW846 8082/GPL SOPs N.6, Q.7	2, 1L, amber glass	1L	cool to 4 ± 2° C, light protected	7 days/40 days
	TAL Total Metals	SW846 6010B&7470A/ GPL SOPs H.8, H.10, H.12	1, 1L, plastic	200ml	HNO ₃ , cool to 4 ± 2° C	6 months
Subsurface and Surface Soil	TCL Volatile Organics	SW846 8260B/GPL SOP M.5	1, 4oz, glass	1g	cool to 4 ± 2° C	14 days
	TCL Semi Volatile Organics	SW846 8270C/GPL SOPs N.12, P.5	1, 8oz, glass	30g	cool to 4 ± 2° C	14/40 days
	TCL Pesticides	SW846 8081A/GPL SOPs N.7, Q.6	1, 8oz, glass	30g	cool to 4 ± 2° C	14/40 days
	TCL PCBs	SW846 8082/GPL SOPs N.7, Q.7	1, 8oz, glass	30g	cool to 4 ± 2° C	14/40 days
	TAL Metals	SW846 6010B&7471A/ GPL SOPs H.21, H.10, H.12	1, 4oz, glass	2g, 2g	cool to 4 ± 2° C	6 months
	Dioxin	SW846-8290/ DC41	1x 4 oz amber	100g	cool to 4 ± 2° C	30 days / 45 days

¹ Refer to the Analytical SOP References table (Worksheet #23).

² Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.

2.2.1 Analytical Methods for Volatile Organic Compounds

USEPA SW-846 Method 8260B–VOCs.

This method uses high-performance liquid chromatography (HPLC) coupled with electrospray ionization mass spectroscopy (MS) or tandem mass spectroscopy (MS/MS) for the identification of perchlorate in surface water, groundwater, waste water, salt water and soil.

Table 2-4 presents the TAL, PALs, and associated RLs for water and **Table 2-5** presents the TAL, PALs, and associated RLs for soil.

TABLE 2-4
TAL Reporting Limits SW-846 Method 8260B Water

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference ¹	Project Quantitation Limit Goal (µg/L)	Laboratory-specific ²	
					CRQLs (µg/L)	MDLs (µg/L)
1,1,1-Trichloroethane	71-55-6	200	NC2LGW & MCL*	1	1	0.19
1,1,2,2-Tetrachloroethane	79-34-5	0.067	RSL	1	1	0.17
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	5900	RSL	1	1	0.30
1,1,2-Trichloroethane	79-00-5	0.24	RSL	1	1	0.23
1,1-Dichloroethane	75-34-3	2.4	RSL	1	1	0.23
1,1-Dichloroethene	75-35-4	7	MCL & NC2LGW	1	1	0.22
1,2,4-Trichlorobenzene	120-82-1	0.82	RSL	1	1	0.14
1,2-Dibromo-3-chloropropane	96-12-8	0.00032	RSL	1	5	1.60
1,2-Dibromoethane	106-93-4	0.0004	NC2LGW	1	1	0.16
1,2-Dichlorobenzene	95-50-1	24	NC2LGW	1	1	0.20
1,2-Dichloroethane	107-06-2	0.15	RSL	1	1	0.24
1,2-Dichloropropane	78-87-5	0.39	RSL	1	1	0.36
1,3-Dichlorobenzene	541-73-1	170	NC2LGW	1	1	0.18
1,4-Dichlorobenzene	106-46-7	0.43	RSL	1	1	0.21
2-Butanone	78-93-3	710	RSL	2	5	0.44
2-Hexanone	591-78-6	200	RSL	1	5	0.34
4-Methyl-2-pentanone	108-10-1	200	RSL	2	5	0.30
Acetone	67-64-1	700	NC2LGW	2	5	0.36
Benzene	71-43-2	0.41	RSL	1	1	0.22
Bromodichloromethane	75-27-4	1.1	RSL	1	1	0.23
Bromoform	75-25-2	4.43	NC2LGW	1	2	0.41
Bromomethane	74-83-9	0.87	RSL	1	1	0.22
Carbon Disulfide	75-15-0	100	RSL	1	1	0.20
Carbon Tetrachloride	56-23-5	0.2	RSL	1	1	0.17

TABLE 2-4
TAL Reporting Limits SW-846 Method 8260B Water

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference ¹	Project Quantitation Limit Goal (µg/L)	Laboratory-specific ²	
					CRQLs (µg/L)	MDLs (µg/L)
Chlorobenzene	108-90-7	9.1	RSL	1	1	0.21
Chloroethane	75-00-3	2100	RSL	1	1	0.29
Chloroform	67-66-3	0.19	RSL	1	1	0.23
Chloromethane	74-87-3	1.8	RSL	1	1	0.25
cis-1,2-Dichloroethene	156-59-2	37	RSL	1	1	0.23
cis-1,3-Dichloropropene	10061-01-5	0.19	NC2LGW	1	1	0.15
Cyclohexane	110-82-7	1300	RSL	1	1	0.20
Dibromochloromethane	124-48-1	0.41	NC2LGW	1	1	0.20
Dichlorodifluoromethane	75-71-8	39	RSL	1	1	0.23
Ethylbenzene	100-41-4	1.5	RSL	1	1	0.20
Isopropylbenzene	98-82-8	68	RSL	1	1	0.18
Methyl Acetate	79-20-9	3700	RSL	1	1	0.17
Methyl tert-Butyl Ether	1634-04-4	12	RSL	1	1	0.27
Methylcyclohexane	108-87-2	NC	N/A	1	1	0.22
Methylene Chloride	75-09-2	4.6	NC2LGW	1	1	0.22
Styrene	100-42-5	100	NC2LGW & MCL	1	1	0.18
Tetrachloroethene	127-18-4	0.11	RSL	1	1	0.28
Toluene	108-88-3	230	RSL	1	1	0.20
trans-1,2-Dichloroethene	156-60-5	11	RSL	1	1	0.22
trans-1,3-Dichloropropene	10061-02-6	0.19	NC2LGW	1	1	0.19
Trichloroethene	79-01-6	1.7	RSL	1	1	0.21
Trichlorofluoromethane	75-69-4	130	RSL	1	1	0.21
Vinyl Chloride	75-01-4	0.015	NC2LGW	1	1	0.31

* NC2LGW = North Carolina 2L Groundwater Standard; MCL = maximum contaminant level

¹List the type and source of the PAL used for each matrix specific analyte (e.g. Background, HH-MCL, HH-region III RBC, eco-WQC, eco-Region III BTAG, etc.)

²Laboratory-specific MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. MDLs may be subject to update.

TABLE 2-5
TAL Reporting Limits SW-846 Method 8260B Soil

Analyte	CAS Number	Project Action Limit (ug/kg)	Project Action Limit Reference ¹	Project Quantitation Limit Goal (ug/kg)	Laboratory-specific ^{2,3}	
					CRQLs (ug/kg)	MDLs (ug/kg)
1,1,1-Trichloroethane	71-55-6	1670	NCSSL	10	5	0.5
1,1,2,2-Tetrachloroethane	79-34-5	0.953	NCSSL	10	5	0.52
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	940000	Residential & Industrial RSL	10	5	0.52
1,1,2-Trichloroethane	79-00-5	17	NCSSL	10	5	0.82
1,1-Dichloroethane	75-34-3	382	NCSSL	10	5	0.29
1,1-Dichloroethene	75-35-4	44.5	NCSSL	10	5	0.42
1,2,4-Trichlorobenzene	120-82-1	2610	NCSSL	10	7	2.1
1,2-Dibromo-3-chloropropane	96-12-8	0.15	NCSSL	5	5	1.6
1,2-Dibromoethane	106-93-4	0.00197	NCSSL	10	5	0.61
1,2-Dichlorobenzene	95-50-1	280	NCSSL	10	5	0.42
1,2-Dichloroethane	107-06-2	1.84	NCSSL	10	5	0.71
1,2-Dichloropropane	78-87-5	2.6	NCSSL	10	5	0.62
1,3-Dichlorobenzene	541-73-1	6500	NCSSL	10	5	0.48
1,4-Dichlorobenzene	106-46-7	23	NCSSL	10	5	0.35
2-Butanone	78-93-3	17100	NCSSL	10	10	2.4
2-Hexanone	591-78-6	1190	NCSSL	10	10	2
4-Methyl-2-pentanone	108-10-1	8125	NCSSL	10	10	4.5
Acetone	67-64-1	2810	NCSSL	10	10	3.6
Benzene	71-43-2	5.62	NCSSL	10	5	0.32
Bromodichloromethane	75-27-4	2.92	NCSSL	10	5	0.39
Bromoform	75-25-2	29	NCSSL	10	5	1.4
Bromomethane	74-83-9	790	Residential RSL	10	5	1.6
Carbon Disulfide	75-15-0	4940	NCSSL	10	5	0.45
Carbon Tetrachloride	56-23-5	2.74	NCSSL	10	5	0.74
Chlorobenzene	108-90-7	438	NCSSL	10	5	0.49
Chloroethane	75-00-3	13600	NCSSL	10	5	0.63
Chloroform	67-66-3	300	Residential RSL	10	5	0.52
Chloromethane	74-87-3	20	NCSSL	10	5	0.44
cis-1,2-Dichloroethene	156-59-2	350	NCSSL	10	5	0.67
cis-1,3-Dichloropropene	10061-01-5	1700	Residential RSL	10	5	0.68
Cyclohexane	110-82-7	120000	Residential & Industrial RSL	10	5	0.6
Dibromochloromethane	124-48-1	1.71	NCSSL	10	5	0.61

TABLE 2-5
TAL Reporting Limits SW-846 Method 8260B Soil

Analyte	CAS Number	Project Action Limit (ug/kg)	Project Action Limit Reference ¹	Project Quantitation Limit Goal (ug/kg)	Laboratory-specific ^{2,3}	
					CRQLs (ug/kg)	MDLs (ug/kg)
Dichlorodifluoromethane	75-71-8	19000	Residential RSL	10	5	0.24
Ethylbenzene	100-41-4	5700	Residential RSL	10	5	0.56
Isopropylbenzene	98-82-8	1680	NCSSL	10	5	0.55
Methyl Acetate	79-20-9	7800000	Residential RSL	10	5	1.4
Methyl tert-Butyl Ether	1634-04-4	916	NCSSL	10	5	0.9
Methylcyclohexane	108-87-2	NC	N/A	10	5	0.75
Methylene Chloride	75-09-2	20.2	NCSSL	10	5	0.76
Styrene	100-42-5	2240	NCSSL	10	5	0.51
Tetrachloroethene	127-18-4	7.42	NCSSL	10	5	0.38
Toluene	108-88-3	7270	NCSSL	10	5	0.28
trans-1,2-Dichloroethene	156-60-5	540	NCSSL	10	5	0.38
trans-1,3-Dichloropropene	10061-02-6	1700	Residential RSL	10	5	0.31
Trichloroethene	79-01-6	18.3	NCSSL	10	5	0.44
Trichlorofluoromethane	75-69-4	31500	NCSSL	10	5	0.63
Vinyl Chloride	75-01-4	0.0952	NCSSL	10	5	0.95
Xylenes, total	1330-20-7	4960	NCSSL	10	15	1.2

¹List the type and source of the PAL used for each matrix specific analyte (e.g. Background, HH-MCL, HH-region III RBC, eco-WQC, eco-Region III BTAG, etc.)

²Laboratory-specific MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. MDLs may be subject to update.

SW846 8270C–Semivolatile Organic Compounds. This method provides procedures for the detection and quantitative measurement of selected semivolatile organic compounds. The analytical method calls for the use of gas chromatograph/mass spectrometer (GC/MS) on sample extracts.

Table 2-6 presents the TAL, PALs, and associated RLs for water and **Table 2-7** presents the TAL, PALs, and associated RLs for soil.

TABLE 2-6
TAL Reporting Limits SW-846 Method 8270C Water

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference ¹	Project Quantitation Limit Goal (µg/L)	Laboratory-specific ²	
					CRQLs (µg/L)	MDLs (µg/L)
1,1'-Biphenyl	92-52-4	180	RSL	10	0.3	0.074
2,2'-oxybis(1-Chloropropane)	108-60-1	0.32	RSL	10	0.2	0.057
2,4,5-Trichlorophenol	95-95-4	370	RSL	25	1.0	0.27
2,4,6-Trichlorophenol	88-06-2	3.7	RSL	10	1.0	0.13
2,4-Dichlorophenol	120-83-2	11	RSL	10	1.0	0.24
2,4-Dimethylphenol	105-67-9	73	RSL	10	1.0	0.12
2,4-Dinitrophenol	51-28-5	7.3	RSL	25	1.0	0.1
2,4-Dinitrotoluene	121-14-2	7.3	RSL	10	0.3	0.09
2,6-Dinitrotoluene	606-20-2	3.7	RSL	10	0.3	0.084
2-Chloronaphthalene	91-58-7	290	RSL	10	0.3	0.076
2-Chlorophenol	95-57-8	0.36	NC2LGW	10	1.0	0.2
2-Methylnaphthalene	91-57-6	14	NC2LGW	10	0.3	0.074
2-Methylphenol	95-48-7	180	RSL	10	1.0	0.19
2-Nitroaniline	88-74-4	NC	N/A	25	0.3	0.072
2-Nitrophenol	88-75-5	18	RSL	10	1.0	0.19
3,3'-Dichlorobenzidine	91-94-1	0.15	RSL	10	1.1	0.36
3-Nitroaniline	99-09-2	1.1	RSL	25	0.3	0.073
4,6-Dinitro-2-methylphenol	534-52-1	0.37	RSL	25	1.0	0.14
4-Bromophenyl-phenylether	101-55-3	NC	N/A	10	0.4	0.13
4-Chloro-3-methylphenol	59-50-7	18	RSL	10	1.0	0.2
4-Chloroaniline	106-47-8	1.2	RSL	10	0.6	0.18
4-Chlorophenyl-phenyl ether	7005-72-3	18	RSL	10	0.2	0.031
4-Methylphenol	106-44-5	3.5	NC2LGW	10	1.0	0.33
4-Nitroaniline	100-01-6	3.2	RSL	25	0.4	0.12
4-Nitrophenol	100-02-7	NC	N/A	25	1.2	0.4
Acenaphthene	83-32-9	80	NC2LGW	10	0.2	0.051
Acenaphthylene	208-96-8	210	NC2LGW	10	0.2	0.057
Acetophenone	98-86-2	370	RSL	10	0.3	0.086
Anthracene	120-12-7	1100	RSL	10	0.3	0.088
Atrazine	1912-24-9	0.29	RSL	10	0.5	0.15
Benzaldehyde	100-52-7	370	RSL	10	0.6	0.2
Benzo(a)anthracene	56-55-3	0.029	RSL	10	0.2	0.065
Benzo(a)pyrene	50-32-8	0.0029	RSL	10	0.3	0.07
Benzo(b)fluoranthene	205-99-2	0.029	RSL	10	0.5	0.14
Benzo(g,h,i)perylene	191-24-2	110	RSL	10	0.6	0.18
Benzo(k)fluoranthene	207-08-9	0.29	RSL	10	0.3	0.1
bis(2-Chloroethoxy) methane	111-91-1	11	RSL	10	0.2	0.054

TABLE 2-6
TAL Reporting Limits SW-846 Method 8270C Water

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference ¹	Project Quantitation Limit Goal (µg/L)	Laboratory-specific ²	
					CROLs (µg/L)	MDLs (µg/L)
bis-(2-Chloroethyl) ether	111-44-4	0.012	RSL	10	0.2	0.036
bis(2-Ethylhexyl) phthalate	117-81-7	2.5	NC2LGW	10	0.4	0.12
Butylbenzylphthalate	85-68-7	35	RSL	10	0.5	0.13
Caprolactam	105-60-2	1800	RSL	10	0.2	0.059
Carbazole	86-74-8	NC	N/A	10	0.2	0.051
Chrysene	218-01-9	2.9	RSL	10	0.3	0.08
Dibenzo(a,h)-anthracene	53-70-3	0.0029	RSL	10	0.3	0.1
Dibenzofuran	132-64-9	3.7	RSL	10	0.2	0.06
Diethylphthalate	84-66-2	2900	RSL	10	0.6	0.2
Dimethylphthalate	131-11-3	NC	N/A	10	0.3	0.077
Di-n-butylphthalate	84-74-2	370	RSL	10	0.3	0.072
Di-n-octylphthalate	117-84-0	140	NC2LGW	10	0.2	0.064
Fluoranthene	206-44-0	150	RSL	10	0.2	0.046
Fluorene	86-73-7	150	RSL	10	0.3	0.07
Hexachlorobenzene	118-74-1	0.02	NC2LGW	10	0.3	0.082
Hexachlorobutadiene	87-68-3	0.44	NC2LGW	10	0.2	0.05
Hexachlorocyclopentadiene	77-47-4	22	RSL	10	0.2	0.049
Hexachloroethane	67-72-1	3.7	RSL	10	0.3	0.1
Indeno(1,2,3-cd)-pyrene	193-39-5	0.029	RSL	10	0.3	0.094
Isophorone	78-59-1	36.8	NC2LGW	10	0.2	0.057
Naphthalene	91-20-3	0.14	RSL	10	0.3	0.072
Nitrobenzene	98-95-3	0.34	RSL	10	0.3	0.1
N-Nitroso diphenylamine	86-30-6	14	RSL	10	0.3	0.094
N-Nitroso-di-n propylamine	621-64-7	0.0096	RSL	10	0.3	0.084
Pentachlorophenol	87-86-5	0.29	NC2LGW	25	1.0	0.047
Phenanthrene	85-01-8	210	NC2LGW	10	0.2	0.061
Phenol	108-95-2	300	NC2LGW	10	1.0	0.22
Pyrene	129-00-0	110	RSL	10	0.3	0.067

¹List the type and source of the PAL used for each matrix specific analyte (e.g. Background, HH-MCL, HH-region III RBC, eco-WQC, eco-Region III BTAG, etc.)

²Laboratory-specific MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. MDLs may be subject to update.

TABLE 2-7
TAL Reporting Limits SW-846 Method 8270C Soil

Analyte	CAS Number	Project Action Limit (ug/kg)	Project Action Limit Reference ¹	Project Quantitation Limit Goal (ug/kg)	Laboratory-specific ^{2,3}	
					CRQLs (ug/kg)	MDLs (ug/kg)
1,1'-Biphenyl	92-52-4	8910	NCSSL	250	6.7	1.6
2,2'-oxybis(1-Chloropropane)	108-60-1	1.29	NCSSL	250	6.7	0.72
2,4,5-Trichlorophenol	95-95-4	40500	NCSSL	500	33	7.8
2,4,6-Trichlorophenol	88-06-2	6100	Residential RSL	250	33	5.4
2,4-Dichlorophenol	120-83-2	18000	Residential RSL	250	33	4.6
2,4-Dimethylphenol	105-67-9	1150	NCSSL	250	33	9.1
2,4-Dinitrophenol	51-28-5	12000	Residential RSL	500	33	4.1
2,4-Dinitrotoluene	121-14-2	12000	Residential RSL	250	6.7	1.9
2,6-Dinitrotoluene	606-20-2	6100	Residential RSL	250	6.7	1.7
2-Chloronaphthalene	91-58-7	210000	Residential & Industrial RSL	250	6.7	2.2
2-Chlorophenol	95-57-8	4.3	NCSSL	250	33	5
2-Methylnaphthalene	91-57-6	1720	NCSSL	250	6.7	1.6
2-Methylphenol	95-48-7	10500	NCSSL	250	33	8.4
2-Nitroaniline	88-74-4	NC	N/A	500	6.7	1.2
2-Nitrophenol	88-75-5	39000	Residential RSL	250	33	3.1
3,3'-Dichlorobenzidine	91-94-1	1100	Residential RSL	250	6.7	1.6
3-Nitroaniline	99-09-2	1800	Residential RSL	500	8	2.4
4,6-Dinitro-2-methylphenol	534-52-1	610	Residential RSL	500	33	3.6
4-Bromophenyl-phenylether	101-55-3	NC	N/A	250	6.7	1.8
4-Chloro-3-methylphenol	59-50-7	39000	Residential RSL	250	33	8.5
4-Chloroaniline	106-47-8	9000	Residential RSL	250	6.7	1.2
4-Chlorophenyl-phenyl ether	7005-72-3	31000	Residential RSL	250	6.7	2.1
4-Methylphenol	106-44-5	17.4	NCSSL	250	33	9.8
4-Nitroaniline	100-01-6	18000	Residential RSL	500	6.7	1.7
4-Nitrophenol	100-02-7	NC	N/A	500	40	13
Acenaphthene	83-32-9	8160	NCSSL	250	6.7	1.4
Acenaphthylene	208-96-8	11400	NCSSL	250	9	2.9
Acetophenone	98-86-2	780000	Residential RSL	250	6.7	0.82
Anthracene	120-12-7	995000	NCSSL	250	6.7	1.5
Atrazine	1912-24-9	4	NCSSL	250	15	4.9
Benzaldehyde	100-52-7	780000	Residential RSL	250	6.7	8.2
Benzo(a)anthracene	56-55-3	150	Residential RSL	100	6.7	1.7
Benzo(a)pyrene	50-32-8	15	Residential RSL	10	8	2.4
Benzo(b)fluoranthene	205-99-2	150	Residential RSL	100	9	2.7
Benzo(g,h,i)perylene	191-24-2	170000	Residential RSL	250	12	3.9
Benzo(k)fluoranthene	207-08-9	1500	Residential RSL	250	10	3.1

TABLE 2-7
TAL Reporting Limits SW-846 Method 8270C Soil

Analyte	CAS Number	Project Action Limit (ug/kg)	Project Action Limit Reference ¹	Project Quantitation Limit Goal (ug/kg)	Laboratory-specific ^{2,3}	
					CRQLs (ug/kg)	MDLs (ug/kg)
bis(2-Chloroethoxy) methane	111-91-1	18000	Residential RSL	250	6.7	1.4
bis-(2-Chloroethyl) ether	111-44-4	.171	NCSSL	250	6.7	0.4
bis(2-Ethylhexyl) phthalate	117-81-7	6670	NCSSL	250	13	4.1
Butylbenzylphthalate	85-68-7	27800	NCSSL	250	6.7	2.1
Caprolactam	105-60-2	3100000	Residential RSL	250	6.7	1.5
Carbazole	86-74-8	NC	N/A	250	6.7	1.4
Chrysene	218-01-9	15000	Residential RSL	250	6.7	2.2
Dibenzo(a,h)-anthracene	53-70-3	15	Residential RSL	10	6.7	1.7
Dibenzofuran	132-64-9	4660	NCSSL	250	6.7	1.2
Diethylphthalate	84-66-2	28200	NCSSL	250	10	3
Dimethylphthalate	131-11-3	NC	N/A	250	10	2.9
Di-n-butylphthalate	84-74-2	24800	NCSSL	250	6.7	0.56
Di-n-octylphthalate	117-84-0	10000000	NCSSL	250	6.7	1.1
Fluoranthene	206-44-0	230000	Residential RSL	250	6.7	2.2
Fluorene	86-73-7	44300	NCSSL	250	6.7	1.6
Hexachlorobenzene	118-74-1	32.1	NCSSL	250	10	3
Hexachlorobutadiene	87-68-3	257	NCSSL	250	6.7	1.4
Hexachlorocyclopentadiene	77-47-4	37000	Residential RSL	250	6.7	1.4
Hexachloroethane	67-72-1	6100	Residential RSL	250	8	2.6
Indeno(1,2,3-cd)-pyrene	193-39-5	150	Residential RSL	100	6.7	1.9
Isophorone	78-59-1	182	NCSSL	100	6.7	1.2
Naphthalene	91-20-3	585	NCSSL	250	6.7	1
Nitrobenzene	98-95-3	3100	Residential RSL	250	6.7	1.8
N-Nitroso diphenylamine	86-30-6	99000	Residential RSL	250	6.7	1.3
N-Nitroso-di-n propylamine	621-64-7	69	Residential RSL	50	6.7	1.5
Pentachlorophenol	87-86-5	22.3	NCSSL	20	33	3.2
Phenanthrene	85-01-8	59600	NCSSL	250	7	2.3
Phenol	108-95-2	1750	NCSSL	250	33	8.2
Pyrene	129-00-0	170000	Residential RSL	250	6.7	1.9

¹List the type and source of the PAL used for each matrix specific analyte (e.g. Background, HH-MCL, HH-region III RBC, eco-WQC, eco-Region III BTAG, etc.)

²Laboratory-specific MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. MDLs may be subject to update.

SW846 8081A—Pesticides. This method is intended for the trace analysis of explosives residues by HPLC using a UV detector. **Table 2-8** presents the TAL, PALs, and associated RLs for water and **Table 2-9** presents the TAL, PALs, and associated RLs for soil.

TABLE 2-8
TAL Reporting Limits SW-846 Method 8081A Pesticides Water

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference ¹	Project Quantitation Limit Goal (µg/L)	Laboratory-specific ²	
					CRQLs (µg/L)	MDLs (µg/L)
alpha-BHC	319-84-6	0.011	RSL	0.05	0.05	0.0017
beta-BHC	319-85-7	0.019	NC2LGW	0.05	0.05	0.0026
delta-BHC	319-86-8	0.019	NC2LGW	0.05	0.05	0.0024
gamma-BHC (Lindane)	58-89-9	.2	NC2LGW	0.05	0.05	0.002
Heptachlor	76-44-8	0.0078	NC2LGW	0.05	0.05	0.0019
Aldrin	309-00-2	0.004	RSL	0.05	0.05	0.0023
Heptachlor epoxide	1024-57-3	0.0038	NC2LGW	0.05	0.05	0.003
Endosulfan I	959-98-8	22	RSL	0.05	0.05	0.0028
Dieldrin	60-57-1	0.0022	NC2LGW	0.05	0.05	0.0032
4,4'-DDE	72-55-9	0.2	RSL	0.05	0.05	0.0022
Endrin	72-20-8	1.1	RSL	0.05	0.05	0.0024
Endosulfan II	33213-65-9	22	RSL	0.05	0.05	0.0031
4,4'-DDD	72-54-8	0.14	NC2LGW	0.05	0.05	0.0032
Endosulfan sulfate	1031-07-8	22	RSL	0.05	0.05	0.003
4,4'-DDT	50-29-3	0.1	NC2LGW	0.05	0.05	0.003
Methoxychlor	72-43-5	18	RSL	0.1	0.05	0.0032
Endrin ketone	53494-70-5	1.1	RSL	0.05	0.05	0.0043
Endrin aldehyde	7421-93-4	1.1	RSL	0.05	0.05	0.0039
alpha-Chlordane	5103-71-9	0.1	NC2LGW	0.05	0.05	0.0026
gamma-Chlordane	5103-74-2	0.1	NC2LGW	0.05	0.05	0.002
Toxaphene	8001-35-2	0.031	NC2LGW	1	1	0.13

¹List the type and source of the PAL used for each matrix specific analyte (e.g. Background, HH-MCL, HH-region III RBC, eco-WQC, eco-Region III BTAG, etc.)

²Laboratory-specific MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. MDLs may be subject to update.

TABLE 2-9
TAL Reporting Limits SW-846 Method 8081A Pesticides Soil

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference ¹	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific ^{2,3}	
					CRQLs (mg/kg)	MDLs (mg/kg)
alpha-BHC	319-84-6	0.077	Residential RSL	1.5	0.0017	0.00009
beta-BHC	319-85-7	0.27	Residential RSL	1	0.0017	0.00010
delta-BHC	319-86-8	0.27	Residential RSL	1	0.0017	0.00008
gamma-BHC (Lindane)	58-89-9	.0062	NCSSL	1	0.0017	0.00009
Heptachlor	76-44-8	0.0023	NCSSL	1	0.0017	0.00010
Aldrin	309-00-2	0.029	Residential RSL	1	0.0017	0.00011
Heptachlor epoxide	1024-57-3	0.00634	NCSSL	1	0.0017	0.00013
Endosulfan I	959-98-8	37	Residential RSL	3.3	0.0017	0.00013
Dieldrin	60-57-1	0.00113	NCSSL	1	0.0017	0.00011
4,4'-DDE	72-55-9	1.4	Residential RSL	1.5	0.0017	0.00013
Endrin	72-20-8	0.44	NCSSL	1	0.0017	0.00014
Endosulfan II	33213-65-9	37	Residential RSL	3.3	0.0017	0.00012
4,4'-DDD	72-54-8	0.129	NCSSL	1	0.0017	0.00012
Endosulfan sulfate	1031-07-8	37	Residential RSL	3.3	0.0017	0.00013
4,4'-DDT	50-29-3	1.36	NCSSL	1.5	0.0017	0.00014
Methoxychlor	72-43-5	56.1	NCSSL	17	0.0017	0.00011
Endrin ketone	53494-70-5	1.8	Residential RSL	1.5	0.0017	0.00011
Endrin aldehyde	7421-93-4	1.8	Residential RSL	1.5	0.0017	0.00009
alpha-Chlordane	5103-71-9	1.6	Residential RSL	1.5	0.0017	0.00011
gamma-Chlordane	5103-74-2	1.6	Residential RSL	1.5	0.0017	0.00024
Toxaphene	8001-35-2	0.0595	NCSSL	15	0.033	0.00730

¹List the type and source of the PAL used for each matrix specific analyte (e.g. Background, HH-MCL, HH-region III RBC, eco-WQC, eco-Region III BTAG, etc.)

²Laboratory-specific MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. MDLs may be subject to update.

SW846 8082—PCBs. Table 2-10 presents the TAL, PALs, and associated RLs for water and Table 2-11 presents the TAL, **project action limits**, and associated RLs for soil.

TABLE 2-10
TAL Reporting Limits SW-846 Method 8082 PCBs Water

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference ¹	Project Quantitation Limit Goal (µg/L)	Laboratory-specific ²	
					CRQLs (µg/L)	MDLs (µg/L)
Aroclor-1016	12674-11-2	0.26	RSL	0.5	0.5	0.084
Aroclor-1221	11104-28-2	0.0068	RSL	0.5	0.5	0.1
Aroclor-1232	11141-16-5	0.0068	RSL	0.5	0.5	0.14
Aroclor-1242	53469-21-9	0.034	RSL	0.5	0.5	0.14
Aroclor-1248	12672-29-6	0.034	RSL	0.5	0.5	0.16
Aroclor-1254	11097-69-1	0.034	RSL	0.5	0.5	0.13
Aroclor-1260	11096-82-5	0.034	RSL	0.5	0.5	0.088

¹List the type and source of the PAL used for each matrix specific analyte (e.g. Background, HH-MCL, HH-region III RBC, eco-WQC, eco-Region III BTAG, etc.)

²Laboratory-specific MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. MDLs may be subject to update.

TABLE 2-11
Target Analyte List Reporting Limits SW-846 Method 8082 PCBs Soil

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference ¹	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific ^{2,3}	
					CRQLs (mg/kg)	MDLs (mg/kg)
Aroclor-1016	12674-11-2	0.39	Residential RSL	0.39	0.017	0.0049
Aroclor-1221	11104-28-2	0.17	Residential RSL	0.17	0.017	0.0068
Aroclor-1232	11141-16-5	0.17	Residential RSL	0.17	0.017	0.0032
Aroclor-1242	53469-21-9	0.22	Residential RSL	0.22	0.017	0.0027
Aroclor-1248	12672-29-6	0.22	Residential RSL	0.22	0.017	0.005
Aroclor-1254	11097-69-1	0.11	Residential RSL	0.11	0.017	0.0061
Aroclor-1260	11096-82-5	0.22	Residential RSL	0.22	0.017	0.0048

¹List the type and source of the PAL used for each matrix specific analyte (e.g. Background, HH-MCL, HH-region III RBC, eco-WQC, eco-Region III BTAG, etc.)

²Laboratory-specific MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. MDLs may be subject to update.

USEPA SW-846 Method 6010B/7470A—Metals

Samples will be analyzed for select metals including mercury by inductively coupled plasma atomic emission spectrometry. All matrices—excluding filtered groundwater samples

but including groundwater, aqueous samples, soils, sludges, sediments, and other solid wastes—require digestion before analysis.

Table 2-12 presents the TAL, PALs, and associated RLs for water. **Table 2-13** presents the TAL, PALs, and associated RLs for soil.

TABLE 2-12
TAL List Reporting Limits SW-846 Method 6010B/7470A Metals Water

Analyte	CAS Number	Project Action Limit (µg/L)	Project Action Limit Reference ¹	Project Quantitation Limit Goal (µg/L)	Laboratory-specific ²	
					CRQLs (µg/L)	MDLs (µg/L)
Aluminum	7429-90-5	3700	RSL	200	200	26.7
Antimony	7440-36-0	1.5	RSL	10	20	3.9
Arsenic	7440-38-2	0.045	RSL	10	20	4.2
Barium	7440-39-3	730	RSL	200	5	0.16
Beryllium	7440-41-7	7.3	RSL	5	2	0.1
Cadmium	7440-43-9	1.75	NC2LGW	1	6	0.21
Calcium	7440-70-2	NC	N/A	5000	1000	56.2
Chromium	7440-47-3	11	RSL	10	5	0.63
Cobalt	7440-48-4	1.1	RSL	50	5	0.67
Copper	7440-50-8	150	RSL	25	10	1.4
Iron	7439-89-6	300	MCL & NC2LGW	100	150	15.4
Lead	7439-92-1	15	MCL & NC2LGW	3	10	1.2
Magnesium	7439-95-4	NC	N/A	5000	250	11.6
Manganese	7439-96-5	50	NC2LGW	15	5	0.19
Mercury	7439-97-6	1.05	NC2LGW	0.2	0.2	0.019
Nickel	7440-02-0	73	RSL	40	10	0.9
Potassium	7440-09-7	NC	N/A	5000	250	47.6
Selenium	7782-49-2	18	RSL	5	20	1.7
Silver	7440-22-4	17.5	NC2LGW	10	5	0.47
Sodium	7440-23-5	NC	N/A	5000	2500	255
Thallium	7440-28-0	0.24	RSL	2	30	4.1
Vanadium	7440-62-2	26	RSL	20	10	0.83
Zinc	7440-66-6	1050	NC2LGW	20	20	1.6

¹List the type and source of the PAL used for each matrix specific analyte (e.g. Background, HH-MCL, HH-region III RBC, eco-WQC, eco-Region III BTAG, etc.)

²Laboratory-specific MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. MDLs may be subject to update.

TABLE 2-13
Target Analyte List Reporting Limits SW-846 Method 6010B/7470A Metals Soil

Analyte	CAS Number	Project Action Limit (mg/kg)	Project Action Limit Reference ¹	Project Quantitation Limit Goal (mg/kg)	Laboratory-specific ^{2,3}	
					CRQLs (mg/kg)	MDLs (mg/kg)
Aluminum	7429-90-5	7700	Residential RSL	40	20	2.19
Antimony	7440-36-0	3.1	Residential RSL	2	6	0.24
Arsenic	7440-38-2	0.39	Residential RSL	2	1	0.26
Barium	7440-39-3	848	NCSSL	40	20	0.014
Beryllium	7440-41-7	3.38	NCSSL	1	0.5	0.0088
Cadmium	7440-43-9	0.95	NCSSL	0.7	0.5	0.023
Calcium	7440-70-2	NC	N/A	1000	500	5.52
Chromium	7440-47-3	27.2	NCSSL	2	1	0.059
Cobalt	7440-48-4	2.3	Residential RSL	10	5	0.072
Copper	7440-50-8	310	Residential RSL	5	2.5	0.14
Iron	7439-89-6	151	NCSSL	20	10	1.91
Lead	7439-92-1	270	NCSSL	1	1	0.17
Magnesium	7439-95-4	NC	N/A	1000	500	1
Manganese	7439-96-5	65.2	NCSSL	3	1.5	0.013
Mercury	7439-97-6	0.015	NCSSL	0.2	0.1	0.027
Nickel	7440-02-0	56.4	NCSSL	8	4	0.11
Potassium	7440-09-7	NC	N/A	1000	500	0.75
Selenium	7782-49-2	112.2	NCSSL	2	3.5	0.45
Silver	7440-22-4	0.217	NCSSL	2	1	0.034
Sodium	7440-23-5	NC	N/A	1000	500	13.3
Thallium	7440-28-0	0.51	Residential RSL	3	2.5	0.55
Vanadium	7440-62-2	55	Residential RSL	10	5	0.043
Zinc	7440-66-6	550	NCSSL	4	6	0.21

¹List the type and source of the PAL used for each matrix specific analyte (e.g. Background, HH-MCL, HH-region III RBC, eco-WQC, eco-Region III BTAG, etc.)

²Laboratory-specific MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. MDLs may be subject to update.

TABLE 2-14

USEPA SW-846 Method 8290 - Dioxin

This method provides procedures for the detection and quantitative measurement of polychlorinated dibenzo-p-dioxins in a variety of environmental matrices.

Target Analyte List Reporting Limits SW-846 Method 8290 Dioxin Soil

Analyte	CAS Number	Project Action Limit (ug/kg)	Project Action Limit Reference ¹	Project Quantitation Limit Goal (ug/kg)	Laboratory-specific ^{2,3}	
					CRQLs (ug/kg)	MDLs (ug/kg)
2,3,7,8-Tetrachlorodibenzo-p-dioxin	1746-01-6	.0145	NC SSL	.0100	0.001	0.000119

¹List the type and source of the PAL used for each matrix specific analyte (e.g. Background, HH-MCL, HH-region III RBC, eco-WQC, eco-Region III BTAG, etc.)

²Laboratory-specific MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method. MDLs may be subject to update.

2.2.2 Analytical Laboratory

The analytical laboratory contracted to provide analytical services to support this project is GPL Laboratories, LLLP. GPL Laboratory holds the applicable Navy and state certifications.

Detection, Quantitation and Reporting Limits

The RLs are listed in **Tables 2-4** through **2-13**.

The laboratory will supply analyte-specific quantification limits, with laboratory-specific MDL studies, as part of its laboratory CQAPP.

Method Detection Limits

The MDL is the minimum amount of an analyte that can be routinely identified using a specific method and instrument measured and reported with 99 percent confidence that the analyte concentration is greater than zero. MDLs are operationally determined as three times the standard deviation of seven replicate spiked samples run according to the complete method. However, the evaluation is routinely completed on reagent grade water. As a result, potentially significant matrix interferences that decrease analyte recoveries are not addressed.

Determine the MDL for each analyte as follows:

$$\text{MDL} = 3.14(s)$$

Where:

- s - The standard deviation for each analyte from the seven replicate analyses.
- 3.14 - The one-sided t-statistic at the 99 percent confidence level appropriate for determining the MDL using seven replicates.

When the concentration of concern (or project-specific action level) is greater than the MDL, to the extent that the confidence limits of both the MDL and concentration of concern do not overlap, then both “non-detect” and “detect” results can be used with confidence. There will be a possibility of false positives and false negatives if the confidence limits of the MDL and the concentration of concern overlap. When the concentration of concern is sufficiently less than the MDL that the confidence limits do not overlap, then there is a strong possibility of false negatives and only “detect” results are useable.

The laboratory will establish MDLs for each method, matrix, and analyte for each instrument the laboratory plans to use for the project. The laboratory will revalidate these MDLs at least once per 12-month period. The laboratory will provide the MDL at the beginning of the project. Project and laboratory specific MDLs will be included in the project-specific addendum.

40 CFR 136, Appendix B, or Chapter 1 of SW846 Methods, has not set frequency requirements for revalidating the MDLs. The MDLs be validated once per 12-month period.

Where multiple instruments are used, the MDL used for reporting purposes will represent the least sensitive instrument.

Instrument Detection Limit

The instrument detection limit (IDL) includes only the instrument portion of the detection, not sample preparation, concentration/dilution factors, or method-specific parameters. The IDL is operationally defined as three times the standard deviation of seven replicate analyses at the lowest concentration that is statistically different from a blank. This represents 99 percent confidence that the signal identified is the result of the presence of the analyte, not random noise. The IDL is not the same as the MDL. There is no formal procedure for IDL outside the USEPA Contract Laboratory Program (CLP) Statement of Work for inorganic analysis.

Quantitation Limits

The quantitation limit (QL) as defined in SW-846 methods, is the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The sample quantitation limit (SQL) is the QL adjusted to reflect sample-specific actions such as dilution or use of smaller aliquot sizes than prescribed in the method, or for percent moisture. These adjustments may be due to matrix effects or the high concentration of some analytes. The SQL is the more useful limit for data users such as risk assessors.

For the same chemical, the SQL in one sample may be higher than, lower than, or equal to the SQL values for other samples. In addition, preparation or analytical adjustments, such as dilution of the sample for quantitation high levels target and non-target analytes, could result in non-detects for other analytes included in the analysis, even though target analytes may have been present at trace quantities in the undiluted sample.

All results will be reported on a dry-weight basis.

Reporting Limits

The laboratories participating in this work effort will compare the results of the experimental MDLs to RLs for each analyte. The MDL may not be more than one-half the corresponding RL. The laboratories also will verify RLs by including a standard at the RL as the lowest point on the calibration curve. For methods that do not include the RL as the low point of the calibration curve, a RL verification standard will be analyzed immediately following calibration. The RL verification standard must include all target analytes. All results will be reported at or above the MDL values. No numerical results will be reported below the MDL; however, for those results falling between the MDL and the RL, a "J" flag will be applied to the results indicating the variability associated with the result.

Analytical SOPs

The laboratory has submitted a list of specific SOPs that will be used to perform off-site analysis of samples. Refer to **Table 2-15** for Analytical SOP References. Laboratory SOPs are provided in **Attachment A**.

TABLE 2-15
Analytical SOP References

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? ¹ (Y/N)
F.2	Sample Receipt, Inspection, Preservation and Storage Condition Requirements; Ver 19, 04/08	Definitive	All	N/A	GPL Laboratories, LLLP	N
F.3	Sample Logging and Record Keeping; Ver. 6, 02/03 (reviewed 09/08)	Definitive	All	N/A	GPL Laboratories, LLLP	N
G.12	SOP for Reports Generation; Ver. 7, 10/06 (reviewed 10/08)	Definitive	All	N/A	GPL Laboratories, LLLP	N
H.8	Acid Digestion of Aqueous Samples, EP and TCLP Extracts and Wastes that contain Suspended Solids for inductively coupled plasma (ICP) & ICPMS analysis; Ver. 13, 03/08	Definitive	GW	N/A	GPL Laboratories, LLLP	N
H.10	Trace ICP Quantitation for HSL Metals According to Method 6010B; Ver. 19, 11/08	Definitive	All	ICP	GPL Laboratories, LLLP	N
H.12	Cold Vapor Analysis for Mercury in Accordance with SW846 Methods 7470A and 7471B; Ver. 24, 11/08	Definitive	All	N/A	GPL Laboratories, LLLP	N
H.21	Acid Digestion of Soli, Sludge, Sediment and other Solid Waste samples for ICP & ICPMS by SW846 Method 3050B; Ver. 8, 08/07 (reviewed 11/08)	Definitive	Soils	N/A	GPL Laboratories, LLLP	N
J.4	Percent Solids Determination Procedure; Ver.8, 11/07 (reviewed 11/08)	Definitive	Soils	N/A	GPL Laboratories, LLLP	N

TABLE 2-15
Analytical SOP References

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? ¹ (Y/N)
M.5	Volatile Organics - 8260B; Ver.18, 08/07 (reviewed 10/08)	Definitive	All	GC/MS	GPL Laboratories, LLLP	N
N.6	Method 3520C, Continuous Liquid-Liquid Extraction for Pest/PCBs; Ver. 8, 06/08	Definitive	GW	N/A	GPL Laboratories, LLLP	N
N.7	Method 3550B, Ultrasonic Extraction for Pest/PCBs; Ver. 8, 09/02 (reviewed 06/08)	Definitive	Soils	N/A	GPL Laboratories, LLLP	N
N.11	Method 3520C, Continuous Liquid-Liquid Extraction for Semivolatile Organics; Ver. 8, 06/08	Definitive	GW	N/A	GPL Laboratories, LLLP	N
N.12	Soil Extraction for Semivolatile Organics by Method 3550B; Ver. 8, 10/02 (reviewed 06/08)	Definitive	Soils	N/A	GPL Laboratories, LLLP	N
P.5	SOP for SW827C -GC/MS Analysis of Semivolatile Organics; Ver. 15, 08/07 (reviewed 11/08)	Definitive	All	GC	GPL Laboratories, LLLP	N
Q.6	SOP for Method 8081B - Organochlorine Pesticides; Ver. 13, 10/08	Definitive	All	GC	GPL Laboratories, LLLP	N
Q.7	SOP for Method 8082A - Aroclor (PCB, PCT) and PCB Congeners; Ver. 11, 10/08	Definitive	All	N/A	GPL Laboratories, LLLP	N
DC41	8290 Data Analysis and Reporting, 3/28/08, version 17	Definitive	Soils	Micromass HRMS	SGS Wilmington	N
DC37	Sample Processing, 3/31/08, version 28	Definitive	Soils	N/A	SGS Wilmington	N

TABLE 2-15
Analytical SOP References

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work?¹ (Y/N)
MI3	Standard Operating Procedures for the Login and Storage of Samples, September 2007, version 9	Definitive	Soils	N/A	SGS Wilmington	N

¹ If yes, then specify the modification that has been made. Note that any analytical SOP modification made relative to project specific needs must be reviewed and approved by the Navy Quality Assurance Officer.

Data Package Deliverables

There are no data package requirements for Level I (screening results). The FTL is responsible for reviewing the field logbooks, which will contain the following information for Level I field screening results.

QC data package deliverables are summarized in **Table 2-16** by analytical fraction and will include sample results and QC summary forms, as well as raw instrument data. The laboratory will provide Level IV data packages for all analytical data associated with this project.

TABLE 2-16
Data Package Deliverables

All Analytical Fractions				
Case Narrative – A detailed case narrative per analytical fraction is required and will include explanation of any non-compliance and/or exceptions and corrective action. Exceptions will be noted for receipt, holding times, methods, preparation, calibration, blanks, spikes, surrogates (if applicable), and sample exceptions.				•
Sample ID Cross Reference Sheet (Lab IDs and Client IDs)				•
Completed Chain of Custody and any sample receipt information				•
Sample preparation (extraction/digestion) logs				•
Copies of non-conformance memos and corrective actions				•
Form *	GC/MS Organic Fractions	Level II	Level III	Level IV
1	Sample results	•	•	• + raw
2	Surrogate Recovery Summary (w/ applicable control limits)	•	•	•
3	MS/MSD Accuracy & Precision Summary **	•	•	• + raw
3	LCS Accuracy Summary	•	•	• + raw
4	Method Blank Summary	•	•	• + raw
5	Instrument Tuning Summary (including tuning summary for applicable initial calibrations)		•	•
6	Initial Calibration Summary (including concentration levels of standards)		•	• + raw
7	Continuing Calibration Summary		•	• + raw
8	IS Summary (including applicable initial calibrations)		•	•
Form *	GC/HPLC Organic Fractions	Level II	Level III	Level IV
1	Sample results	•	• ****	• + raw
2	Surrogate Recovery Summary (w/ applicable control limits)	•	•	•
3	MS/MSD Accuracy & Precision Summary **	•	•	• + raw
3	LCS Accuracy Summary	•	•	• + raw
4	Method Blank Summary	•	•	• + raw
6	Initial Calibration Summary (including concentration levels of standards) ***		•	• + raw
7	Continuing Calibration Summary ***		•	• + raw
7	Degradation Summary (Organochlorine Pesticides only) ***		•	• + raw
8	Analytical Sequence (including IS area performance where applicable) ***		•	•
10	Compound Identification Summary (where confirmation required) ***		•	•
Form *	Metals Inorganic Fractions	Level II	Level III	Level IV
1	Sample Results	•	•	• + raw
2A	Initial and Continuing Calibration Summary		•	• + raw
3	Initial and Continuing Calibration Blanks and Method Blanks Summary	•	•	• + raw
4	Interference Check Standard Summary		•	• + raw
5A	Pre-digestion Matrix Spike Recoveries Summary	•	•	• + raw
5B	Post-digestion Spike Recoveries Summary		•	• + raw
6	Native Duplicate or MS/MSD Precision Summary **	•	•	• + raw
7	LCS Recovery Summary	•	•	• + raw
8	Method of Standard Addition (if necessary)		•	• + raw
9	Serial Dilution		•	• + raw
10	Instrument or Method Detection Limit Summary		•	•
11	ICP Interelement Correction Factors		•	•
12	Linear Range Summary		•	•
13	Preparation Log Summary		•	• + raw
14	Analytical Run Sequence and GFAA Post-spike Recovery Summary		•	• + raw

* CLP Form or summary form with equivalent information

** with RPD calculated according to method specifications (CLP using % recovery, SW-846 using concentration)

*** including deliverables for primary and confirmation analysis (where applicable)

In addition, the laboratory must have the capability of providing the data package on compact disk (CD) in a scanned .pdf format. At this time, it is anticipated that the laboratory will provide one hardcopy data package and one CD to the PC, and one CD to the PM.

2.3 Quality Control Requirements

The following text describes this project's QC requirements.

2.3.1 Field QC Blank Samples and Duplicate Field Samples

The type and frequency of field QC samples should be evaluated as part of the project planning process. In the following subsections, typical field QC blank samples and duplicate field samples are defined.

Blank samples should not contain any target parameter of interest. There are certain organic compounds known to be common laboratory contaminants, such as acetone, methylene chloride, and the common phthalates. However, the laboratory must make all efforts to eliminate these compounds as contaminants. The concentration of all target compounds must be less than the RL, except for the common contaminants; the concentration of the common contaminants must be less than five times the RL.

2.3.2 TBs

TBs (TBs) are used to monitor potential VOC contamination introduced during sample shipping and handling. TBs are 40-mL VOC vials of ASTM Type II water, which are filled in the laboratory, transported to the sampling site, and returned to the laboratory with the VOC samples. TBs are prepared and analyzed for VOCs only; they should not be opened in the field. One TB will be included with each cooler containing samples for VOC analysis (aqueous and solid phase). No VOC analyses are being performed in this phase of the investigation, consequently; no TBs will be collected.

2.3.3 Equipment Rinsate Blank Samples

Equipment rinsate blanks (ERBs) are samples of ASTM Type II water passed through and over the surface of decontaminated sampling equipment. The rinse water is collected in sample bottles, preserved, and handled in the same manner that is used when collecting aqueous samples, even if the ERBs are being collected for soil samples. ERBs are used to monitor the effectiveness of the decontamination process. One ERB will be collected per day, per type of sampling equipment and analyzed for the same parameters as the corresponding samples.

2.3.4 Field/Decontamination Source Water Blanks

FBs are samples of the source water used for decontamination and steam cleaning. This blank is used to monitor potential contaminants present in the source water during field decontamination procedures. One FB will be collected for each source of water used for decontamination and analyzed for the same parameters as the corresponding samples. One FB will be collected per week while sampling.

2.3.5 Temperature Blanks

Temperature blanks are sent with each cooler shipped to the offsite laboratory containing samples requiring preservation at 4 °C. Temperature blanks consist of a non-preserved VOC vial, or similar laboratory container, filled with ASTM reagent grade water. Temperature blanks are measured at the laboratory upon receipt to verify the temperature of the samples contained in the cooler. One temperature blank will be shipped with each cooler to each offsite laboratory.

2.3.6 Duplicate Field Samples

Duplicate field samples are collected to monitor the precision of the field sampling process. The FTL will choose at least 10 percent (per matrix) of the total number of sample locations known or suspected to contain moderate contamination, and duplicate field samples will then be collected at these locations. The identity of the duplicate samples will be recorded in the field sampling logbook, and this information will be forwarded to the DQE team to aid in reviewing and evaluating the data. A control limit of ± 20 percent for the RPD will be used for original and duplicate concentrations greater than five times the RL in water matrices. A control limit of ± 35 percent for the RPD will be used for original and duplicate concentrations greater than five times the RL in soil matrices. A control limit of \pm the RL will be used for waters and \pm two times the RL for soils when concentrations are reported as less than five times the RL.

2.3.7 Laboratory Method/Preparation Blanks

Laboratory method blanks are blank matrices (such as ASTM Type II water or Ottawa sand) that are treated as environmental samples, being prepared and analyzed along with the field samples. Laboratory method blanks are used to monitor laboratory performance and to check for contamination introduced during the preparation and analytical procedures. A method blank is required for every 20 field samples or for each analytical batch, whichever is more frequent.

Blank samples should not contain any target parameter of interest. There are certain organic compounds known to be common laboratory contaminants, such as acetone, methylene chloride, and the common phthalates. However, the laboratory must make all efforts to eliminate these compounds as contaminants. The concentration of all target compounds must be less than the RL, except for the common contaminants; the concentration of the common contaminants must be less than five times the RL.

2.3.8 Matrix Spike/Matrix Spike Duplicate Samples

For MS/MSD samples, three aliquots of a single sample are analyzed: one native and two spiked with target compounds or metals. Spike recovery is used to evaluate potential matrix interferences, as well as accuracy. The duplicate spike results (MS and MSD) are compared to evaluate precision. MS/MSDs will be collected at a frequency of 5 percent (1 MS/MSD sample set for every 20 field samples) of the number of field samples. The MS/MSD accuracy limits are 75 to 125 percent for metals. The MS/MSD RPD limits for metals are 20 percent for waters and 35 percent for soils.

The laboratory MS/MSD accuracy and precision limits for the remaining analyses will be used as guidance when evaluating the data.

2.3.9 Surrogate Spikes

Surrogate spike compounds are added to each sample for the organic analytical methods. Surrogate spike compounds are structurally similar (but not identical) to target compounds and should behave in a similar manner during analysis. Surrogate spike recoveries are used to monitor both laboratory performance and matrix interferences. Surrogate spike recoveries from field and laboratory blanks are used to evaluate laboratory performance because these blanks represent an ideal sample matrix. Surrogate spike recoveries for field samples are used to evaluate the potential for matrix interferences. When surrogate spike recoveries for field samples fall outside the method target acceptance windows, the samples are re-extracted if appropriate, then re-analyzed. If the surrogate spike recovery is still outside the acceptance window for the re-analyzed sample, then the sample results are qualified as affected by matrix interferences.

2.3.10 Laboratory Control Spike Samples

The LCSs are analyte-free water (for aqueous analyses) or Ottawa sand (for soil analyses) (except metals where glass beads of 1-millimeter (mm) diameter or smaller may be used) spiked with all target analytes. The appropriate spiking concentration will be spiked at a level less than or equal to the midpoint of the calibration curve for each analyte.

The LCS will be carried through the complete sample preparation and analysis procedure. The LCS is used to evaluate each preparation and analytical batch and to determine if the method is in control. The LCS cannot be used as the continuing calibration verification. One LCS will be included in every preparation and analytical batch. If more than one LCS is analyzed in an analytical batch, results from all LCSs analyzed will be reported.

Whenever an analyte in a LCS is outside the acceptance limit, corrective action will be performed. After the system problems have been resolved and system control has been reestablished, all samples in the analytical batch will be reanalyzed for the out-of-control analyte(s). When an analyte in a LCS exceeds the upper or lower control limit and no corrective action is performed or the corrective action was ineffective, the laboratory should discuss the issue with the PC or QA personnel.

2.3.11 Interference Check Samples

The interference check sample (ICS), used in ICP analyses only, contains both interfering and analyte elements of known concentrations. The ICS is used to verify background and interelement correction factors and is run at the beginning and end of each run sequence.

When the ICS results are outside of the acceptance limits as prescribed in the method, corrective action will be performed. After the system problems have been resolved and system control has been re-established, re-analyze the ICS. If the ICS result is acceptable, re-analyze all affected samples.

2.3.12 Internal Standards

ISs are known amounts of certain compounds added after preparation or extraction of a sample. These compounds are used in an IS calibration method to correct sample results affected by column injection losses, purging losses, or viscosity effects. ISs will be added to environmental samples, control samples, and blanks in accordance with the method requirements.

When the IS results are outside of the acceptance limits, corrective actions will be performed. After the system problems have been resolved and system control has been reestablished, all samples analyzed while the system was malfunctioning will be reanalyzed.

2.3.13 Retention Time Windows

Retention time windows are established to compensate for minor shifts in absolute retention times resulting from normal chromatographic variability. Absolute retention times are used for analyte identification in all GC and HPLC methods that do not employ IS calibration. Retention time windows are used in GC and HPLC analysis for qualitative identification of analytes. They are calculated from replicate analyses of a standard on multiple days. If the analyte retention time is outside the established window, new retention windows must be established.

2.3.14 Confirmation of Identification

Quantitative confirmation of results at or above the RL for samples analyzed by GC or HPLC will be required and will be completed within the method-required holding times. For GC methods, a second column is used for confirmation. For HPLC methods, a second column or a different detector is used. The result from the lowest quantitation between the primary and secondary column/detector will be used for reporting purposes. The lowest quantitation will be reported to minimize the reporting of bias high results arising from co-elution of non-target analytes with the analyte of interest.

2.3.15 Standard Materials

Standard materials, including second source materials, used in calibration and to prepare samples will be traceable to National Institute of Standards and Testing (NIST), USEPA, American Association of Laboratory Accreditation (A2LA) or other equivalent approved source, if available. If an NIST, USEPA, or A2LA standard material is not available, the standard material proposed for use will be included in an addendum to the QAPP and approved before use. The standard materials will be current, and the following expiration policy will be followed: The expiration dates for ampulated solutions will not exceed the manufacturer's expiration date or 1 year from the date of receipt, whichever comes first. Expiration dates for laboratory-prepared stock and diluted standards will be no later than the expiration date of the stock solution or material or the date calculated from the holding time allowed by the applicable analytical method, whichever comes first. Expiration dates for pure chemicals will be established by the laboratory and be based on chemical stability, possibility of contamination, and environmental and storage conditions. Expired standard materials will be either revalidated prior to use or discarded. Revalidation may be

performed through assignment of a true value and error window statistically derived from replicate analyses of the material as compared to an unexpired standard. The laboratory will label standard and QC materials with expiration dates.

A second source standard is used to independently confirm initial calibration. A second source standard is a standard purchased from a different vendor than the vendor supplying the material used in the initial calibration standards. The second source material can be used for the continuing calibration standards or for the LCS (but will be used for one of the two). Two different lot numbers from the same vendor do not constitute a second source.

Table 2-17 presents Laboratory QC Samples.

TABLE 2-17
Laboratory QC Samples

Matrix	Groundwater					
Analytical Group	TCL VOCs					
Analytical Method/ SOP Reference	SW846 8260/GPL SOP M.5					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	Once per tune (12 hrs)	Recovery < 1/2 RL, except Methylene Chloride & Cyclohexane < 2.5x RL, and Acetone & 2-Butanone < 5x RL	Rinse sample pathway, Rerun	Nathan Krueger	Contamination/ Bias	Recovery < 1/2 RL, except Methylene Chloride & Cyclohexane < 2.5x RL, and Acetone & 2-Butanone < 5x RL
Matrix Spike and Matrix Spike Duplicate Analyses	Once each SDG, matrix within an SDG, or group of samples of similar concentration level (whichever is most frequent).	see attached recovery list (with allowable marginal exceedances)	Laboratory Judgment		Accuracy/ Precision	see attached recovery list (with allowable marginal exceedances)
ISs	Spiked in all analyses	Peak area must be within -50 to +100% of most recent CCV; Peak retention time must be within 30 seconds of most recent CCV RT.	Rerun affected analysis		System Performance	Peak area must be within -50 to +100% of most recent CCV; Peak retention time must be within 30 seconds of most recent CCV RT.
System Monitoring Compounds/ Surrogates Spike Duplicate		Percent Recoveries: 1,2-Dichloroethane-d4 70-120% 4-Bromofluorobenzene 75-120% Toluene-d8 80-122% 1,2-dichlorobenzene-d4 64-132%			Accuracy/Bias	Percent Recoveries: 1,2-Dichloroethane-d4 70-120% 4-Bromofluorobenzene 75-120% Toluene-d8 80-122% 1,2-dichlorobenzene-d4 64-132% Control Limits

Matrix	Surface Soil Subsurface Soil					
Analytical Group	TCL VOCs					
Analytical Method/ SOP Reference	SW846 8260B/GPL SOP M.5					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	Once per tune (12 hrs)	Recovery < 1/2 RL, except Methylene Chloride & Cyclohexane < 2.5x RL, and Acetone & 2-Butanone < 5x RL	Rinse sample pathway, Rerun	Nathan Krueger	contamination, bias	Recovery < 1/2 RL, except Methylene Chloride & Cyclohexane < 2.5x RL, and Acetone & 2-Butanone < 5x RL
Matrix Spike and Matrix Spike Duplicate Analyses	Once each SDG, matrix within an SDG, or group of samples of similar concentration level (whichever is most frequent).	see attached recovery list (with allowable marginal exceedances)	Laboratory Judgment		accuracy, precision	see attached recovery list (with allowable marginal exceedances)
ISs	Spiked in all analyses	Peak area must be within -50 to +100% of most recent CCV; Peak retention time must be within 30 seconds of most recent CCV RT.	Rerun affected analysis		system performance	Peak area must be within -50 to +100% of most recent CCV; Peak retention time must be within 30 seconds of most recent CCV RT.
System Monitoring Compounds/ Surrogates	Spiked in all analyses	Percent recoveries: 1,2-Dichloroethane-d4 65-125% 4-Bromofluorobenzene 85-120% Toluene-d8 85-115% 1,2-dichlorobenzene-d4 65-123%	Rerun affected analysis		accuracy, bias	Percent recoveries: 1,2-Dichloroethane-d4 65-125% 4-Bromofluorobenzene 85-120% Toluene-d8 85-115% 1,2-dichlorobenzene-d4 65-123%

Matrix	Groundwater					
Analytical Group	TCL SVOCs					
Analytical Method/ SOP Reference	SW846 8270C/GPL SOPs N.11, P.5					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	Each extraction batch	All target analytes < CRQL, except the phthalate esters, which must be < 5x the CRQL	Re-extract the batch if any failing analyte is also present in any field sample.	Hall Moore	Contamination	All target analytes < CRQL, except the phthalate esters, which must be < 5x the CRQL
LCS		All ISs with +/- 30seconds of the corresponding CCV RT. All surrogates within +/- 0.06 RRT units of the corresponding CCV. Analyte recoveries should meet the attached recovery limits (with allowable marginal exceedances)	Re-analyze. If the re-analysis fails, re-extract the batch.		Extraction efficiency	All ISs with +/- 30seconds of the corresponding CCV RT. All surrogates within +/- 0.06 RRT units of the corresponding CCV. Analyte recoveries should meet the attached recovery limits (with allowable marginal exceedances)
MS / MSD		One set per 20 field samples extracted	All ISs with +/- 30seconds of the corresponding CCV RT. All surrogates within +/- 0.06 RRT units of the corresponding CCV. Analyte recoveries should meet the attached recovery limits (with allowable marginal exceedances)		Re-analyze.	Precision and Accuracy

Matrix	Groundwater					
Analytical Group	TCL SVOCs					
Analytical Method/ SOP Reference	SW846 8270C/GPL SOPs N.11, P.5					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate spike	All field and QC samples	Relative retention time of each surrogate must be within 0.06 RRT unite of the corresponding CCV RT. No Surrogate recovery <10% and no more than one Acid surrogate and one Base/Neutral Surrogate outside the following limits. Nitrobenzene-d5(B/N): 20-137 2-Fluorobiphenyl(B/N): 23-110 p-Terphenyl-d14(B/N): 20-138 Phenol-d5(A): 29-133 2-Fluorophenol(A): 20-118 2,4,6-Tribromophenol(A): 24-177	Re-analyze. If the re-analysis still fails, re-extract unless the failing surrogate recoveries are high and the affected sample(s) show no target compounds.	Hall Moore	Extraction efficiency	Relative retention time of each surrogate must be within 0.06 RRT unite of the corresponding CCV RT. No Surrogate recovery <10% and no more than one Acid surrogate and one Base/Neutral Surrogate outside the following limits. Nitrobenzene-d5(B/N): 20-137 2-Fluorobiphenyl(B/N): 23-110 p-Terphenyl-d14(B/N): 20-138 Phenol-d5(A): 29-133 2-Fluorophenol(A): 20-118 2,4,6-Tribromophenol(A): 24-177
Internal Standard spike		RT of each Internal Standard must be within +/- 30 seconds of the corresponding CCV RT. The EICP area of each IS must be within -50% to +100% of the corresponding CCV IS area.	Re-analyze. If the re-analysis fails, report results from both analyses		Instrument performance	RT of each Internal Standard must be within +/- 30 seconds of the corresponding CCV RT. The EICP area of each IS must be within -50% to +100% of the corresponding CCV IS area.

Matrix	Surface Soil Subsurface Soil					
Analytical Group	TCL SVOCs					
Analytical Method/ SOP Reference	SW846 8270C/GPL SOPs N.12, P.5					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	Each extraction batch	All target analytes < CRQL, except the phthalate esters, which must be < 5x the CRQL	Re-extract the batch if any failing analyte is also present in any field sample.	Hall Moore	All target analytes < CRQL, except the phthalate esters, which must be < 5x the CRQL	Re-extract the batch if any failing analyte is also present in any field sample.
LCS	Each extraction batch	All ISs with +/- 30seconds of the corresponding CCV RT. All surrogates within +/- 0.06 RRT units of the corresponding CCV. Analyte recoveries should meet the attached recovery limits (with allowable marginal exceedances)	Re-analyze. If the re-analysis fails, re-extract the batch.	Hall Moore	All ISs with +/- 30seconds of the corresponding CCV RT. All surrogates within +/- 0.06 RRT units of the corresponding CCV. Analyte recoveries should meet the attached recovery limits (with allowable marginal exceedances)	Re-analyze. If the re-analysis fails, re-extract the batch.

Matrix	Surface Soil Subsurface Soil					
Analytical Group	TCL SVOCs					
Analytical Method/ SOP Reference	SW846 8270C/GPL SOPs N.12, P.5					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS / MSD	One set per 20 field samples extracted	All ISs with +/- 30seconds of the corresponding CCV RT. All surrogates within +/- 0.06 RRT units of the corresponding CCV. Analyte recoveries should meet the attached recovery limits (with allowable marginal exceedances)	Re-analyze.	Hall Moore	All ISs with +/- 30seconds of the corresponding CCV RT. All surrogates within +/- 0.06 RRT units of the corresponding CCV. Analyte recoveries should meet the attached recovery limits (with allowable marginal exceedances)	Re-analyze.

Matrix	Surface Soil Subsurface Soil					
Analytical Group	TCL SVOCs					
Analytical Method/ SOP Reference	SW846 8270C/GPL SOPs N.12, P.5					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate spike	All field and QC samples	Relative retention time of each surrogate must be within 0.06 RRT unite of the corresponding CCV RT. No Surrogate recovery <10% and no more than one Acid surrogate and one Base/Neutral Surrogate outside the following limits. Nitrobenzene-d5(B/N): 34-113 2-Fluorobiphenyl(B/N): 24-120 p-Terphenyl-d14(B/N): 20-152 Phenol-d5(A): 30-119 2-Fluorophenol(A): 33-110 2,4,6-Tribromophenol(A): 23-160	Re-analyze. If the re-analysis still fails, re-extract unless the failing surrogate recoveries are high and the affected sample(s) show no target compounds.	Hall Moore	Relative retention time of each surrogate must be within 0.06 RRT unite of the corresponding CCV RT. No Surrogate recovery <10% and no more than one Acid surrogate and one Base/ Neutral Surrogate outside the following limits. Nitrobenzene-d5(B/N): 34-113 2-Fluorobiphenyl(B/N): 24-120 p-Terphenyl-d14(B/N): 20-152 Phenol-d5(A): 30-119 2-Fluorophenol(A): 33-110 2,4,6-Tribromophenol(A): 23-160	Re-analyze. If the re-analysis still fails, re-extract unless the failing surrogate recoveries are high and the affected sample(s) show no target compounds.

Matrix	Groundwater					
Analytical Group	TCL Pesticides					
Analytical Method/ SOP Reference	SW846 8081A/GPL SOPs N.6, Q.6					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	Each extraction batch	All target analytes < CRQL, surrogate recoveries must be within 30-150%	Re-extract the batch if any failing analyte is also present in any field sample.	Rehka Patel	contamination	All target analytes < CRQL, surrogate recoveries must be within 30-150%
PEM – Breakdown Check	Daily	Breakdown of 4,4'-DDT and Endrin must be less than 15%	Clip column, clean injection port and reanalyze		contamination	<15% breakdown of 4,4' DDT and Endrin
LCS	Each extraction batch	Surrogates must be within established RT windows. Target analytes should meet the attached recovery limits	Re-analyze. If the re-analysis fails, re-extract the batch.		extraction efficiency	Surrogates must be within established RT windows. Target analytes should meet the attached recovery limits
MS / MSD	One set per 20 field samples	Surrogates must be within established RT windows. Target analytes should meet the attached recovery limits	Re-analyze.		precision and accuracy	Surrogates must be within established RT windows. Target analytes should meet the attached recovery limits
Surrogate Spike	All field and QC samples	Surrogates must fall within established RT windows. Surrogate recoveries should meet recovery limits: decachlorobiphenyl 20-112%, tetrachloro-m-xylene 43-113%	Re-analyze. If the re-analysis still fails, re-extract unless the failing surrogate recoveries are high and the affected sample(s) show no target compounds.		extraction efficiency	Surrogates must fall within established RT windows. Surrogate recoveries should meet recovery limits: decachlorobiphenyl 20-112%, tetrachloro-m-xylene 43-113%

Matrix	Surface Soil Subsurface Soil					
Analytical Group	TCL Pesticides					
Analytical Method/ SOP Reference	SW846 8081A/GPL SOPs N.7, Q.6					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	Each extraction batch	All target analytes < CRQL, surrogate recoveries must be within 30-150%	Re-extract the batch if any failing analyte is also present in any field sample.	Rehka Patel	contamination	All target analytes < CRQL, surrogate recoveries must be within 30-150%
LCS	Each extraction batch	Surrogates must be within established RT windows. Target analytes should meet the attached recovery limits.	Re-analyze. If the re- analysis fails, re-extract the batch.		extraction efficiency	Surrogates must be within established RT windows. Target analytes should meet the attached recovery limits.
PEM – Breakdown Check	Daily	Breakdown of 4,4'-DDT and Endrin must be less than 15%	Clip column, clean injection port and reanalyze		contamination	Breakdown of 4,4'- DDT and Endrin must be less than 15%
MS / MSD	One set per 20 field samples	Surrogates must be within established RT windows. Target analytes should meet the attached recovery limits.	Re-analyze.		precision and accuracy	Surrogates must be within established RT windows. Target analytes should meet the attached recovery limits.

Matrix	Groundwater					
Analytical Group	TCL Pesticides / PCBs					
Analytical Method/SOP Reference	SW846 8082 / GPL SOPs N.6, Q.7					
QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	Each extraction batch	All target analytes < CRQL, surrogate recoveries must be within 30-150%	Re-extract the batch if any failing analyte is also present in any field sample.	Rehka Patel	contamination	All target analytes < CRQL, surrogate recoveries must be within 30-150%
LCS	Each extraction batch	Surrogates must be within established RT windows. Target analytes should meet the following advisory recovery limits [Analyte : lower-upper]: Ar-1016 : 35-145 Ar-1260 : 30-145	Re-analyze. If the re-analysis fails, re-extract the batch.		extraction efficiency	Surrogates must be within established RT windows. Target analytes should meet the following advisory recovery limits [Analyte : lower-upper]: Ar-1016 : 35-145 Ar-1260 : 30-145
MS / MSD	One set per 20 field samples	Surrogates must be within established RT windows. Target analytes should meet the following advisory recovery and RPD limits [Analyte : lower-upper, RPD]: Ar-1016 : 35-145, 20 Ar-1260 : 30-145, 20	Re-analyze.		precision and accuracy	Surrogates must be within established RT windows. Target analytes should meet the following advisory recovery and RPD limits [Analyte : lower-upper, RPD]: Ar-1016 : 35-145, 20 Ar-1260 : 30-145, 20
Surrogate Spike	All field and QC samples	Surrogates must fall within established RT windows. Surrogate recoveries should meet recovery limits: Decachlorobiphenyl 16-148%, Tetrachloro-m-xylene 21-129%	Re-analyze. If the re-analysis still fails, re-extract unless the failing surrogate recoveries are high and the affected sample(s) show no target compounds.		extraction efficiency	Surrogates must fall within established RT windows. Surrogate recoveries should meet recovery limits: Decachlorobiphenyl 16-148%, Tetrachloro-m-xylene 21-129%

Matrix	Surface Soil Subsurface Soil					
Analytical Group	TCL PCBs					
Analytical Method/ SOP Reference	SW846 8082 / GPL SOPs N.7, Q.7					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	Each extraction batch	All target analytes < CRQL, surrogate recoveries must be within 30-150%	Re-extract the batch if any failing analyte is also present in any field sample.	Rehka Patel	contamination	All target analytes < CRQL, surrogate recoveries must be within 30-150%
LCS	Each extraction batch	Surrogates must be within established RT windows. Target analytes should meet the following advisory recovery limits [Analyte : lower-upper]: Ar-1016 : 40-140 Ar-1260 : 60-130	Re-analyze. If the re- analysis fails, re-extract the batch.		extraction efficiency	Surrogates must be within established RT windows. Target analytes should meet the following advisory recovery limits [Analyte : lower-upper]: Ar-1016 : 40-140 Ar-1260 : 60-130
PEM – Breakdown Check	Daily	Breakdown of 4,4'-DDT and Endrin must be less than 15%	Clip column, clean injection port and reanalyze		contamination	Breakdown of 4,4'- DDT and Endrin must be less than 15%
MS / MSD	One set per 20 field samples	Surrogates must be within established RT windows. Target analytes should meet the following advisory recovery limits [Analyte : lower-upper]: Ar-1016 : 40-140 Ar-1260 : 60-130	Re-analyze.		precision and accuracy	Surrogates must be within established RT windows. Target analytes should meet the following advisory recovery limits [Analyte : lower- upper]: Ar-1016 : 40-140 Ar-1260 : 60-130

Matrix	Groundwater					
Analytical Group	TAL Metals					
Analytical Method/ SOP Reference	SW846 6010B, 7470A/ GPL SOPs H.8, H.10, H.12					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per batch of 20	concentration < R.L.	Conditional Re-digestion	Kelvin Choi	contamination, bias	concentration < R.L.
LCS		80-120% Recovery	Re-digestion		accuracy, bias	80-120% Recovery
Sample Duplicate		RPD < 20%	Qualify data		accuracy, precision	RPD < 20%
MS		75-125% Recovery	Qualify data		accuracy, precision	75-125% Recovery

Matrix	Surface Soil Subsurface Soil					
Analytical Group	TAL Metals					
Analytical Method/ SOP Reference	SW846 6010B, 7471B/ GPL SOPs H.21, H.10, H.12					
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per batch of 20	concentration < R.L.	Conditional Re-digestion	Kelvin Choi	contamination, bias	concentration < R.L.
LCS	1 per batch of 20	80-120% Recovery	Re-digestion		accuracy, bias	80-120% Recovery
Sample Duplicate	1 per batch of 20	RPD < 20%	Qualify data		accuracy, precision	RPD < 20%
MS	1 per batch of 20	75-125% Recovery	Qualify data		accuracy, precision	75-125% Recovery

Matrix	Surface Soil Subsurface Soil					
Analytical Group	Dioxins/ Furans					
Analytical Method/SOP Reference	SW-846 8290/ DC41					
QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
OPR (LCS)	1 per 20 samples	70-130 % recovery	Re-extract batch if low and detected. Evaluate data quality for high and non-detected.	Jim Plassard, SGS Wilmington	accuracy, bias	70-130 % recovery
OPRD (LCSD)	2 per 20 samples	70-130% recovery, ≤ 20% RSD.	If needed and fails, re-extract batch.		accuracy, bias	70-130% recovery, ≤ 20% RSD.
Lab Duplicate	1 per 20 samples	difference < 25%	Qualify data		accuracy, precision	difference < 25%
Extraction Standards (ES)	Every sample must receive ES before extraction.	40-135%.	Evaluate data quality, and if needed re-extract sample.		extraction efficiency	40-135%.
Method Blank	One per extraction batch.	PCDD/F < SOP RL or <10% of level in sample.	Affected samples must be re-extracted.		contamination, bias	PCDD/F < SOP RL or <10% of level in sample.

2.4 Laboratory Corrective Action

The procedures that will be followed in identifying problems and performing corrective actions in the laboratory are described below.

2.4.1 Field Corrective Action

The task manager is responsible for overseeing the corrective action process, but any team member may initiate it. The corrective action process consists of identifying a problem, acting to eliminate the problem, monitoring the effectiveness of the corrective action, verifying that the problem has been eliminated, and documenting the corrective action.

Examples of corrective action are correcting chain-of-custody forms; problems associated with sample collection, packaging, shipping, or field record keeping; or additional training in sampling and analysis. Additional approaches may include re-sampling or evaluating and amending sampling procedures.

2.4.2 Laboratory Corrective Action

The laboratory department supervisors will review the data generated to verify that all QC samples have been run as specified in the procedure. Laboratory personnel are alerted that corrective actions may be necessary under the following conditions:

- QC data are outside the warning or acceptable windows for precision and accuracy established for laboratory samples.
- Blanks contain contaminants at concentrations above the levels specified in the laboratory QA plan for any target compound.
- Deficiencies are detected by the laboratory QA director during internal or external audits, or from the results of performance evaluation samples.

Corrective actions are implemented immediately when non-conformances in QC sample results are identified by the bench analyst. Corrective action procedures are handled initially at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors and checks such parameters as instrument calibration, spike and calibration mixes, and instrument sensitivity.

The analyst immediately notifies his or her supervisor of the problem and the investigation being conducted. If the problem persists or cannot be identified, the matter must be referred to the laboratory supervisor and the QA/QC officer for further investigation. At this point, the PC and the PM must be notified about the nonconformance. All laboratory QC problems that will affect the final data must be discussed with the PC as part of the corrective action process. Once resolved, full documentation of the corrective action procedure must be filed with the laboratory supervisor, and the QA/QC officer must be provided with a corrective action memorandum for inclusion in the project file if data are affected. A copy of the corrective action memorandum must be included in the laboratory data package deliverable.

Corrective actions may include the following:

- Reanalyzing suspect samples
- Recalibration with new standards
- Eliminating blank contamination
- Resampling and analyzing new samples
- Evaluating and amending sampling and analytical procedures
- Accepting data with an acknowledged level of uncertainty
- Recalibrating analytical instruments
- Qualifying or rejecting the data

After implementation of the required corrective action measures, data that are deemed unacceptable may not be accepted by the PM, and follow-up corrective actions may be explored. Details of laboratory corrective actions are provided in the laboratory Comp QAM.

2.4.3 Laboratory Equipment

Laboratory instruments will be calibrated in accordance with the manufacturer's directions and applicable method specifications. Laboratory instrument calibration procedures will be summarized in the laboratory CQAPP, which will be reviewed and approved by the PM or designee before samples are submitted for analysis. The calibration of all laboratory equipment will be documented in the specific maintenance logbook, or analytical logbook, as described in the laboratory's CQAPP.

Analytical instruments will be calibrated in accordance with the analytical methods (**Table 2-18**). All target analytes reported will be present in the initial and continuing calibrations. All results reported will be within the calibration range. Records of standard preparation and instrument calibration will be maintained. Records will unambiguously trace the preparation of standards and their use in calibration and quantitation of sample results. Calibration standards will be traceable to standard materials.

TABLE 2-18
Analytical Instrument Calibration

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference ^a
Trace ICAP 61E	Analyte-specific curve using 4 cal standard w/ increasing concentration	Daily	0.9950-0.9999 Correlation Coefficient		Kelvin Choi, GPL Laboratories	H10
Trace ICAP 6500						H12
Hydra AA Mercury analyzer	Analyte-specific curve using 6 cal standard w/ increasing concentration					
GC-MS	Minimum 5 point ICAL	Prior to an analysis. Recalibrate as needed.	Relative standard deviation (RSD) \leq 30.0% for CCCs and \leq 15% for non-CCCs. A linear fit ($r \Rightarrow 0.995$, 5 points minimum) or quadratic fit ($r^2 \Rightarrow 0.99$, minimum 6 points) may be used for non-CCCs where the RSD is $> 15\%$. One or more non-CCCs may fail calibration criteria as long as the average RSD for the non-CCCs is $\leq 15\%$ and the failing analyte(s) are not detected in the associated field samples. Average response factor (RF) for the SPCCs must be $\Rightarrow 0.05$. Average RF for non-SPCCs must be $\Rightarrow 0.01$.	Recalibrate	Hall Moore, Mona Abdelmeguid, GPL Laboratories	P.5
	CCV	At the start of each 12-hour analysis period, unless full ICAL is performed	RF for the SPCCs must be $\Rightarrow 0.05$. RF for non-SPCCs must be $\Rightarrow 0.01$. %D for CCCs within $\pm 20\%$; %D for non-CCCs within $\pm 25\%$. For non-CCCs with a %D $< -25\%$ (indicating elevated sensitivity), the calibration is acceptable if the affected analyte is not detected in the associated field samples.	Perform injection port maintenance and repeat. If a passing CCV cannot be obtained, a new ICAL is required.	Hall Moore, Mona Abdelmeguid, GPL Laboratories	P.5

TABLE 2-18
Analytical Instrument Calibration

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference ^a
GC-ECD	ICAL	Prior to analysis. Recalibrate as needed.	%RSD for each single-component target compound $\leq 20.0\%$, except alpha-BHC and delta-BHC are $\leq 25.0\%$. Up to two of these analytes may exceed these criteria, but must be $\leq 30.0\%$. %RSD for the surrogates must be $\leq 30.0\%$	Recalibrate	Rehka Patel, Matt Clark GPL Laboratories	Q.6, Q.7
	CCV	Every twelve hours. Alternating Instrument Blank and PEM with Instrument Blank, INDAM, and INDBM	Instrument Blanks: Surrogates must be within the established RT windows. All pesticide target analytes must be $< 1/2$ the CRQL. PEM, INDAM, INDBM: 75%-125% recovery for all analytes. PEM: Endrin and DDT breakdown each $\leq 20\%$ and total breakdown $\leq 30\%$	Perform injection port maintenance and repeat. If a passing CCV cannot be obtained, a new ICAL is required.		
GC/MS	5 point initial calibration	Initial Calibration prior to sample analysis	Average RF for Chlorobenzene & 1,1,2,2-Tetrachloroethane ≥ 0.30 ; Chloromethane, bromoform, & 1,1-Dichloroethane ≥ 0.1 ; %RSD for Vinyl Chloride, 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, & Ethylbenzene $\leq 30\%$; All other compounds: RSD $\leq 15\%$, $r \geq 0.995$ (linear regression), or $r^2 \geq 0.99$ (quadratic regression).	Correct Problem and Rerun	Nathan Krueger, GPL Laboratories	M.5
	CCV	Once every tune (12 hours)	Average RF for Chlorobenzene & 1,1,2,2-Tetrachloroethane ≥ 0.30 ; Chloromethane, bromoform, & 1,1-Dichloroethane ≥ 0.1 ; %D for Vinyl Chloride, 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, & Ethylbenzene $\leq 20\%$	Correct Problem and Rerun, recalibrate if necessary		
Micromass HRMS	5 Point calibration	After major maintenance or every 6 months.	$\pm 30\%$ RSD for all 17 congeners	Instrument repair and reanalyze curve.	Jim Plassard, SGS Environmental	DC41

TABLE 2-18
Analytical Instrument Calibration

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference ^a
	ICAL	As needed to maintain acceptable CCAL, after major maintenance, or at a minimum one per year.	20% RPD for native and 30% RPD for labeled species.	An acceptable ICAL must be established before sample reporting may begin.		
	CCAL	Pre and post sample analysis within 12h.	20/30%. Average CCALs if no more than 2 unrelated compounds above 20/30% but less than 25/35% on the back end.	Acceptable bracketing CCALs must be established before sample reporting may begin.		

¹ Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

² Name or title of responsible person may be used.

Table 2-19 describes the procedures for analytical instrument maintenance, testing, and inspections.

TABLE 2-19
Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person ²	SOP Reference ¹
ICAP Trace Analyzer 6500	Clean torch, clean tubing	Calibration Daily	See SOP	Daily	Coellation Coefficient 0.995-0.9999	Recalibrate	Kelvin Choi	H.10
Hydra AA Mercury Analyzer	Gas separator and tubing cleaned							H12
GC/MS	Change Septum and liner, trim column, clean source	Calibration Check			Acceptable Chromatography	Repeat	Nathan Krueger/Hall Moore/ Rekha Patel	M.5, P.5
GC/ECD								A.6, Q.7
Micromass HRMS	Injection port main, column clip, source cleaning weekly	Calibration	CCAL analysis	See SOP DC41	meets CCAL criteria, meets curve criteria	Place service call	Jim Plassard, SGS	DC41

¹ Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

² Name or title of responsible person may be used.

Instrument calibration will be checked using all of the target analytes. This applies equally to multi-response analytes. All calibration criteria will satisfy SW-846, Update III requirements at a minimum. The initial calibration will be checked at the frequency specified in the method using materials prepared independently of the calibration standards. Multipoint calibrations will contain the minimum number of calibration points specified in the applicable method including a standard at or below the corresponding RL. Analyte concentrations are determined with either calibration curves or response factors (RFs). For GC and GC/MS methods, when using RFs to determine analyte concentrations, the average RF from the initial five-point calibration will be used. The continuing calibration will not be used to update the RFs from the initial five-point calibration. The continuing calibration verification cannot be used as the LCS.

If more than the required minimum number of standard concentrations is used in the initial calibration, all standard concentrations must be included in calculating the acceptance of the initial curve. All results for field samples will be reported only within the calibration linearity range.

2.5 Inspection and Acceptance Requirements for Supplies and Consumables

All services, including subcontracted services and supplies received from vendors, must meet the project scope, specified levels of quality, and the submittal schedule. Field and laboratory personnel must evaluate the vendor's ability to provide the services and specify acceptance requirements for supplies and consumables. For example, laboratories rely on suppliers for solvents, gases, consumables, and analytical equipment, including instrument maintenance. The laboratory should have and maintain adequate contracts with its vendors to receive uninterrupted supplies, parts, and services.

2.6 Data Acquisition Requirements

In addition to the electronic data, the laboratory provides hard-copy deliverables of the analytical results. Upon receipt, the data packages are prepared for data validation. After the data validation has been completed and a DQE report has been written, the hard-copy data packages are filed onsite until the project is completed. At that time, the data packages are sent to the PM for inclusion into the project files. Alternatively, the hard-copy data packages are stored at an offsite warehouse for a period of 10 years after the project close out.

Laboratory Assessment and Oversight

Assessment and oversight activities are performed to determine whether the QC measures identified in the SAP and in this QAPP are being implemented and documented as required. Audits and reviews are the tools used to implement this process. During an audit or review, the auditor may check for:

- Adherence to the QAPP
- Documentation of the process or system
- Proper identification, resolution, and documentation of nonconformance with the process or system
- Correction of identified deficiencies

3.1 Assessments and Response Actions

The laboratory will be audited in accordance with the Navy requirements. One laboratory audit will be performed before the receipt of samples at the laboratory.

3.1.1 Laboratory Performance and System Audits

Laboratory systems will be audited in accordance with the project-specific requirements. Contracted laboratories must submit a laboratory CQAPP. The CQAPP must reference relevant SOPs and the laboratory's internal procurement policies and corrective action program.

The laboratory audit will address at least the following issues:

- Is the laboratory operation being performed as required by the subcontract?
- Are internal laboratory operations being conducted in accordance with the laboratory CQAPP?
- Are the laboratory analyses being performed in accordance with method requirements?

Any nonconformance noted during an audit will result in a corrective action.

3.2 Reports to Management

Reports to the PM include project status reports, the results of evaluation and system audits, data quality assessments, and significant QA problems and recommended solutions. The status reports, submitted in accordance with the requirements of the site-specific WP, will discuss at least current activities, problems encountered and their resolution, and planned work.

QA reports will be submitted in accordance with the site-specific SAP. QA reports document implementation of the QAPP and the results of the site-specific QA/QC audits. A final QA report must be submitted as part of each project's final report. The topics to be covered are outlined in the site-specific WP, but each will include at least the following information:

- Identification of nonconformances that required corrective action and resolution of the nonconformance
- Data quality assessment in terms of precision and accuracy and how they affect the usability of the analytical results
- Limitations of the qualified results and a discussion of rejected results
- Discussion of the field and laboratory QA/QC sample results
- The results of external laboratory audits

The FTL will provide feedback to the PM discussing all field activities, changes to field procedures, problems encountered, and corrective actions taken.

Data Validation and Usability

This subsection addresses the QA activities that occur after the data collection has been completed. Implementation of these elements, which include data review, validation, and reconciliation to DQOs, will determine the extent to which the data conform to the specified criteria and satisfy the project objectives.

4.1 Data Review, Validation, and Verification Requirements

Data review and validation are processes whereby data generated in support of this project are reviewed against the QA/QC requirements. The data are evaluated for precision, accuracy, and completeness against the analytical protocol requirements. Non-conformances or deficiencies that could affect the usability of data are identified as noted. The types of data that will be validated are described further in the following subsections.

All analytical data will be supported by a data package. The data package will contain the supporting QC data for the associated field samples. Before the laboratory will release each data package, the laboratory QAM (or the analytical section supervisor) must carefully review the sample and laboratory performance QC data to verify sample identity, the completeness and accuracy of the sample and QC data, and compliance with method specifications.

4.2 Verification and Validation Methods

4.2.1 Data Verification

Before the analytical results are released by the laboratory, both the sample and QC data will be reviewed carefully to verify sample identity, instrument calibration, detection limits, dilution factors, numerical computations, accuracy of transcriptions, and chemical interpretations. Additionally, the QC data will be reduced and spike recoveries will be included in control charts, and the resulting data will be reviewed to ascertain whether they are within the laboratory-defined limits for accuracy and precision. Any non-conforming data will be discussed in the data package cover letter and case narrative. The laboratory will retain all of the analytical and QC documentation associated with each data package.

As discussed previously, the data are also verified to assess whether the electronic data deliverables (EDDs) and the hard-copy data deliverables are consistent with one another to ensure an accurate database.

4.2.2 Data Validation

One hundred percent of the laboratory Level IV data reporting packages will be validated.

The data package will be validated by a third-party validator. Analytical methods and laboratory SOPs as presented in this SAP will be used to evaluate compliance against

QA/QC criteria. Should adherence to QA/QC criteria yield deficiencies, data may be qualified. The data qualifiers that may be used are those presented in *National Functional Guidelines for Organic Data Review* (October 1999) and *National Functional Guidelines for Inorganic Data Review* (October 2004). National Functional Guidelines will not be used for data validation; however, the specific qualifiers listed therein may be applied to data should non-conformances against the QA/QC criteria as presented in this SAP be identified. The data review and validation process is independent of the laboratory's checks; it focuses on the usability of the data to support the project data interpretation and decision-making process.

Sample results that do not meet the acceptance limit criteria will be indicated with a qualifying flag, which is a one or two-letter abbreviation that indicates a possible problem with the data. Flags used in the text may include the following:

- **U** Undetected. Samples were analyzed for this analyte, but it was not detected above the MDL or IDL.
- **UJ** Detection limit estimated. Samples were analyzed for this analyte, but the results were qualified as not detected. The result is estimated.
- **J** Estimated. The analyte was present, but the reported value may not be accurate or precise.
- **R** Rejected. The data are unusable. (Note: Analyte/compound may or may not be present.)

It is important to note that laboratory qualifying flags are included on the data summary forms (Form I) that are submitted to the project by the laboratory. However, during the data review and validation process, the laboratory qualifying flags are evaluated and replaced with the project-specific validation flags.

4.2.3 DQE Process

The PC or designee will perform the DQE. The DQE process is used to assess the effect of the overall analytical process on the usability of the data. The two major categories of data evaluation are laboratory performance and matrix interferences. Evaluation of laboratory performance is a check for compliance with the method requirements. It is a straight-forward examination—either the laboratory did, or did not, analyze the samples within the limits of the analytical method. Evaluation of the matrix interferences is more subtle and involves analysis of several results, including surrogate spike recoveries, matrix spike recoveries, and duplicate sample results. The project team will evaluate the data validation results. This evaluation will assess how the data, as qualified by the data validation, can be used on the project.

Once each of the data packages has been validated, and the data validation worksheets completed, then the entire data set will be evaluated for overall trends in data quality and usability. Information summarized as part of the DQE may include chemical compound frequencies of detection, dilution factors that might affect data usability, and patterns of target compound distribution. The data set also will be evaluated to identify potential data limitations or uncertainties in the laboratory.

4.3 Reconciliation with Data Quality Objectives

The final activity of the data evaluation process is to assess whether the data meet the planned DQOs for the project. The final results, as adjusted for the findings of any data validation and data evaluation, will be checked against the DQOs, and an assessment will be made as to whether the data are of sufficient quality to support the DQOs. The decision as to data sufficiency may be affected by the overall precision, accuracy, and completeness of the data as demonstrated by the data validation process. The main project objective should be met assuming the 90 percent completeness goal is obtained after all of the data have undergone sufficient data validation. If the data, after validation and evaluation, are sufficient to achieve project objectives, the data quality and project managers will release the data and work may proceed.

4.4 Usability Assessment

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:

It is the joint responsibility of CH2M HILL's Project Chemist and the data validation subcontractor to ensure that the analytical data meet the MDLs, reporting limits, and laboratory QC limits listed in this WP, the laboratory Statement of Work, and the various methods. During this assessment, non-conformances are documented, the data are qualified for use in decision-making, and for 10 percent of the results, the entire analytical process is reconstructed and recalculated from the raw data.

Non-detected site contaminants will be evaluated to ensure that project required quantitation limits in Section 2 were achieved. If project quantitation limits were achieved and the verification and validation steps yielded acceptable data, then the data is considered usable. During verification and validation steps, data may be qualified as estimated with the following qualifiers: J, UJ, K, L, or UL. These qualifiers represent minor QC deficiencies which will not affect the usability of the data. When major QC deficiencies are encountered, data will be qualified with an R and in most cases is not considered usable for project decisions.

For statistical comparisons non-detect values will be represented by a concentration equal to one-half the sample reporting limit. For duplicate sample results, the most conservative value will be used for project decisions. Analytical data will be checked to ensure the values and any qualifiers are appropriately transferred to the electronic database. These checks include comparison of hardcopy data and qualifiers to the electronic data deliverable. Once the data has been uploaded into the electronic database, another check will be performed to ensure all results were loaded accurately. Field and laboratory precision will be compared as RPD between the two results. Deviations from the SAP will be reviewed to assess whether corrective action is warranted and to assess impacts to achievement of project objectives.

Describe the evaluative procedures used to assess overall measurement error associated with the project:

In-depth assessment occurs during the data validation process. The third-party validation contractor will follow the analytical methods to assess conformance with the QC limits. The findings of the data validation reports and the qualifiers applied to the data will be considered in context with field logs and corrective action reports to assess overall usability.

To assess whether a sufficient quantity of acceptable data are available for decision making, the data will be reconciled with measurement performance criteria following validation and review of data quality indicator. If significant biases are detected with laboratory QA/QC samples it will be evaluated to assess impact on decision making. Low biases will be described in greater detail as they represent a possible inability to detect compounds that may be present at the site. If significant deviations are noted between lab and field precision the cause will be further evaluated to assess impact on decision making.

Identify the personnel responsible for performing the usability assessment:

Project Chemist – Anita Dodson/CH2M HILL
 Project Manager – Keri Hallberg/CH2M HILL

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:

The data validation reports will identify precision and accuracy exceedances with respect to the laboratory performance for each batch of samples, as well as comparability of field and lab duplicates. All the results will be assembled and statistically reported for an overall quality assessment provided in the final project event report. Discussion will cover completeness and representativeness. Attachments supporting this report will include data validation narratives, CA forms, and field audit reports.

Data tables will be produced to reflect detected and non-detected site contaminants and geochemical parameters. Data qualifiers will be reflected in the tables and discussed in the data quality evaluation. Figures will be produced representing concentrations of contamination.

Table 4-1 presents project documentation and records.

TABLE 4-1
 Project Documents and Records

Document	Where Maintained
Field Notebooks	Electronic .pdf copies in the project file. Hardcopy (bound notebook) in the project file. Archived at project closeout.
Chain-of-Custody Records	Electronic .pdf copies in the project file. Hardcopy in the project file. Archived at project closeout.
Air Bills	Hardcopy in the project file. Archived at project closeout.
Telephone Logs	Hardcopy in the project file. Archived at project closeout.
Corrective Action Forms	Electronic .pdf copies in the project file. Hardcopy in the project file. Archived at project closeout.
PID/FID readings	Recorded in Field Notebook. Stored in EnDat.

TABLE 4-1
Project Documents and Records

Document	Where Maintained
Water quality parameters collected during sediment sampling	Recorded in Field Notebook. Stored in EnDat.
OVM/OVA readings	Recorded in Field Notebook. Stored in EnDat.
Various field measurements	Recorded in Field Notebook.
All equipment calibration information	Recorded in Field Notebook.
Pertinent telephone conversations	Recorded in Field Notebook.
Equipment maintenance records	Inspected by FTL. Not maintained.
Sample Receipt, Custody, and Tracking Records	Electronic .pdf copies in the project file. Hardcopy in the full data package.
Standard Traceability Logs	Hardcopy in the full data package. Archived at project closeout.
Equipment Calibration Logs	Hardcopy in the full data package. Archived at project closeout.
Sample Prep Logs	Hardcopy in the full data package. Archived at project closeout.
Run Logs	Hardcopy in the full data package. Archived at project closeout.
Equipment Maintenance, Testing, and Inspection Logs	Hardcopy in the full data package. Archived at project closeout.
Reported Field Sample Results	Electronic .pdf copies in the project file. Hardcopy in the data package. Archived at project closeout.
Reported Results for Standards, QC Checks, and QC Samples	Hardcopy in the full data package. Archived at project closeout.
Instrument Printouts (raw data) for Field Samples, Standards, QC Checks, and QC Samples	Hardcopy in the full data package. Archived at project closeout.
Data Package Completeness Checklists	Hardcopy in the data validation report. Archived at project closeout.
Sample Disposal Records	Maintained by the laboratory.
Extraction/Clean-up Records	Maintained by the laboratory.
Raw Data	Hardcopy in the full data package. Archived at project closeout.
Field Sampling Audit Checklists	Hardcopy in the project file. Archived at project closeout.
Fixed Laboratory Audit Checklists	If completed, hardcopy in the project file. Archived at project closeout.
Data Validation Reports	Electronic .pdf copies in the project file. Hardcopy stored with the data package. Archived at project closeout.

SECTION 5

References

CH2M HILL. 2008. *Marine Corps Base Camp Lejeune Master Project QAPP*.

U.S. Environmental Protection Agency USEPA. 1983. *Methods for Chemical Analysis of Water and Wastes*.

USEPA. 1998. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*. USEPA SW-846, Third Edition, and its updates. USEPA. 2000. *Guidance for Data Quality Objectives Process*.

USEPA. 2002. EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, QAMS.

USEPA. 2005. Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP).

Attachment A
SOPs

SOP No: F.2
Title: Sample Receipt, Inspection, Preservation and Storage Condition Requirements
Scope: This Standard Operating Procedure describes procedures to be used by Sample Control personnel in the inspection of incoming samples and the preservation and storage requirements of those samples.

1.0 SAFETY CONSIDERATIONS

- 1.1 When working with laboratory samples, safety must be of prime concern. It is required that safety glasses, a lab coat, and gloves be worn while unpacking or handling samples. Broken samples should be treated as a chemical spill and should be managed accordingly, using proper spill cleanup procedures (see SOP "Spill Cleanup"). Bench tops should routinely be covered with a bench liner, which should be changed as needed. Good personal hygiene procedures should be followed while in the Sample Control Area, specifically, it is recommended that hands be washed before or immediately after leaving the area, food or drink shall not be consumed, cosmetics shall not be applied, and smoking is forbidden in the Sample Control Area and throughout all areas of the building.
- 1.2 Sample shipping containers must be placed under the fume hood prior to opening. Once the shipping container is opened, the contents should be examined for hazards prior to removal from the hood. If no obvious hazards are identified, the container may be removed from the hood for unpacking.
- 1.3 If samples are from DOE site of known or suspected radioactive hazard, the sample shipping containers must be screened for radioactivity prior to opening.

2.0 DOCUMENTATION OF INSPECTION

- 2.1 The inspection of all incoming sample shipments shall be documented on the Sample Receipt Checklist (Figure 1), except that those samples received under the Contract Laboratory Program shall be documented on CLP Form DC-1 (Figure 2). The Checklists shall be used to coordinate the inspection of the shipment and to record important information about the condition of the shipment and the documentation received.

UNCONTROLLED COPY

SOP No: F.2
Title: Sample Receipt, Inspection, Preservation and Storage Condition Requirements
Scope: This Standard Operating Procedure describes procedures to be used by Sample Control personnel in the inspection of incoming samples and the preservation and storage requirements of those samples.

1.0 SAFETY CONSIDERATIONS

1.1 When working with laboratory samples, safety must be of prime concern. It is required that safety glasses, a lab coat, and gloves be worn while unpacking or handling samples. Broken samples should be treated as a chemical spill and should be managed accordingly, using proper spill cleanup procedures (see SOP "Spill Cleanup"). Bench tops should routinely be covered with a bench liner, which should be changed as needed. Good personal hygiene procedures should be followed while in the Sample Control Area, specifically, it is recommended that hands be washed before or immediately after leaving the area, food or drink shall not be consumed, cosmetics shall not be applied, and smoking is forbidden in the Sample Control Area and throughout all areas of the building.

1.2 Sample shipping containers must be placed under the fume hood prior to opening. Once the shipping container is opened, the contents should be examined for hazards prior to removal from the hood. If no obvious hazards are identified, the container may be removed from the hood for unpacking.

1.3 If samples are from DOE site of known or suspected radioactive hazard, the sample shipping containers must be screened for radioactivity prior to opening.

2.0 DOCUMENTATION OF INSPECTION

2.1 The inspection of all incoming sample shipments shall be documented on the Sample Receipt Checklist (Figure 1), except that those samples received under the Contract Laboratory Program shall be documented on CLP Form DC-1 (Figure 2). The Checklists shall be used to coordinate the inspection of the shipment and to record important information about the condition of the shipment and to documentation received.

- 2.2 The Sample Receipt Checklist documents the following information: The top section of the page documents receipt of the shipment, the identity of the client, the assigned Work Order Number and information relevant to the client's project. The next section of the checklist section contains specific items which must be checked and documented. Included in the section are spaces to document the sample and sample bottle count for comparison to the client supplied paper work and to document the necessity of a pH check, when applicable. If a pH check is required, it shall be documented on the Sample Preservation Check Documentation Form (Figure 3).
 - 2.3 The final section for comments is used to explain any problems or discrepancies with the shipment. Any "no" response to the previous section must be documented in the spaces provided. The sample control personnel will sign and date the form as indicated at the bottom of the page.
 - 2.4 CLP Form DC-1 is used in the same manner as the Sample Receipt Checklist and shall be used in place of that form for the documentation of inspection of CLP sample shipments. All CLP samples received must be documented individually on Form DC-1.
 - 2.5 The completed checklist is included with the original paper work submitted to the Project Management Group to be placed in the central project file. A copy of the completed Sample Receipt Checklist (or Form DC-1) is filed with copies of client supplied paper work, the Work Order and Chain-of-Custody documentation in the Sample Log book maintained by Sample Control.
 - 2.6 Documentation of any problems or discrepancies associated with sample receipt is performed by the Sample Coordinator on the Sample Receipt Checklist (or Form DC-1). The Project Management Group is then informed of these items and is responsible in resolving the issues with the client. Resolution to any problems must be documented by the Project Management Group.
- 3.0 SAMPLE SHIPMENT RECEIPT
- 3.1 Each shipment of samples arriving by commercial carrier must be accompanied by an airbill, airbill sticker, or manifest. The Sample Coordinator (also referred to as the Sample Custodian for EPA CLP work), upon taking custody of the shipment, shall sign the airbill/manifest and record the date and time the shipment was received in the space provided on the document. If no space is provided, documentation may be recorded on any open space or the back of the document.
 - 3.2 The Sample Coordinator shall next initiate a Sample Receipt Checklist or in the case of a CLP shipment, Form DC-1. The samples must be accompanied with a Chain of Custody (COC) document. The sample acceptance policy must include that:

- A. The person submitting the sample must provide full documentation with the sample, which must include:
- Sample Identification
 - The location, date and time of collection
 - The collector name, preservative added
 - Matrix
 - Any special remarks concerning the sample
- B. Each sample or group of samples must include trip blanks, field blanks, equipment blanks, duplicates or other field submitted quality measures as required by the method.
- C. Each sample must show evidence of proper preservation and use of sample containers allowed by the test method.
- D. Each sample must be of adequate volume for the requested testing.

Any deviation from the policy, the client will be contacted and documented. The sample(s) will not be processed.

Where required by method, pH of the samples must be determined and reported on the checklist. The client name, date and time received, the carrier name, and the name of the Sample Coordinator shall be recorded in the appropriate sections of the forms. The presence or absence of the airbill/manifest shall be documented and, if present, the airbill/manifest number shall be recorded. Then, the temperature blank vial should be located, and the cooler temperature must be obtained, by inserting the calibrated thermometer into the temp. blank vial, and reading the temp. after 60-90 seconds. If no temp. blank exists, then the thermometer must be inserted in the center of the cooler, where the samples are located, and the temp. should be read after 60-90 seconds of thermometer insertion.

- 3.3 In the event that samples must be received on a weekend or holiday, special arrangements between the client and the laboratory must be established. Generally, clients must contact the Project Management Group to arrange for the special service. At that time Client Services shall ascertain if any samples requiring immediate analysis are to be included in the shipment. If this is the case, arrangements shall be made to have analytical staff available to perform the required analyses. Project Management Group shall arrange to have a person from Sample Control, or a properly trained alternate, available to receive the shipment. The shipment shall be received and inspected as per this procedure. The appropriate paperwork shall be completed and, if necessary, sample preservation shall be verified. Samples requiring immediate analysis are released upon receipt to the lab by the sample control personnel, before the completion of the transmission of the work order. Thereby enabling the laboratory to meet the required holding time criteria.

3.4 Short Holding Times and Rush Samples

Samples received that require either rush TAT (<72 hours) and or short holdings (<72 hours) require immediate attention by the Sample Management division. It is the responsibility of the Sample Management division to insure the appropriate laboratory receives the samples and are notified of the urgent circumstances. The Sample Management division will bring the samples to the appropriate laboratory manager along with a copy of the original COC or equivalent (written documentation showing the sample ID and the tests required). After the samples are logged in and approved, the analysts will fill out the appropriate internal COC.

4.0 SHIPPING CONTAINER INSPECTION

4.1 All sample shipping containers shall be inspected for integrity and physical damage upon receipt. This shall be documented on Line 2 of the Checklist. Any problems found must be documented in the comments section at the bottom of the page. For Form DC-1, comments shall be made in the Remarks section.

4.2 Safety Note: If a strong smell is emanating from a shipping container, place the container in the hood to be unpacked. If a liquid is leaking from a shipping container, place container in the hood on a metal or plastic tray to contain the liquid and prevent gross contamination of the Sample Control Area. Any leaking material should be considered as a hazardous material and should be treated as such. All sample shipments originating from the Sample Management Office (SMO) for the Contract Laboratory Program (CLP) must be opened and unpacked under the hood. In all cases, proceed with caution while checking for broken sample vessels.

4.3 Once the shipping container has been examined for damage, and the presence or absence of custody seals is determined. The sample receiving personnel will evaluate and document the use of chain of custody seals that are of the non-tamper evident variety. Then the overall condition should be ascertained. This check shall be documented in the appropriate spaces on the forms.

5.0 SAMPLE VESSEL INSPECTION

5.1 The shipping container shall be carefully opened, any paper work should be removed, and it shall be determined whether ice or cold packs are present. The temperature of the shipping container shall be measured by placing a thermometer in the provided temp blank or among the samples, closing the lid and reopening the container and reading the thermometer for approximately 60-90 seconds. On the sample receipt checklist, document the presence or absence of ice or cold packs and the temperature of the shipping container. Please note that if the samples do not require cold preservation, for example preserved water samples for metals analysis, "No" must be checked and an explanation of N/A included in the Comments section.

- 5.2 The shipping container should be carefully unpacked and the incoming samples checked for:
- Physical damage due to inadequate packing and/or protection.
 - Loss of sample due to inadequate and/or improper sealing of the sample container (i.e., leakage of liquids, loss of particulate material from filters, etc.).
 - Possible contamination because of inadequate separation of sample types or bulk sample materials (i.e., charcoal tubes or VOA vials shipped in the same container as bulk liquid organics).
 - Adequate containment of volatile organic samples and total organic halide samples in septum vials; there should be zero headspace. If headspace is found, this shall be documented. A widely accepted standard for document the approximate size is to judge the headspace to be smaller or larger than a green pea. This should be noted the Checklist or noted in the sample Remarks section of Form DC-1 and the Project Manager/Client Services Group contacted, as the client may wish to re-sample.
 - Proper use of special shipping procedures required to preserve the samples. For example, if shipping instructions note that samples are to be kept frozen, then samples should be frozen upon receipt.

If samples are broken or evidence that sample integrity may have been compromised, the incident shall be documented in the designated space of the Checklist and detailed in the Comments section. For CLP samples this shall be documented on Form DC-1, Line 8 and explained in the remarks section for the affected samples and shall also be noted on the Sample Traffic Report Form. The Project Management Group shall be notified of any such problems as soon as possible.

6.0 CONTRACT LABORATORY PROGRAM SAMPLE VESSEL INSPECTION

- 6.1 For samples submitted by SMO, a review of sample documentation is required once the shipping container has been unpacked. Each shipping container should possess a Chain-of-Custody Form and a Sample Traffic Report Form for the samples contained within the shipping vessel. Each sample should have a Sample Label and a Sample Tag attached to the bottle (where applicable). EPA sample bottles should also have a custody seal over the cap to detect tampering. All items shall be reviewed and documented on Form DC-1.
- 6.2 Presence or absence of custody seals should be determined. If present, condition of custody seals must be assessed. This shall be documented on Line 1 in the Remarks section of Form DC-1.

- 6.3 Presence of EPA Sample Tags on sample bottles should be determined. The presence or absence of tags shall be documented on Line 7.
- 6.4 It shall be determined if the EPA Traffic Report or SAS Packing list was included in the shipment. Documentation shall be made on Line 4.
- 6.5 It shall be determined if the Chain-of-Custody document was included in the shipment. Documentation shall be made on Line 3.
- 6.6 The Chain-of-Custody document shall be reviewed to verify the entry of Sample Tag numbers on the Chain-of-Custody. If tag numbers have not been recorded on the Chain-of-Custody, the Sample Coordinator shall perform the document entries and shall document the information in the remarks section for the affected sample.
- 6.7 The Chain-of-Custody shall be compared with the Sample Labels. Any discrepancies shall be documented. This shall be documented in the remarks section for the affected sample.
- 6.8 The Chain-of-Custody shall be compared with the Sample Tags. Any discrepancies shall be documented. This shall be documented in the remarks section for the affected sample.
- 6.9 The Chain-of-Custody shall be compared with the EPA Traffic Reports. Discrepancies shall be documented in the remarks section for the affected sample.

7.0 SAMPLE PRESERVATION INSPECTION

7.1 EPA Contract Laboratory Program (CLP) Samples

7.1.1 Samples which are submitted under the Contract Laboratory Program for inorganic parameters (metals and cyanide) are preserved in the field and may require pH adjustment. These samples are to be checked for proper preservation upon receipt. Because of the nature of these samples proper safety precautions must be observed. Safety glasses, lab coats and disposable gloves shall be worn when handling the sample bottles while performing pH measurements. Gloves shall be changed when soiled to prevent contamination between both the samples or of the employee.

7.1.2 The pH check shall be performed by removing an aliquot of the sample, pouring it into a small disposable cup and depositing the drop onto a pH strip paper. Match the resulting color of the pH paper to the color scale provided with the pH paper. Various ranges of pH papers are used for preservation check. For low range pH measurement, the pH indicator of 0-6 or 0-2.5 will be used. Other wise, pH indicator range 11-13 or 7.5-14 will be used. This will enable to obtain a definitive measurement of pH.

Samples for metals analysis must have a pH of less than 2, and samples for cyanide must have a pH greater than 12.

- 7.1.3 Record the pH reading on a Sample Preservation Check Documentation Form (Figure 3). The pH may be recorded as less than the value required for acid preserved samples (i.e. <2), or greater than the required value for NaOH preserved samples (i.e. >12 for NaOH preserved cyanide samples). If a sample is discovered to be inadequately preserved, the exact pH reading from the pH check must be recorded. The form shall be signed and dated by the responsible person.
- 7.1.4 If the pH check indicates that the sample was not adequately preserved, contact the Project Manager immediately to receive instructions from the Sample Management Office. Documentation of sample preservation deficiencies, and instructions given by the Sample Management Office shall be documented in the case file and the case narrative by the Project Manager. If the Sample Management Office representative dictates that the samples shall be preserved in the laboratory, documentation of this activity shall be indicated on the Sample Preservation Check Documentation Form (Figure 3). The samples may be preserved by Sample Control or laboratory personnel, where applicable. In either case, suitable quantities of the appropriate preservative shall be added to the inadequately preserved samples. Documentation of these activities shall be indicated on the Sample Preservation Check Documentation Form (Figure 3). Copy of the documentation form shall be filed in the case file.
- 7.1.5 Table 1 provides information on required containers, holding instructions, and holding times for USEPA CLP submitted samples.

7.2 Commercial Samples

- 7.2.1 EPA requires chemical preservation of water samples to be analyzed for selected parameters. Table 2 presents information which lists parameters, required containers, sample size needed, preservation techniques and holding times for sample analysis performed by methods specified in 40 CFR Part 136, Test Methods for Evaluating Solid Waste - SW 846, and Methods for Chemical Analysis of Water and Wastes (MCAWW). The Sample Coordinator is responsible for verifying that any samples requiring pH adjustment have been appropriately preserved in the field.
- 7.2.2 The pH check shall be performed by removing an aliquot of the sample, pouring it into a small disposable cup and depositing the drop onto a pH strip paper. Match the resulting color of the pH paper to the color scale provided with the pH paper. Various ranges of pH papers are used for preservation check. For low range pH measurement, the pH indicator of 0-6 or 0-2.5 will be used. Other wise, pH indicator range 11-13 or 7.5-14 will be used. This will enable to obtain a definitive measurement of pH.

Samples for metals analysis must have a pH of less than 2, and samples for cyanide must have a pH greater than 12.

- 7.2.3 Record the pH reading on a Sample Preservation Check Documentation Form (Figure 3). The pH may be recorded as less than the value required for acid preserved samples (i.e. <2), or greater than the required value for NaOH preserved samples (i.e. >12 for NaOH preserved cyanide samples). As an alternative a check mark may be used to signify that the sample met the preservation criteria. If a sample is discovered to be inadequately preserved the exact pH reading from the pH check must be recorded. The form shall be signed and dated by the responsible person.
- 7.2.4 If the pH check indicates that the sample was not adequately preserved, contact the Sample Control Supervisor immediately. The Sample Control Supervisor will contact the appropriate Project Manager who will, after consultation with the client, instruct the Sample Control Supervisor of the appropriate action to follow. Generally, the samples are preserved during that time and the necessary documentation of the problem and the resolution are performed. Also, notations are indicated in the final report describing the situation. Samples may be preserved by Sample Control or laboratory personnel. In either case, suitable quantities of the appropriate preservative shall be added to the inadequately preserved samples. See Table 2 for information on preservatives and criteria. Documentation of these activities shall be made on the Sample Preservation Check Documentation Form (Figure 3). Copy of the documentation form shall be filed in the case file.
- Note:** If drinking water metals samples are acidified in the laboratory, the time that the samples are acidified must be recorded on the preservation check form (Fig.3) in order to meet the 16 hour hold time requirement.
- Note:** If Radiochemistry samples are acidified in the laboratory, the time that the samples are acidified must be recorded on the preservation check form (Fig.3) in order to meet the 16 hour hold time requirement.
- Note:** Samples for Radiochemistry Tritium analysis should not be preserved.
- 7.2.5 Failure to appropriately preserve the samples in the field may result in invalid analytical data. It is the responsibility of Project Management Group to advise the client of this potential.

All acid preservatives are verified upon arrival to the lab.

- 7.2.6 Samples for State of North Carolina, which are chlorine sensitive, i.e., organics, ammonia, TKN and cyanide must be checked for the presence of chlorine and treated, if required, before storage and analysis. Documentation of these activities shall be noted on the sample residual chlorine check form (Figure 4).

7.3 pH Adjustment Procedure

- 7.3.1 Samples which require pH adjustment must have appropriate amounts of the required preservative added. This procedure must be carefully performed and must be properly documented (Figure 3).
- 7.3.2 Generally, the preservative solutions are relatively concentrated (i.e. 1:1 Nitric or Sulfuric Acid, 5N NaOH) to avoid significant changes in volume. Therefore, only a small quantity of preservative should be added. Due to sample compositions, proper safety precautions must be observed. While performing pH measurements, safety glasses, lab coats and disposable gloves shall be worn. Gloves shall be changed when soiled to prevent sample contamination.
- 7.3.3 When a particular sample requires additional preservation the sample shall be opened and a small quantity of the preservative added. The amount of preservative added should be in increments of approximately 0.5ml/250ml of sample. Once the preservative is added the bottle shall be securely capped and shaken to distribute the preservative. The bottle shall then be carefully opened, and observed for evidence of escaping pressure before rechecking the pH. The pH check shall be performed by removing an aliquot of the sample, pouring it into a small plastic cup and depositing the drop onto a strip of pH paper. Match the resulting color of the pH paper to the color scale provided with the pH paper. If the pH does not yet meet the method specification, repeat the process until an acceptable value is maintained.
- 7.3.4 Documentation of the preservation adjustment and reagent lot number shall be indicated on the Sample Preservation Adjustment Documentation Form (Figure 3). Each space shall be completed for each listed sample and the form shall be signed and dated by the responsible person.

Figure 2

SAMPLE LOG-IN SHEET

Lab Name				Page ___ of ___		
Received By (Print Name)				Log-in Date		
Received By (Signature)						
Case Number		Sample Delivery Group No.			NRAS Number	
Remarks: 1. Custody Seal(s) Present/Absent* Intact/Broken 2. Custody Seal Nos. _____ 3. Traffic Reports/Chain of Custody Records or Packing Lists Present/Absent* 4. Airbill Airbill/Sticker Present/Absent* 5. Airbill No. _____ 6. Sample Tags Present/Absent* Sample Tag numbers Listed/Not Listed on Traffic Report/Chain of Custody Record 7. Sample Condition Intact/Broken*/Leaking 8. Cooler Temperature Indicator Bottle Present/Absent* 9. Cooler Temperature _____ 10. Does information on Traffic Reports/Chain of Custody Records and sample tags agree? Yes/No* 11. Date Received at Lab _____ 12. Time Received _____	EPA Sample #	Aqueous Sample pH	Corresponding		Remarks: Condition of Sample Shipment, etc.	
			Sample Tag #	Assigned Lab #		
Sample Transfer						
Fraction	Fraction					
Area #	Area #					
By	By					
On	On					
* Contact SMO and attach record of resolution						
Reviewed By			Logbook No.			
Date			Logbook Page No.			

Table 1
Summary of Contract Laboratory Program Required
Containers, Preservation Techniques, and Holding Times¹

Parameter	Container	Holding Instructions	Maximum Sample Holding Time
Metals	P, G	Room Temperature	180 days from VTSR
Mercury	P, G	Room Temperature	26 days from VTSR
Cyanide (Total and amenable to chlorination)	P, G	Room Temperature	12 days from VTSR
Volatiles	G(TS)	Store at 4° ± 2°C	10 days from VTSR
Semivolatiles (water)	G(TC)	Store at 4° ± 2°C (in dark)	5 days from VTSR ^a
Semivolatiles (soil/sediment)	G(TC)	Store at 4° ± 2°C (in dark)	10 days from VTSR ^a
Pesticides/PCBs (water)	G(TC)	Store at 4° ± 2°C (in dark)	5 days from VTSR ^a
Pesticides/PCBs (soil/sediment)	G(TC)	Store at 4° ± 2°C (in dark)	10 days from VTSR ^a

^a - Extract holding time is 40 days after extraction

P = plastic

G = glass

G(TC) = glass, TFE-lined cap

G(TS) = glass, TFE-lined septum

¹ - USEPA Contract Laboratory Program; Statement of Work for Inorganics Analysis; Multi-media, Multi-concentration; SOW No. 3/90

USEPA Contract Laboratory Program; Statement of Work for Organics Analysis; Multi-media, Multi-concentration; SOW No. 3/90

Table 2
Summary of Required Sample Containers, Sample Volumes,
Preservation Techniques And Storage Requirements
(from 40 CFR Part 136)

Parameter Name	Container ¹	Minimum Sample Size (ml)	Preservation ^{2,3}	Maximum Holding Time ⁴
BACTERIAL TESTS				
Coliform, fecal and total	P, G (sterile)	200	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵	6 hours
Fecal Streptococci	P, G (sterile)	200	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵	6 hours
INORGANIC TESTS⁷				
Acidity	P, G	200	Cool, 4°C	14 days
Alkalinity	P, G	200	Cool, 4°C	14 days
Ammonia	P, G	200	Cool, 4°C, H ₂ SO ₄ to pH < 2	28 days
Biochemical Oxygen Demand	P, G	1,000	Cool, 4°C	48 hours
Bromide	P, G	250	None	28 days
Chloride	P, G	50	None Required	28 days
Chlorine, total residual	P, G	500	None Required	Analyze immediately
Color	P, G	500	Cool, 4°C	48 hours

Table 2
Summary of Required Sample Containers, Sample Volumes,
Preservation Techniques And Storage Requirements
(from 40 CFR Part 136)

Parameter Name	Container ¹	Minimum Sample Size (ml)	Preservation ^{2,3}	Maximum Holding Time ⁴
Cyanide, total and amenable to chlorination	P, G	1,000	Cool, 4°C, H ₂ SO ₄ to pH > 12, 0.6g ascorbic acid ⁵	14 days ⁶
Fluoride	P	1,000	None Required	28 days
Hardness	P, G	200	HNO ₃ or H ₂ SO ₄ to pH < 2	6 months
Hydrogen ion (pH)	P, G	250	None Required	Analyze Immediately
3jeldahl and organic nitrogen	P, G	500	Cool, 4°C, H ₂ SO ₄ to pH < 2	28 days
Chromium VI	P(A), G(A)	300	Cool, 4°C	24 hours
Mercury	P(A), G(A)	500	HNO ₃ to pH < 2	28 days
Metals, except Chromium VI and Mercury	P(A), G(A)	500	HNO ₃ to pH < 2	6 months
Nitrate	P, G	200	Cool, 4°C	48 hours
Nitrate-nitrite	P, G	200	Cool, 4°C, H ₂ SO ₄ to pH < 2	28 days
Nitrite	P, G	200	Cool, 4°C	48 hours

Table 2
Summary of Required Sample Containers, Sample Volumes,
Preservation Techniques And Storage Requirements
(from 40 CFR Part 136)

Parameter Name	Container ¹	Minimum Sample Size (ml)	Preservation ^{2,3}	Maximum Holding Time ⁴
Oil and Grease	G, (wide mouth)	2000	Cool, 4°C, H ₂ SO ₄ to pH < 2	28 days
Organic Carbon	P, G	100	Cool, 4°C, H ₂ SO ₄ to pH < 2	28 days
Orthophosphate	P(A), G(A)	200	Filter Immediately, Cool 4°C	48 hours
Oxygen, dissolved (probe)	G Bottle and Top	300	None Required	Analyze Immediately
Winkler	G Bottle and Top	300	Fix on site and store in dark	8 hours
Phenols	G only	1000	Cool, 4°C, H ₂ SO ₄ to pH < 2	28 days
Phosphorous, elemental	G	200	Cool, 4°C	48 hours
Phosphorous, total	P, G	200	Cool, 4°C, H ₂ SO ₄ to pH < 2	28 days
Residue, total	P, G	200	Cool, 4°C	7 days
Residue, filterable (TDS)	P, G	200	Cool, 4°C	48 hours
Residue, nonfilterable (TSS)	P, G	200	Cool, 4°C	7 days
Residue, settleable	P, G	200	Cool, 4°C	48 hours

Table 2
Summary of Required Sample Containers, Sample Volumes,
Preservation Techniques And Storage Requirements
(from 40 CFR Part 136)

Parameter Name	Container ¹	Minimum Sample Size (ml)	Preservation ^{2,3}	Maximum Holding Time ⁴
Residue, volatile	P, G	200	Cool, 4°C	7 days
Silica	P	500	Cool, 4°C	28 days
Specific Conductance	P, G	500	Cool, 4°C	28 days
Sulfate	P, G	50	Cool, 4°C	28 days
Sulfide	P, G	200	Cool, 4°C, add zinc acetate plus sodium hydroxide to pH > 9	7 days
Sulfite	P, G	100	None Required	Analyze Immediately
Surfactants	P, G	500	Cool, 4°C	48 hours
Temperature	P, G	250	None Required	Analyze
Turbidity	P, G	250	Cool, 4°C	48 hours

Table 2
Summary of Required Sample Containers, Sample Volumes,
Preservation Techniques And Storage Requirements
(from 40 CFR Part 136)

Parameter Name	Container ¹	Minimum Sample Size (ml)	Preservation ^{2,3}	Maximum Holding Time ⁴
ORGANIC TESTS⁸				
Purgeable Halocarbons	G, Tef.-lined septum	2X 40	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵	14 days
Purgeable aromatic hydrocarbons	G, Tef.-lined septum	2X 40	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵ , HCl to pH 2 ⁹	14 days
Acrolein and acrylonitrile	G, Tef.-lined septum	2X 40	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵ , Adjust pH to 4-5 ¹⁰	14 days
Phenols ¹¹	G, Tef.-lined cap	2000	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction; 40 days after extraction
Benzidines ¹¹	G, Tef.-lined cap	2000	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction ¹³
Phthalate esters ¹¹	G, Tef.-lined cap	2000	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitrosamines ^{12,14}	G, Tef.-lined cap	2000	Cool, 4°C, store in dark, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction; 40 days after extraction
PCBs ¹¹ acrylonitrile	G, Tef.-lined cap	2500	Cool, 4°C	7 days until extraction; 40 days after extraction

Table 2
Summary of Required Sample Containers, Sample Volumes,
Preservation Techniques And Storage Requirements
(from 40 CFR Part 136)

Parameter Name	Container ¹	Minimum Sample Size (ml)	Preservation ^{2,3}	Maximum Holding Time ⁴
Nitroaromatics and isophorone ¹¹	G, Tef.-lined cap	2000	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ . ⁵ Store in dark	7 days until extraction; 40 days after extraction
Polynuclear aromatic hydrocarbons ¹¹	G, Tef.-lined cap	2000	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction 40 days after extraction
Haloethers ¹¹	G, Tef.-lined cap	2000	Cool, 4°C, 0.008% Na ₂ S ₂ O ₃ ⁵	7 days until extraction; 40 days after extraction
Chlorinated hydrocarbons ¹¹	G, Tef.-lined septum	2X 40	Cool, 4°C	7 days until extraction; 40 days after extraction
PESTICIDES				
Pesticides ¹¹	G, Tef.-lined cap	1000	Cool, 4°C, pH 5-9 ¹⁵	7 days until extraction 40 days until extraction
RADIOLOGICAL TESTS				
Alpha, beta, radium	P, G	2000	HNO ₃ to pH 2	6 months

1. Polyethylene (P) or Glass (G), G(A) or P(A) = rinsed with 1:1 Nitric Acid
2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
3. When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight 0000000 or less (pH about 1.96 or greater); Nitric acid (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator under 136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See 136.3(e) for details.
5. Should only be used in the presence of residual chlorine.
6. Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
7. Samples should be filtered immediately on-site before adding preservative for dissolved metals.
8. Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
9. Sample receiving no pH adjustment must be analyzed within 7 days of sampling.

10. The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
11. When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for four days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine), and footnotes 12 and 13 (re: the analysis of benzidine).
12. If 1,2-Diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.
13. Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
14. For the analysis of diphenylnitrosamine, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
15. The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$.

Effective Date: February 2003
Version Number: 6
Initiated By: _____
Approved By: _____

SOP No: F.3

Title: Sample Logging and Record Keeping

Scope: This Standard Operating Procedure describes steps to be taken in the logging of samples and the records to be maintained by the Sample Control operation.

1.0 SAMPLE LOGGING PROCEDURES - INITIATION

- 1.1 All incoming samples must be inspected as outlined in SOP F.2, Sample Receipt, Inspection, Preservation and Storage Condition Requirements.
- 1.2 Individual incoming samples must have identification to differentiate between samples. Organize the samples into series according to the identification provided on the client sample submittal paperwork and check the actual samples received against the client paperwork. If there is an inconsistency between the sample labeling and the paperwork, the project manager will be notified and will contact the client. Discrepancies shall be noted in the Comments section of the Sample Receipt Checklist as described in SOP F.2.
- 1.3 Leave the samples in sequence until new GPL Laboratories sample labels are attached (See Section 2 of this SOP). The sample labels must be durable and indelible. Each sample container must be given a unique sample identification number provided by the LIMS. Samples requiring refrigeration should be placed on the "temporary holding" cart in the refrigerator (location A) until data entry procedures are complete and a permanent storage location is assigned. Note that samples requiring volatile analyses should be stored in the stand-alone refrigerators.
- 1.4 If a sample does not pass inspection, the Sample Coordinator should immediately notify the Sample Control Supervisor. Any discrepancies should be recorded in the Comments section of the Sample Receipt Checklist (Figure 1). The Sample Control supervisor will verify that the Project Manager/Client Services group is notified and provides specific instructions to the Sample Coordinator.

- 1.5 To facilitate the logging process, a Sample Receipt Checklist Form must be completed. If the project is governed by the EPA CLP program, then the CLP form DC-1(Figure 2) must be used, instead of the sample receipt checklist form.
- 1.6 A Sample Preservation Check Documentation Form (Figure 3) must also be completed if any samples containing acid preservative are received.
- 1.7 Attach the client paperwork to the sample receipt checklist, preservation check form and forward to the project manager group. After review by the project manager, 2nd approval, the paperwork must be returned to sample receiving

2.0 SAMPLE LOGGING SUMMARY - DATA ENTRY

2.1 The Laboratory Information Management Systems (LIMS)

- Two LIMS programs are used for entering sample information into the LIMS system: The "Project Management" program and the "Sample Log-In" program.
- The Project Manager/Client Services Group is primarily responsible for entering project information into the LIMS using the "Project Management" program.
- The Sample Control Group is primarily responsible for entering sample receipt information into the LIMS using the "Sample Log-In" program.
- The LIMS automatically assigns unique lab identifiers to the samples and produces forms used by managers to track every sample from log-in to invoicing.

2.1.1 Entering Project Information

Log on to Xenco

2.1.1.1 Create client information

- Select Contacts & Companies icon at left of screen (icon has faces on it).
- Select the Clients/Contacts tab.
- Clear screen (brush icon at top of screen).
- Select new client (green + icon at top of screen).
- Check Company button.
- Check Client? box at the right of the screen.
- Create client code using company name and city: ie, CH2M_GAINESVILLE.
- Fill in company name, address, phone number, fax number and e-mail, as applicable.
- Select the zip code from the drop down menu. If the zip code is not on the list click on the Cities icon on the left of the screen. (It

is currently blank).

Type in the name of the city that you need in the indicated field.

From the drop down menu select the state and then Save (blue cassette icon at top of screen).

Click on the Zip Code button at the bottom of the screen.

Type in the new zip code in the indicated field.

From the drop down menu select the city and then Save.

The new zip code should now be in LIMS.

If the city name but not the correct zip code is already in LIMS click on the Zip Code icon on the left of the screen. (It is currently blank).

Type in the zip code in the indicated field.

From the drop down menu select the city and then Save.

The new zip code should now be in LIMS.

Close the Cities and Zip Code screens.

Go back to the Contacts and Companies screen and from the drop down menu select the zip code that you created.

When all information is entered Save the Contacts and Companies screen.

Successfully Saved message should appear.

2.1.1.2 Create contact information

Still in Contacts & Companies; select Contacts Tab.

Select Add Contact/Relationship icon (green + on right side of screen).

In Add New Contact screen, type in first name and last name of client contact in indicated boxes.

Click on Code box to automatically create name code.

Save screen. Successfully Saved message should appear.

Close Add New Contact screen.

Contact name should appear in Contact/Company column.

Select Contacts Info button on right, below the green + button.

Project Managers screen will appear with the new contact name showing.

Click on Name box at the bottom of the screen.

Use the drop down menu to select the company name to associate with the contact.

(Typing in the first few letters of the company name will take you to that part of the list.)

Highlight the company name and click OK or Enter.

The company should appear in the Name box.

Save screen and close the Project Managers screen.

The person should now be added as a contact for the company.

Close Contacts & Companies.

2.1.1.3 Create Project

Select Project & Quotes Management (P icon below W icon at left of screen).

Select the first tab, Project Specifications.

Clear screen (brush icon).

Select new project (green + icon).

Position cursor in Client box.

Click on the Client drop down menu and find the company that you created.

(Typing in the first few letters of the company name will take you to that part of the list.)

Highlight the company and click OK or Enter.

The company name should appear in the Client box.

Using the tab key or mouse, go to Project Name and type in the project name.

Go to Project ID and type in the ID (optional).

Click on Location box. This automatically brings up Gaithersburg as the default (lab location).

Click on Manager box and from the drop down menu highlight the contact that you created.

Click OK or Enter. The contact name should appear in the Manager Box.

Click on Director box and from the drop down menu highlight the contact.

Click OK or Enter. The contact name should appear in the Director Box.

Click on Lab PM box and from the drop down menu highlight the GPL project manager.

Click OK or Enter. The name should appear in the Lab PM Box.

Click on the Project Client box and from the drop down menu highlight the company name.

Click OK or Enter. The company name should appear in the Project Client Box.

Click on the Default TAT Box and from the drop down menu highlight the project TAT.

Click OK or Enter. The TAT should appear in the Default TAT Box.

Click on the Comments box and type in whatever info is needed to provide all analysts with the project requirements. At a minimum the type of data package and EDD requirements should be shown.

Save the screen.

Xenco will automatically assign the numbered Project Code at the top of the screen.

2.1.1.4 Create Methods

Still in Project & Quotes Management; select the second tab, Methods Requested.

The Method box at the left top of the screen will be highlighted. In the Method box type in the method that you want to add to the project. SW846 methods all begin as SW###. MCAWW methods begin as E###. CLP methods begin with CLP_XXX. The % sign can be used as a wild card if you don't know the exact method number.

After you have typed in a method number, double click on it. This will bring up all methods, which begin with the name that you typed in, in the Methods Selection list on the bottom left of the screen.

From Methods Selection click anywhere on the line for the method/matrix that you want to add to the project.

Click on the single right arrow  to the left of the list to move this method to the Methods For This Project list on the right.

Continue selecting methods until all of the required methods/matrices needed for the project are on the Methods For This Project list.

If a method is moved to the right in error, select the method and click the single left arrow to move it back.

Click on a selected method and arrow right until you get to the Prep 1 column.

Check that the correct prep method for each analysis is showing.

If you want to change the prep method, click on the box containing the prep code that you want to change and then click on the Refresh button at the top of the tab. This will bring up all associated prep methods in the box below the selected methods.

Click on the prep code that you want to use and then click on the large blue up arrow on the bottom left of the screen. This will move the selected prep up to the selected method.

You may save as you add each method or after you have all methods and prep codes selected.

Xenco will bring up the message: Column 10 <QC Group {29}> at Row X is Required.

Click on the No box and the Successfully Saved message should come up.

The project should now contain all methods needed to log-in samples.

If you need to make a change after you have saved a method; select the box that needs to be revised, make the change and then highlight the entire row by clicking on the line number to the left of the line. Click Save. This will re-save only the line that had the change. If you want to delete an entire method/matrix, highlight the entire row by clicking on the line number on the left and then click on the Delete button at the top of the screen (red

X). Xenco will ask if you want to delete this information. Click Yes and the line will be removed. In order for a change or addition to work you may have to click anywhere in the section where you are making the change and click the Refresh button. You should then be able to make and save the change.

2.1.1.5 Select Method Analytes

Still in Project & Quotes Management; select the Analyses Requested tab.

All saved project methods will appear in the top box of the screen. Select the first method and click on the Refresh button at the top of the tab.

This will bring up all analytes associated with this method in the box on the bottom left.

Double click on the Description header bar and the analytes will sort in ascending or descending order, compounds beginning with numbers first.

Select each compound that should be reported per project specifications, and then click on the single right arrow, to the left of the box, to move the analyte to the right box. If you want to select multiple analytes you can click on the first compound, hold down the left mouse button, drag down to highlight all analytes at the same time and then use the right arrow to move them all to the right.

If an analyte is selected in error hit the single left arrow to move it back to the left.

If you can't find a needed compound check the From Master box (only "rom mas" shows) at the top left of the tab. This will refresh the list of analytes to bring up every compound associated with the method. If you still can't find the compound that you need ask the System Admin to add it to the method/matrix.

You may Save as each compound is added or after all compounds have been selected and moved to the right. The message X Rows Successfully saved will appear.

The project is now ready to use for logging in samples.

2.1.1.6 Making Changes

Projects may be updated/revised as follows:

Company address, phone numbers or e-mail can be revised at any time by doing the following:

Select Contacts & Companies.

Click the Enter Query icon at the top of the screen (red light turns green).

Check the Company box.

Type in the company code or company name. The % sign

can be used as a wild card.

Click the Search Query icon at the top of the screen (binoculars).

Companies matching the code or name will appear.

If more than one company is found click on the right arrow at the top left of the screen, next to the number 1, to move from one to the next until you find the company that you want.

Edit the company information as needed (except Code) and then Save.

Project name, Project ID, Manager, Director, Lab PM, Project Client, Default TA and Comments may be changed at any time by doing the following:

Select Project & Quotes Management.

Select the Project Specifications tab.

Click the Enter Query icon at the top of the screen (red light turns green).

Type in the project code number or use the drop down menu to select the Client and type in part or all of the Project Name.

The % sign can be used as a wild card.

Click the Search Query icon at the top of the screen (binoculars).

Company projects matching the code or name will appear.

If more than one company is found click on the right arrow at the top left of the screen, next to the number 1, to move from one to the next until you find the company that you want.

Type in the new information or use the drop down menus to make the changes and then Save.

List of analytes for each method may be changed at any time by doing the following:

Follow the steps above to find the correct company and project.

Select the Analytes Requested tab.

From the top box, select the method that you want to revise and click on the Refresh button to bring up the list of analytes.

For each analyte that you want to remove from the bottom right box; highlight the entire row by clicking on the line number on the left.

Click the Delete icon (red X) at the top of the screen.

Xenco will ask if you want to delete this information.

Click Yes and the analyte(s) will be removed.

To add analytes select them from the bottom left box and use the single right arrow to move them to the right box. Save.

Methods can be removed as long as they have never been used to log-in samples.

Follow the steps above to find the correct company and project.

Select the Analytes Requested tab first.

In the top box, select the method that you want to remove and click the Refresh button to bring up the list of analytes. All analytes must be removed from the bottom right box, using the procedure in the previous paragraph, before the method can be deleted.

After all analytes have been deleted go back to the Methods tab.

You may have to refresh the methods list before you are able to delete it.

Highlight the entire row containing the method that you want to delete and click the Delete button (red X).

Xenoco will ask if you want to delete this information.

Click Yes and the method will be removed.

2.1.2 Entering Sample Information

After logging in to a computer work station, there will be a configured menu item labeled "LIMS". A sub-menu item labeled "Sample Log-In" must be activated to load the Sample Log-In program. Either the program will prompt for a user ID and password, or else security information will be automatically supplied by the operating system.

Entering sample information is comprised of 4 parts: entering shipment (work order) record, entering sample records, entering sample bottle (fraction) records, and assigning analytical jobs to the samples.

The details of the procedure will depend on the software version being used, but the following are the essential basic steps for entering sample information.

- Enter the shipment (work order) information: project ID, sampling information, shipping information, date received, and turnaround time.
- Add samples, entering sample information: client's sample ID, matrix, date of sampling.
- Add sample bottles (fractions), entering the storage location, and the container type.

- Assign analytical jobs to the samples by adding jobs and assigning the appropriate job codes.

2.1.3 Scheduling the Shipment (Work Order)

After entering all the information pertinent to a shipment (work order), the records must be inserted into the LIMS. Activate the function (available from the program's menu), for transferring the shipment (work order) into the LIMS. Once completed, the records are visible to all laboratory status checking and scheduling modules. New work orders are given status 25 when completed by sample management. Work orders are then checked by the project manager and given status 30 when completed.

2.1.4 Printing Forms

After the work order is complete, forms and sample labels should be printed. Labels contain the laboratory sample IDs for each bottle, as well as the tests to be performed on the samples.

Available forms are:

- Internal Chain of Custody (Figure 4)
- Work Order/Fraction Sheet, which lists all scheduled tests (Figure 5).
- Subcontracted Chain of Custody (Figure 6).

Forms may be printed individually, or using batches to redirect the form to it's proper destination within the facility.

2.1.5 Making Corrections

After a shipment has been logged in, it may still be modified (for example, a client may request additional analyses). Samples may be added or deleted, and jobs may be assigned or deleted after the shipment has been scheduled. Paperwork must be re-submitted after changes have been made to alert project managers and laboratory personnel.

A correction may be required using the Analysis Request Form (Figure 7).

2.2 Labeling the Samples

- ### 2.2.1 Obtain the labels from the label printer. Remove the labels from the wax paper backing and place on the corresponding samples. Verify that the correct label is being attached to the appropriate sample. Do not conceal any information that the client has provided on the sample.

2.3 pH Check for Preserved Samples

2.3.1 Chemical preservation of water samples is required by EPA for certain test parameters. SOP 7.2 "Sample Receipt, Inspection, Preservation, and Storage Condition Requirements" provides detailed information on this aspect of sample log-in. **Special Note:** Samples submitted from the Sample Management Office (SMO) under the Contract Laboratory Program (CLP) shall NOT be pH checked.

2.4 Organization of the Paperwork

2.4.1 Sample Receiving

- Internal Chain of Custody
- Subcontracted Chain of Custody
- Work Order(shipment)/Fraction (sample bottle)/Test Sheet
- Labels

2.4.2 Subcontractors

- Subcontracted Chain of Custody

2.4.3 Labs

- Work Order(shipment)/Fraction (sample bottle)/Test Sheet

2.5 Samples Requiring Immediate Analysis

2.5.1 The Sample Control Supervisor will notify the appropriate Project Manager when samples arrive that have a maximum holding time of 48 hours or less, or require immediate analysis.

2.6 Sample Receipt Notification

2.6.1 Some clients require notification that samples have been received. In such cases, the project manager supplies a copy of the COC, along with a copy of the sample receipt check list (figure 8).

3.0 SAMPLE STORAGE

3.1 All samples received for analyses are logged in according to the procedures detailed above and must be stored in such a manner as to maintain their integrity. Discrete sample locations based upon type of sample have been identified and are listed in SOP F.4, "Secure Sample Storage". Sample Control is maintained with restricted access at all times.

4.0 SAMPLE TRACKING

- 4.1 Samples may be distributed for analysis only after they have been logged into the LIMS system. Once a sample has been logged and stored, laboratory personnel must submit a request to Sample Control for the samples in accordance with the procedure described in SOP F.1, "Sample Chain-of-Custody". The analyst is ultimately responsible for the integrity of the sample while it is in his/her custody. Samples are to be returned to Sample Control immediately following analysis.

5.0 FINAL DISPOSITION OF SAMPLES

- 5.1 Commercial samples are kept for at least 90 days from the time the samples are received. After 90 days the samples are disposed of unless otherwise specified by the client. Disposal of all samples must be recorded in Sample Disposal Logbook.
- 5.2 Samples submitted for inorganic analysis under the Contract Laboratory Program will be retained for a period of 60 days after data submission. Extracts of samples submitted for organic analysis will be retained for a period of one year after data submission.
- 5.3 After the retention period, the samples are disposed of unless a written notice to the contrary is received. Ultimate disposition of all samples must be recorded in the Sample Disposal Logbook.

SOP D.1, Analytical Division Laboratory Waste Handling and Storage Procedures provides detailed guidance on the disposal of laboratory samples.

Figure 2
Form DC-1

SAMPLE LOG-IN SHEET

Lab Name		Page <u> </u> of <u> </u>		
Received By (Print Name)		Login Date		
Received By (Signature)				
Case Number	Sample Delivery Group No.	SAS Number		
Remarks: 1. Custody Seal(s) Present/Absent* Intact/Broken 2. Custody Seal Nos. _____ 3. Chain-of-Custody Records Present/Absent* 4. Traffic Reports or Packing Lists Present/Absent* 5. Airbill Airbill/Tracker Present/Absent* 6. Airbill No. _____ 7. Sample Tags Present/Absent* Sample Tag Numbers Listed/Not Listed on Chain-of-Custody 8. Sample Condition Intact/Broken*/ Leaking 9. Cooler Temperature Indicator Bottle Present/Absent* 10. Cooler Temperature _____ 11. Does information on custody records, Traffic Reports, and sample tags agree? Yes/No* 12. Date Received at Lab _____ 13. Time Received _____	EPA Sample #	Corresponding Sample Tag #	Assigned Lab #	
	Remarks		Condition of Sample	
	Sample Transfer			
Fraction	Fraction			
Area #	Area #			
By	By			
On	On			
* Contact SMO and attach record of resolution.				
Reviewed By	Logbook No.			
Date	Logbook Page No.			

Figure 5
GPL Work Order



Work Order Approval

Page 1 of 1

Work Order #: 211092
 GPL P Amy Edwards
 Project #: 7833
 Project Nam L14 25024

Date Received Nov-16-2002
 Fax Due Date:
 HC Due Date: Dec-16-2002
 EDD Due Date Dec-16-2002

Client: O'Brien & Gere
 Address: 7001 North Atlantic Ave, Suite 202
 Cape Canaveral, FL 32920

Contact: Roger Baldwin
 Phone: 3217992200
 Fax: 3217998115
 E-Mail:

APPROVED

Comments: EXCEL EDD
 GPL Level IV

Lab ID : 211092-001 Field ID : MCW02-5 Date Collected : 14-NOV-02

Method Name	tx	Cont	Storage Loc	resev Class	AT	omments
Mercury by EPA 7470A	W	2	A-5B	NCPH2	28D	
Residue, Filterable (TDS) by EPA 160.1	W	2	A-5B	COOL	28D	
Total Metals by EPA 6010B	W	2	A-5B	NCPH2	7D	

Lab ID : 211092-002 Field ID : MCW02-3 Date Collected : 14-NOV-02

Method Name	tx	Cont	Storage Loc	resev Class	AT	omments
Mercury by EPA 7470A	W	2	A-5B	NCPH2	28D	
Residue, Filterable (TDS) by EPA 160.1	W	2	A-5B	COOL	28D	
Total Metals by EPA 6010B	W	2	A-5B	NCPH2	7D	

Lab ID : 211092-003 Field ID : MCW02-4 Date Collected : 14-NOV-02

Method Name	tx	Cont	Storage Loc	resev Class	AT	omments
Mercury by EPA 7470A	W	2	A-5B	NCPH2	28D	
Residue, Filterable (TDS) by EPA 160.1	W	2	A-5B	COOL	28D	
Total Metals by EPA 6010B	W	2	A-5B	NCPH2	7D	

Lab ID : 211092-004 Field ID : MCWR02 Date Collected : 14-NOV-02

Method Name	tx	Cont	Storage Loc	resev Class	AT	omments
Mercury by EPA 7470A	W	2	A-5B	NCPH2	28D	
Residue, Filterable (TDS) by EPA 160.1	W	2	A-5B	COOL	28D	
Total Metals by EPA 6010B	W	2	A-5B	NCPH2	7D	

Approved By: _____

Date and Time Approved: _____

Figure 6
Subcontracted C-O-C

Samples Transferred To: _____

 Alt: _____
 Phone: _____

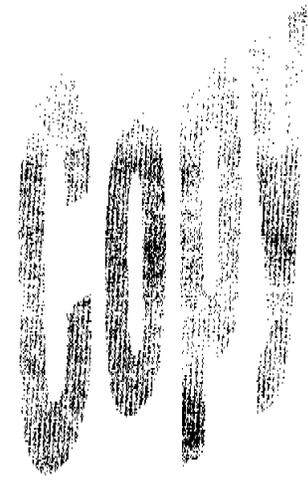
Chain of Custody Form for
Subcontracted Analytes

CP W.O. No.: 211107
 CP P.O. No.: _____

GPL Laboratories, LLLP
 202 Perry Parkway
 Cambridge, MD, 20877
 (301) 926-6802
 (301) 840-1209 (Fax)

GP Lab Sample ID	Field Sample ID	Date Sampled	Time Sampled	Matrix	Analysis Requested	Method	Type of Container	Preservative
211107-002-023-1/1	E19-GW00-45-049	15-NOV-02	08:20	WATER	ALPHABETA BY METHOD 900.0	E900.0	1 L HDPE	NCPH2
211107-003-029-1/1	E19-GW00-45-050	15-NOV-02	08:30	WATER	ALPHABETA BY METHOD 900.0	E900.0	500 ML HDPE	NCPH2

Carrier: _____
 Report Due On: _____
 Condition Upon Receipt: _____
 Comments: _____
 Samples Relinquished By: _____ Date: _____ Time: _____
 Ambill No.: _____
 Sent Report Attn: _____
 Perform Q.C. on Sample: _____
 Samples Received By: _____ Date: _____ Time: _____



UNCONTROLLED DOCUMENT
AND
BUSINESS CONFIDENTIAL

SOP No: G.12

Title: Standard Operating Procedures for Submitting Reports

Scope: This standard operating procedure describes the procedures to be used by reports generation personnel in the generation of sample data reports and electronic data deliverables (EDD) to be sent out to clients.

1.0 WORKORDER RECEIPT, CHECKING AND FILING

- 1.1 Upon delivery of samples, the coolers are unpacked by the Sample Receiving personnel and the samples are checked against the chain of custody for discrepancies.
- 1.2 The samples are logged into the LIMS system and a work order is created. The work order is then forwarded by Sample Receiving to the Project Manager for approval. Once the workorder is approved, it is forwarded to the reporting generation department who then assembles a work order file consisting of a work order with fraction test list, the chain-of-custody form, the subcontracted chain-of-custody form (if applicable), the sample receipt form and the shipping label, if applicable.

2.0 DATA ENTRY INTO LIMS

- 2.1 All data acquired from sample analysis for each Workorder as per required test or Job must be entered into the LIMS database either manually or 99.9% of sample analysis is acquired automatically with XENCO.

2.1.1 Organics and Inorganics analyses

Results for all analyses are exported to the LIMS database by the analyst or supervisor in the laboratory. The exporting of data to the LIMS is accomplished through use of various software.

2.1.2 Subcontracted Analysis

The subcontracted analysis report must have the following parameter fields; analyte, result (concentration), analyzed date, preparation date (if required), detection limit and units. If any information is missing, the subcontracted lab has to be contacted to obtain the required parameters.

3.0 GENERATION OF LIMS REPORT

Data that has been exported into the LIMS data entry system database can be used to generate a LIMS report which can then be used for different reporting purposes.

3.1 Reporting Options

To generate a LIMS report using GPL LRD, first open GPL LRD by double clicking the GPL LRD icon on desktop. Choose the "STATUS" tab from the list of 5 tabs across top of screen by clicking on it once. Enter GPL work order number in box "SDG". Upon entering work order number click the "GO>" button once, which is located directly right of the GPL work order number. After clicking the "GO>" button a list of batches in LRD will appear. Once the batches in LRD appear, click the "Full Summary" box on the right hand side of screen one time. After the "Full Summary" box has been clicked, the LIMS report will be generated and appear on user's screen. Review data on screen by clicking arrow buttons back and forth to different pages. Print report by pressing print button on menu bar.

4.0 REPORTING DATA TO THE CLIENT

After data has been entered and a LIMS report generated and checked, the data is ready to be reported to the client. Data can be reported in various formats depending on the client's needs. The client may opt for faxed or e-mailed results, complete hard copy results, CD's, or all 4 options.

4.1 Faxed or E-mailed Results

4.1.1 Clients often request to have their sample results faxed or e-mailed by a specific date. The date that the results are due depends on the turn around time requested by the client and can be found on the work order sheet. The data required for fax or e-mail is sample results only.

4.1.2 The LIMS report is faxed or e-mailed in its entirety or partiality as requested by the client. When faxing, each work order is logged into the fax logbook which can be found next the fax machine. The date, time, client, contact, fax number and any comments is logged into the fax logbook. The above information is taken from the client information section of the work order sheet. After the results have been faxed, the confirmation form from the fax is placed in the work order file.

4.2 Packages (when required)

4.2.1 Depending on the report type requested by the client, a completed hardcopy and/or CD is sent to the client on the due date as determined by the contract turn around time.

4.2.2 After the completed package from each department is submitted to the report generation department, its status is updated on the daily status reports from missing to complete. When packages for work orders that do not require faxing get turned in to the reports generation as complete, their status is noted as complete in the daily status report.

4.2.3 All packages that are ready to be sent to the client are paginated and copied. The client gets the copies, the number of which depends on the contract or client request. Different clients get different types of GPL report packages. The different types of report packages are GPL Level I, II, III and IV packages. All completed packages are scanned in its entirety and a CD is burned and stored in the Reporting Dept.

4.3 Data package Verification

4.3.1 When a department turns in a completed hardcopy package, it must have been checked for completeness and accuracy by the preparer (analyst) and that department's supervisor or designate. Both personnel must sign on the case narrative and conformance summary report.

4.3.2 When the package gets to reports generation, the technician that prepares the work order package and sends it out to the client also checks for the completeness of the package and, when completed, a data package check list must be signed.

4.3.3 A complete report package will always include:

4.3.4.1 A cover page

- Signatures by the Laboratory Director and Project Manager or designees, with the printed title.
- The address and phone number of GPL Laboratories, LLLP.
- The client's name, project name and project manager name, which appears on the cover page.
- The date the report was issued.

4.3.4.2 A copy of the chain-of-custody, which includes the date and time of sample collection, preservations, sample analysis requested and date and time of the sample receipt. The shipping label is included also.

4.3.4.3 A sample receipt checklist that is used to document the condition of the samples upon arrival at the laboratory.

- 4.3.4.4 An Analytical Summary Report that includes the reporting limits, test results, methods, date and time of sample collection, analysis and preparation for each sample. The Report also contains the work order number, fraction numbers, and the client's identification for each sample.
- 4.3.4.5 A case narrative that details the condition of the samples upon arrival at the laboratory. The case narrative also documents any quality control failures or deviations from the methods cited.
- 4.3.4.6 A definition page that provides a key for the notes used in the analytical summary report.
- 4.3.4.7 An original copy of all subcontracted data and supporting documentation (if applicable).
- 4.3.4.8 For the non-NELAP accredited analysis, the report will be flagged and the information will be stated in the case narrative
- 4.3.5 After the package has been checked for completeness, it is assembled according to the package type as designated on the work order sheet. It is then paginated. A table of content sheet is generated according to the content of the package. The final package is copied and the copies packaged for shipping. The analytical summary report (including the Cover Page), Chain-of-Custody, Sample Receipt Checklist, Qualifier page, and the Case Narrative will be paginated.
- 4.3.6 Prior to signature by the Project Manager, the entire data package is reviewed for: completeness, confirmation that correct data package was presented, and that the correct mailing address was used. The Case Narrative is also reviewed, edited and signed by the Project Manager and Lab Director. The project manager then returns the completed package to the reporting department for corrections, if necessary, and the completed package is sent to the client.
- 4.3.7 After the report has been sent, the sent date and the mailing method, e.g., Federal Express, UPS, Courier or mail is noted on the work order sheet.

5.0 GENERATION OF STATUS REPORT

- 5.1 The daily status report is generated each and every work day. It is used in the daily production meeting to track the status of all work orders in house.

6.0 GENERATION OF ELECTRONIC DATA DELIVERABLES (EDD/ADR)

Most clients will request to have their data submitted separately in an electronic format (EDD/ADR). The format of an EDD/ADR varies greatly from client to client and even within different projects for a single client. EDDs/ADRs are produced using customized programs and applications that query the GPL database and then reformat the data to suit individual client needs. The resulting EDD/ADR file is then submitted to the client via e-mail. The process has 3 steps:

- Locking the work order in the database
- Generating and reformatting the data, and
- Submitting the data to the client.

6.1 Locking the work order

6.1.1 This process is accomplished by opening an application that links to the GPL database and allows the user to enter the work order number to be locked. The user then selects a check box in the application and locks the work order. Locking a work order prevents any further modifications being made to the data by the lab. Only after locking occurs can data be generated for an EDD/ADR.

6.2 Generating and Reformatting the EDD/ADR

6.2.1 Most EDDs/ADRs are generated by querying the GPL database using Microsoft Excel and its query feature. For each client the EDD/ADR analyst will prepare a separate customized generator based on the client's specified EDD/ADR requirements. The generator lays out the data fields and automatically converts certain data (for example dates) to the format required by the client within the framework of the Excel program. The data in the EDDADR is then reviewed against the hard copy report and any correction of data is performed. Once the data has been reviewed the EDD/ADR file can be saved either as an Excel file if that is the client's need or saved and converted to various other file formats directly from Excel or by exporting the data to Microsoft Access.

6.2.2 If a client requests an EDD/ADR but does not supply requirements, GPL has a standard format that can be used.

6.3 Archiving and Submitting the EDD/ADR

6.3.1 Completed EDD/ADR files are saved to a specified directory on GPL's server. Each client has a separate folder to hold that client's EDDs/ADRs. The files are stored in the EDD/ADR directory indefinitely and the folder is routinely backed up to tape.

- 6.3.2 Once the file(s) has been saved the ED/ADR analyst will generally then e-mail the EDD/ADR file(s) for the work order to that clients GPL project manager who will submit the EDD/ADR to the client as required. Other methods of delivery include direct e-mail, saving the file to the data package CD or direct transmittal of the EDD/ADR from the analyst to the client.

7.0 ARCHIVING COMPLETED WORKORDERS

Work orders that have been sent out, checked for completeness and the data package check list signed by the project manager, is ready to be archived. Completed work orders are archived either in-house or offsite.

7.1 In-house Archiving

- 7.1.1 Work orders that are ready to be archived are filed in a file cabinet in the reports generation section by month. As space fills up work order files from earlier months are removed to make room for work order files from recent months. As workorders from earlier months are removed, placed in archived boxes and the boxes are given a number in sequence, generated from a list that details work orders and their box numbers. This list is maintained conservatively. As the boxes are filled up with workorders and given a number, they are moved into an in-house storage space designated for the storage of archived work orders. The in-house storage area is kept locked and accessible only to authorized personnel.

7.2 Retrieving Work Orders from in-house Archive Location

- 7.2.1 When a file is stored in-house and is later needed by a Project Manager, the Project Manager must sign out the report and/or CD on the sign out sheet, which is located in the reporting department table next to the storage area. When the report and/or CD is returned back to the storage area, it is then signed back in on the sign in sheet. The storage of CD's are kept in a secure area and only accessible to authorized personnel.

7.3 Offsite Archiving

- 7.3.1 When the space designated for in-house storage becomes full, older archived boxes are moved to an offsite location. A list is maintained that catalogs the date that the boxes were moved to an offsite location (Iron Mountain), the boxes that were moved and the corresponding box number that were designated for each box by the offsite warehouse.

- 7.4 Retrieving Work Orders from Offsite Archive Location
 - 7.4.1 Archived work orders from the offsite location can be requested to be sent back to the lab if needed. To do this a call is placed to the offsite location (Iron Mountain). The operator will request that the caller provide them with our account number, password, the box the desired work order is located in and it's corresponding offsite number. When the work order is no longer needed, it is returned to the warehouse.

- 7.5 Revisions, Additions or Deletions to Previously Issued Reports of Analysis
 - 7.5.1 Any changes made to a final Report must be documented using "Report of Analysis Revision/Addition/Deletion Form"(Fig 1). Copies of the form are available from the Reporting Department supervisor. The form will be completed by the person requesting a revision to a previously issued Report of Analyses. The appropriate section of the form will be checked and the reason for the revision, addition or deletion will be described in detail.

 - 7.5.2 The completed Form, will be returned to the Reporting Department along with any supporting documentation (i.e., copies of logbooks, chromatograms, etc.). The requested changes will be made to the Report of Analysis. The entire data package, including the completed "Report of Analysis Revision/Addition/Deletion Form' will be given to the Lab Director for review. If the Lab Director approves the changes, he will sign on the indicated line of the form.

 - 7.5.3 When the data package and signed Form is returned to the Reporting Department, they will send the revised Report of Analysis to the client along with any required supplemental documentation. The original form and all supplementary documentation will be archived with original data package.

Fig 1

GPL Laboratories, LLLP

Report of Analysis Revision/ Addition/ Deletion Form

Work Order#: _____

Client: _____

REVISION	Date of Revision: _____
Reason for Revision: _____	

ADDITION:	Date of Addition: _____
Reason for Addition: _____	

DELETION:	Date of Deletion: _____
Reason for Deletion: _____	

Revision / Addition / Deletion Approved by:

Project Manager: _____

Lab Director: _____

SOP No: H.8

Title: Acid Digestion of Aqueous Samples, EP and TCLP Extracts and Wastes that Contain Suspended Solids for ICP and ICPMS Analyses in Accordance with SW846 Method 3010A.

Scope: The method detailed in this procedure is performed to prepare aqueous samples and extracts for quantitation of certain metallic analytes using Inductively Coupled Plasma (ICP) and Inductively Coupled Plasma/Mass Spectrometer (ICPMS) in accordance with SW 846 method 3010A.

1.0 PURPOSE

1.1 The method detailed in this procedure is used to prepare aqueous samples and extracts for analysis using an Inductively Coupled Plasma (ICP) and (ICPMS) spectrophotometer. The sample holding time before digestion is 180 days. Samples must be stored in refrigerator at 4°C until time of preparation. The elements to be analyzed using this procedure are:

Ag, Al, As, Ba, Be, Cd, Ca, Cr, Co, Cu, K, Fe, Pb, Na, Ni, Mg, Mn, Se, Sb, Tl, V, Zn, B, Sr, Ti, Sn, Mo, Te, Zr.

2.0 REFERENCES

- SW846 method 3010A revision 1

3.0 EQUIPMENT AND SUPPLIES

- 150mL glass beakers
- 100ml volumetric flasks (Class A)
- Hot plate
- Whatman No. 41 filter paper
- 125ml sample bottle (plastic)
- Watch glass (ribbed)
- Plastic disposable funnels
- Thermometer, calibrated, NIST traceable
- Fume hood
- Pipettors (calibrated) VWR Calibrated M23 & M24
- 100ml graduated cylinders

4.0 REAGENTS

- Concentrated Nitric Acid - trace metals grade
- Concentrated Hydrochloric Acid - trace metals grade
- 1:1 Hydrochloric Acid - to 500ml ASTM type II water (see below) add 500ml of Concentrated Hydrochloric Acid
- Metals Standards - (ICP SPK 1,2,3) commercially prepared NBS traceable metals standards with documented concentrations, including impurities and expiration dates. (Vendor: High Purity Standards, Charleston, SC). The spiking solutions cannot be used past the expiration date located on the label.
- Grade and quality of water required is ASTM Type II water (ASTM D1193): Water must be monitored for changes in conductivity by laboratory staff and is currently provided by a laboratory pure water system.

5.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 5.1 All sample containers must be collected in glass or polyethylene containers. Water/aqueous samples must be preserved with nitric acid to pH less than 2 immediately after collection.
- 5.2 All samples must be iced or refrigerated at 4 degree C (+/- 2) from the time of collection until digestion. The maximum holding time for metals is 180 day.
- 5.3 For determination of dissolved metals, the sample must be filtered through a 0.45 micrometer (um) pore diameter membrane filter at the time of collection or as soon as possible. Use a portion of the sample to rinse the filter flask, discard this portion and collect the required volume of filtrate. Preserve the filtrate with nitric acid to pH less than 2 immediately after filtration.

6.0 PROCEDURE (see Figure 1 for flow chart)

6.1 Sample Digestion Procedure

- 6.1.1 Mix the sample thoroughly to achieve homogeneity. Transfer 100ml of sample using a graduated cylinder to a beaker. Transfer 100ml of sample each for matrix spike and matrix spike duplicate analysis and label as matrix spike and matrix spike duplicate. For extracts of TCLP or highly contaminated wastes, reduce size to 10.0ml.
- 6.1.2 If aqueous samples are to be analyzed, to the matrix spike beaker, add 0.1ml of matrix spike solution (ICP spk 1,2,3). Add 0.2ml for TCLP extract.

- 6.1.3 If sample extracts are to be analyzed, to the matrix spike beaker, add 0.1mL of matrix spike solution (ICP spk 1,2,3). Add 0.2ml for TCLP extract.
- 6.1.4 Label one empty beaker "BKS" for the laboratory control sample. Add 100ml ASTM type II water to the beaker. Add 0.1ml of matrix spike solution (ICP spk 1,2,3). Add 0.2ml when doing TCLP extract.
- 6.1.5 Label one empty beaker "BLK" for the preparation blank. Add 100ml ASTM type II water.
- 6.1.6 To all beakers, add 3mls of concentrated nitric acid (HNO_3) and cover the samples with ribbed watch glasses.
- 6.1.7 Heat on a hot plate in the fume hood and evaporate until volume is approximately 5mls. Do not boil or allow beaker to go dry. Remove beaker from hot plate and allow to cool.
- 6.1.8 After cooling, add 3mls of concentrated nitric acid and cover samples with a ribbed watch glass. Return to hot plate and increase temperature to reflux gently.
- 6.1.9 Continue to heat, adding additional acid if necessary, until the digestate is light in color. Uncover the samples and evaporate to approximately 3mls.
- 6.1.10 Remove samples from hot plate and allow to cool. Add 10ml of 1:1 Hydrochloric acid.
- 6.1.11 Cover the beakers and reflux for 15 minutes.
- 6.1.12 Wash down beaker walls and filter sample, if necessary, through Whatman No. 41 filter paper (or equivalent) using disposable funnel. Dilute to 100ml in volumetric flask with Type II water.

NOTE: The diluted digestate solution contains approximately 5% (v/v) HCL and 3% HNO_3 . Transfer to 125ml plastic sample bottle and label with GP work order, fraction and date of digestion. Date of digestion may be put on the box of digestates, instead of on each bottle. For analysis, withdraw aliquots of approximate volume. The sample is now ready for analysis.

7.0 QUALITY CONTROL

7.1 Troubleshooting and corrective action.

- 7.1.1 The temperature should be maintained between 90-95°C on each hot plate / hot block. The temperature of the hot plate / hot block is documented on the bottom of the digestion log.

7.1.2 ICP operators should report to his/her supervisor and lab manager any recoveries outside warning limits for LCS samples for analytes being determined or preparation blanks are above control limits. Sample recoveries for any element which are outside of the control established limits for the laboratory control sample or contaminated preparation blanks are deemed unacceptable. The digestion batch must be re-digested for those analytes. Document the incident on a re-digestion form and submit to supervisor.

8.0 SAFETY

8.1 Safety equipment required

- Fume hood - minimum flow of 100 linear feet/minute
- Safety glasses
- Safety gloves (unpowdered)
- Lab apron
- Face shield, if necessary

8.2 Potential hazards

The most hazardous chemical acids that laboratory personnel are likely to encounter are strong acids such as Hydrochloric Acid and Nitric Acid (HNO₃).

8.3 Special handling requirements

Analysts should always read the label on the bottle. Chemicals require handling with care to include wearing adequate garments for skin protection. Also, acid use should be performed under a ventilated hood.

9.0 DISPOSAL REQUIREMENTS

9.1 Acid wastes should be placed into the acid waste bottle, which is located in the metals digestion lab. Any remaining samples should be returned to sample control. More details concerning disposal characteristics and procedures can be located in the SOP D.1 "Laboratory Waste Handling and Storage Procedure".

10.0 POLLUTION PREVENTION

10.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

11.0 DEFINITIONS

11.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

12.0 REPORTING REQUIREMENTS

- 12.1 Documentation to include Work Orders and Work Sheets (see SOP "Sample Logging and Record Keeping"), Metal Digestion Log Forms must be submitted to the Metals Supervisor with the Digestion Technician's initials and the preparation date documented on each form for each case.

Sample Description Information must be filled out and should contain the following information for aqueous digestates. The fields for color and clarity, before and after digestion, must be completed. The following descriptive terms are recommended:

Color - red, blue, yellow, green, orange, violet, white, colorless, brown, gray, black

Clarity - clear, cloudy, opaque

Note any significant changes that occur during sample preparation (i.e., emulsion formation) in the Comments section. Enter any sample-specific comments concerning the analyte results in the comments section.

Metal Digestion Log Forms (Figure 2) must be documented completely by the Digestion Technician during digestion include the date of digestion, work order number, digestion technician signature, supervisor approval, identification of method used, GPL fraction ID, sample matrix (soil/water), amount of sample used in digestion and final volume of sample, identification of the matrix spiking solution used and the amount used.

- 12.2 QC records are maintained in the form of control charts to document percent recovery of analytes from EPA ICV and independent laboratory control samples subjected to the digestion procedure.

Figure 1
FLOW CHART

ACID DIGESTION OF AQUEOUS SAMPLES OR EXTRACTS
FOR ANALYSIS ICP/FLAME AA OR SB BY GFAA

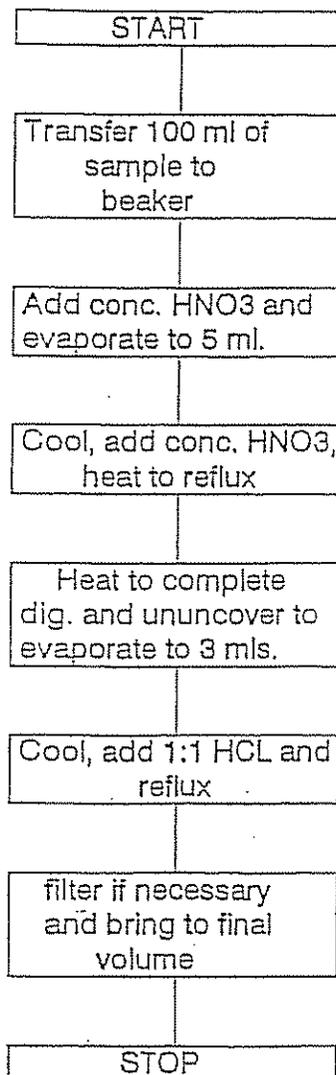


Figure 2
Metal Digestion Log Form

Date: Prep: 200.7 / 3005A / 3020A / 3050B / 3010A / ILC03.1 / ILM04.1 / ILM05.3 Batch No: 185-
Analyst: _____ Comments: _____

Reviewed by: _____ Date: _____ Soils Witness: _____ Rep. Seq: _____

GPL Work Order No.	Sample	Frac	Size mL (g)	Final Vol. (mL)	Matrix S, W Other	Color Before	Color After	Clarity Before	Clarity After	Soil Artifacts
						R, Bl, Y, G, O, V, W, Colorless, Br, Grey, Bk.		or Soil Texture	Clear, Cloudy, Opaque	or Water pH
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										
11										
12										
13										
14										
15										
16										
17										
18										
19										
20										
21										
22										
23										
24										
LCS ID/ Amt. Added (ml ,g) :								HNO3(1+1) ID:		
Spiking ID / Amt. Added (ml) :								HNO3(conc) ID:		
								HCl(conc) ID:		
Temperature C:								HCl(1+1) ID:		
								Peroxide ID:		

SOP No: H.10

Title: Trace ICP Quantitation of HSL Metals plus Boron, Molybdenum, Silicon, Strontium, Titanium, and Tin According to Method 6010B

Scope: The method detailed in this procedure is for the analysis of water, TCLP and EP extracts, soils, sludges, sediments and other solid wastes digestates for Hazardous Substance List (HSL) Metals by Inductively Coupled Plasma (ICP) spectroscopy in accordance with USEPA method 6010B. The use of ionization buffers, internal standards, and special background correction techniques is specified.

1.0 INTERFERENCES

1.1 There are four main categories of interferences: unresolved overlap of molecular band spectra, stray light, overlap from nearby spectral lines, and background emission from continuous or recombination phenomena.

2.0 PURPOSE

2.1 The purpose of this procedure is to describe the simultaneous analysis of metals on the USEPA Hazardous Substances List (Antimony, Arsenic, Lead, Selenium, Silver, Thallium, Sodium, Potassium, Aluminum, Barium, Beryllium, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Magnesium, Manganese, Nickel, Vanadium, and Zinc) plus Boron, Molybdenum, Strontium, Titanium, and Tin at trace levels using a Thermo-Jarrell-Ash 61E Purged Trace Inductively Coupled Plasma Spectrometer and autosampler. All samples are digested in accordance with SOP H.4 or H.5 prior to analysis. Filtered samples for dissolved metals analysis can be analyzed after either digestion or matrix matching. The digestate holding time is 180 days.

2.2 Prior to use this method, the samples should be prepared using appropriate sample preparation methods (SOP#H8 – 3010, SOP#H21 – 3050)

3.0 REFERENCES

- TJA ICAP 61E Operator's Manual (p/n 134542-00).
- SW846 method 6010B revision 1.

4.0 CALIBRATION PARAMETERS

<u>Element</u>	<u>Wavelength</u>	<u>Element</u>	<u>Wavelength</u>
Ag	3280	Mo	2020
Al	3082	Na	3302
As	1890	Ni	2316
B	2496	Pb	2203
Ba	2347	Se	1960
Be	3130	Sb	2068
Ca	3179	Si	2881
Cd	2265	Sn	1899
Co	2286	Sr	4125
Cr	2677	Ti	3349
Cu	3247	Tl	1908
Fe	2714	V	2924
K	7664	Y	3710
Mg	2790	Zn	2062
Mn	2576		

5.0 EQUIPMENT AND SUPPLIES

- 100mL volumetric flasks (Class A)
- 200mL volumetric flasks (Class A)
- 500mL volumetric flasks (Class A)
- 1000mL volumetric flasks (Class A)
- 100mL plastic storage bottles
- 250mL plastic storage bottles
- 500mL plastic storage bottles
- 1000mL plastic storage bottles
- 20.0L Nalgene carboy
- 10.0L Nalgene carboy
- 15mL disposable autosampler tubes
- 28mL disposable autosampler tubes
- Centrifuge tube holder
- Pipetters - Wheaton calibra M25 Finnpiptette II M26
- Pipette Tips
- 5, 10, 20mL Class A volumetric pipets
- Pump windings and tee fittings
- Argon gas (cryogenic liquid source)
- Nitrogen gas (cryogenic liquid source) used for purging of spectrometer

5.1 Instrumentation

- 5.1.1 TJA 61E Trace ICP with nebulizer, cyclonic spray chamber, horizontal torch, and AS-192 autosampler.
- 5.1.2 Simultaneous background correction technique is used for the analysis of lead and selenium to achieve lower instrumental detection limits comparable to graphite furnace.
- 5.1.3 Yttrium Internal standard - Lithium ionization buffer is added on-line using a mixing tee and coil with a ratio of 1:4 (one part standard: four parts samples, resulting in a dilution factor of 5 for the internal standard, approximated 10ppm is the final "mixed-in" concentration.

6.0 REAGENTS

- A deionized water ASTM type II or equivalent
- Concentrated hydrochloric acid, trace metals grade
- Concentrated nitric acid, trace metals grade
- Flame Water - 5% hydrochloric acid, 1% nitric acid

Preparation of Reagent Flame Water

Fill a 20L Nalgene carboy half full with type II water. Add 200ml concentrated nitric acid and 1000ml concentrated hydrochloric acid underneath a hood to contain noxious gases. Dilute to twenty liters with type II water and mix thoroughly.

5% hydrochloric acid, 1% nitric acid

5ppm Arsenic Profile check solution

Preparation of Arsenic Solution:

Pipette 2.5mL of 1000ppm Arsenic stock solution into a 500mL volumetric flask. Dilute to volume with matrix matched water. Transfer to a bottle labeled 5ppm As. Record the date of preparation, expiration date and preparers initials on the bottle label. Prepare Arsenic Profile solution every three months or when depleted, whichever is more frequent.

50ppm Y-1500ppm lithium internal standards ionization buffer

Preparation of 50ppm Y-1500ppm Lithium Internal standard-ionization buffer:

Pipet 100mls of GP mix Li into 1000ml volumetric flask. Dilute to volume with flame water.

All stock and prepared standards are logged into a software program (solutions manager). All stock and prepared standards are assigned a solutions manager reference number. The reference number expiration date, prep date and chemist initials are written on a label and placed on all prepared solutions. The expiration

date chosen by solutions manager for prepared standards is three months for the prepared date.

7.0 GENERAL PRECAUTIONS

- 7.1 AVOID CONTAMINATION of STOCK STANDARDS. Always pour a small volume of standard stock solution into a new microbeaker before pipetting an aliquot. NEVER insert a pipette directly into the bottle. This precautionary measure also applies to quality control standards stock solutions (ICVA, IC1, etc.)
- 7.2 Check pipetters daily for leaks and proper calibration. Record in the pipette log book.
- 7.3 Empty Drain vessel at the end of each day. Transport waste to the waste disposal area for appropriate treatment prior to shipment. Fill vessel up to 6 inches using tap water prior to replacing vessel beneath instrument.
- 7.4 Clean up and neutralize all spills immediately to avoid corrosion damage to the instrument.
- 7.5 Purge the optics with nitrogen at all times. Leave the RF power unit on at all times. Never attempt to perform repairs to the High voltage systems. Leave the instrument PM tubes and heater on at all times.

8.0 PROCEDURE

8.1 Preparation of Calibration Standards

- 8.1.1 To prepare calibration Standard 1 in flame water, pipette 25mL of source solution Sp Mx Std. 12 into a 500mL flask, which has been half filled with flame water. Dilute the flask to volume with flame water. Mix the solution thoroughly, and transfer it to a plastic bottle.

True value of Standard 1

<u>Element</u>	<u>Certified Std. Conc.(ug/ml) Added</u>	<u>Volume (ml)</u>	<u>Calibration Std. Conc. (ug/L)</u>
Aluminum	1000	25	50000
Antimony	10	25	500
Arsenic	10	25	500
Barium	10	25	500
Beryllium	1.000	25	50
Boron	10	25	500
Cadmium	10	25	500
Chromium	10	25	500
Cobalt	10	25	500
Copper	10	25	500
Iron	1000	25	50000
Lead	10	25	500
Manganese	10	25	500
Molybdenum	10	25	500
Nickel	10	25	500
Selenium	10	25	500
Silicon	10	25	500
Silver	10	25	500
Strontium	10	25	500
Thallium	10	25	500
Tin	10	25	500
Titanium	10	25	500
Vanadium	10	25	500
Zinc	10	25	500

8.1.2 To prepare calibration Standard 2 in flame water, pipette 50mL of source solution Sp Mx Std. 12 into a 500mL flask, which has been half filled with flame water. Dilute the flask to volume with flame water. Mix the solution thoroughly, and transfer it to a plastic bottle.

True value of Standard 2

<u>Element</u>	<u>Certified Std. Conc.(ug/ml) Added</u>	<u>Volume (ml)</u>	<u>Calibration Std. Conc. (ug/L)</u>
Aluminum	1000	50	100000
Antimony	10	50	1000
Arsenic	10	50	1000
Barium	10	50	1000
Beryllium	1.000	50	100
Boron	10	50	1000
Cadmium	10	50	1000
Chromium	10	50	1000
Cobalt	10	50	1000
Copper	10	50	1000
Iron	1000	50	100000
Lead	10	50	1000
Manganese	10	50	1000
Molybdenum	10	50	1000
Nickel	10	50	1000
Selenium	10	50	1000
Silicon	10	50	1000
Silver	10	50	1000
Strontium	10	50	1000
Thallium	10	50	1000
Tin	10	50	1000
Titanium	10	50	1000
Vanadium	10	50	1000
Zinc	10	50	1000

8.1.3 To prepare calibration Standard 3 in flame water, add 10ml each of the Mg, Ca, Na stock (10,000ppm) and 1.25ml of K stock standard (10,000ppm) standard from High Purity standard with a Class A volumetric pipets to a 500mL volumetric flask which has been half filled with flame water. Dilute the flask to volume with flame water, mix the solution thoroughly, and transfer to a plastic bottle.

<u>Standard 3</u>	<u>Starting CONC</u>	<u>Volume Added</u>	<u>Final CONC.</u>
Sodium	10,000ug/ml	10ml	200000ug/L
Potassium	10,000ug/ml	1.25ml	25000ug/L
Magnesium	10,000ug/ml	10ml	200000ug/L
Calcium	10,000ug/ml	10ml	200000ug/L

NOTE 1: These formulations are subject to change without an update of SOP to suit various client requirements

NOTE 2: A multipoint calibration is performed daily for those elements in standards 1 and 2. Standard 3 is used to perform a five point calibration that is re-sloped daily.

8.1.4 Document the standard in solutions manager program. Label the standard solution bottle. The label should include the preparers initials, the date of preparation, the expiration date, and the page number on which the standard has been recorded in the log book. The expiration date of standard solutions is three months from the date of preparation or whenever one of the certified standards expires, whichever is first.

8.2 QC Preparation

8.2.1 Preparation of Low level linearity check Solution.(PQL)

Pipette appropriate volume of single element STP's into 1000ml volumetric flask. Fill to volume with flame water.

Note: Additional elements can be added, if necessary, according to client needs.

Document the solution in the Solutions Manager Software Program. Label the bottle as ICP Stock and record the preparation date, the expiration date, the preparers initials, and the ID number.

8.2.2 The CCV is made from the stock solutions used in making the calibration standards. The CCV concentrations are at half the concentrations of the highest standards used in calibration.

8.2.3 Initial Calibration Verification (ICV)

Fill a 1L volumetric flask halfway with flame water and add ten mLs of ICV, certified stock solution. Dilute the flask to volume with flame water. Mix the solution thoroughly and transfer to a plastic bottle labeled ICV.

8.2.4 Interference Check Standard A (ICSA):

Fill a 500mL volumetric flask halfway with flame water and add 50mL of ICSA certified multi-element stock solution. Dilute the flask to volume with flame water. Mix the solution thoroughly and transfer to a plastic bottle labeled ICSA.

8.2.5 Interference Check Standard AB (ICSAB):

Fill a 500mL volumetric flask halfway with flame water and add 50mL of ICSA certified stock solution, and 5mL of SM-421-012 and 10mL of CLP calibration mix #2. Dilute the flask to volume with flame water. Mix the solution thoroughly and transfer to a plastic bottle labeled ICSAB.

8.3 Preparation of 1:5 Serial Dilution (L)

Obtain the sample digestates for the case or SDG to be analyzed. Take the original sample digestate that corresponds to the sample designated for duplicate and matrix spike digestions for each SDG and matrix and prepare its serial dilution as follows:

Transfer 2mls of sample into 8mls flame water. Mix thoroughly.

8.4 Preparation of Post Digestion Spike

Obtain the sample digestates for the batch or SDG analyzed. Take the original sample digestate that corresponds to the sample designated for duplicate and matrix spike digestions for each batch and prepare the post spike as follows.

Transfer 10ml of sample into a sample tube. Remove 0.1mls of sample, then spike with 0.1ml of post spike solution. Mix thoroughly.

Note: If the matrix spike recovery is outside criteria, the sample may require spiking at alternate concentrations

8.5 Tuning and Calibration of the ICP

8.5.1 Conduct a pre-start up inspection.

8.5.1.1 Argon gas supply and drain vessel

Make sure there is an adequate supply of Argon. The argon line pressure regulator should be set at 60psi.

Check the drain vessel beneath the ICP and empty it if full.

8.5.1.2 Torch box

Make sure all connections are secure and air tight, including the drain hose, nebulizer cap, argon lines.

8.5.1.3 Peristaltic pump

Install new flexible pump tubing every other day (the windings have three stops which allow for an extra day of use) or if the old one shows signs of flattening or stretching, and connect to the nebulizer with capillary tubing.

8.5.2 Daily Start Up

8.5.2.1 Reset computer and instrument. Load the operating software called Thermospec by using the windows icon under Thermospec window or by typing "STNRUN" at the C: drive prompt in DOS.

8.5.2.2 Ignite plasma. Engage pump tubings. Under the menu heading SET UP, select CONTROL PANEL; then press F1 for Start Up followed by F9 for continue to begin the start up sequence which takes about 90 seconds.

8.5.2.3 Warm-up. Once the torch has been successfully lit, exit the start up submenu and go to the analysis menu. At the method prompt, enter "6010" and the peristaltic pump should begin turning and the levels adjusted to following:

Torch gas = HIGH
Auxiliary gas = LOW
Nebulizer gas = 0.588mL/min
Approximate RF Power (W) = 950
Pump rate (RPM) = 110

Fill the rinse water reservoir with the same matrix as the samples and set rinse time for 120 seconds. Fill the internal standard - ionization buffer reservoir.

8.5.3 Profile and prepare for sample analysis.

8.5.3.1 Place a 28ml autosampler cup filled with 5ppm As onto the last position (#19) on the "L" rack of the autosampler. Under the analysis submenu, press F6 to move autosampler and begin profile sequence. Once the autosampler has moved it will wait 35 seconds to allow for adequate uptake and equilibration of the test solution.

8.5.3.2 Start the profile sequence. Press F3 and then F1 to start the profile. The procedure takes approximately 63 seconds and returns a peak profile of the Arsenic line at 189.042 x 2nm (second order line).

8.5.3.3 Record the peak position and intensity in the daily maintenance logbook. The peak position should be within 0.1 units of the zero position. A drift greater than the specified tolerance could indicate a drastic barometric or thermal change since the last profile and warrants further investigation. (see trouble shooting.)

8.5.3.4 The calibration curve must consist of a blank and standards (refer to section 7.1.1). Use the average of two exposures for both standards and samples.

8.5.4 Prepare autosampler sequence.

Under Operation menu, select "Autosampler Setup". Load the default table name "trace" and enter the samples to be run under set 2. (maximum 192). Enter a CCV and CCB every 10 samples. Once finished, print out the table assignments by pressing F2.



8.5.5 Load autosampler with standards and samples according to the table printouts. A typical set-up should like this:

(set 1) Load autosampler L rack with 28mL cups

<u>Position</u>	<u>Standard</u>
5/45	ST00
5/46	ST01
5/47	ST02
5/48	ST03
1/1	CRI
5/42	PQL solution
5/44	ICV solution
5/43	ICB,CCB solution
1/2	ICSA solution
1/3	ICSAB solution
1/4	CRII solution
1/7 – 1/18	CCV solution (for long runs)
	CCB solution

(set 2) Load autosampler (48 position racks):

<u>Position</u>	<u>Name</u>
1	PBW (BATCH #)
2	LCSW (BATCH #)
3	SAMPLE
4	DUPLICATE D
5	SPIKE S
6	SERIAL DILUTION L
7	POST DIGESTION SPIKE
8	SAMPLE2
9	SAMPLE3
10	SAMPLE4
	CCV1
	CCB1
11 .. 20	10 more samples
	CCV1
	CCB1
21 .. 30	10 more samples
	CCV1
	CCB1
31 .. 40	10
	CCV1
	CCB1
41 .. 48,	8 more samples
rack 2, 1 .. 2	2 more samples
	CCV1
	CCB1

NOTE 1: The autosampler assigns the positions of the samples and QC in the order in which they are entered. Modifying an existing run by inserting samples may change the assignments of the samples.

NOTE 2: In order to take multiple uptakes from the same QC cup, the identical name must be entered. This would not allow for the numbering of CCV1/CCB1, CCV2/CCB2, etc. or the suffix of "I" or "F" on ICSA, ICSAB, PQL check solution used in CLP type packages.

8.6 Sample Analysis

8.6.1 Initiate autosampler run. Under the Operation menu, select Analysis, enter the method, press F9 for autosampler run; enter the desired autosampler table file and press F1, to start operation.

8.6.2 If the samples to be analyzed have been digested then all the calibration standards and quality control solutions used should be prepared using flame water.

8.6.3 Record the analysis sequence, the instrument identification, the date, the analyst's name, the analyst's signature, the time of analysis initiation, and the work order numbers on the bench sheet. Submit a copy of the bench sheet with the raw data.

8.7 Quality Control Requirements

8.7.1 IDL Study consisting of seven blanks run as samples must be performed every three months.

8.7.2 Calibration Curve

All analyses require that a calibration curve be prepared to cover the appropriate concentration range. The curve must have a correlation coefficient of 0.995. The calibration line is being generated using ordinary least squares. $y = mx + b$.

When multiple concentration standards are used, at least three calibration standards will be used. Alternatively, the initial calibration curve may be prepared daily with a minimum of a calibration blank and a single high standard. The resulting curve must then be verified with mid-level and low-level calibration verification standards. Acceptance range of +/- 20 % will be used for low-level calibration verification standard and +/- 10% for the mid-level calibration verification standard.

8.7.3 ICV/CCV

The ICV/CCV is run immediately after calibration. The CCV is run after every ten samples or every two hours and the end of the analysis sequence. The ICV and CCV must be within 10% of the calibration with RSD < 5% from replicate integrations. When measurements for

any element exceed the control limits the analysis is void for that element. The problem must be corrected and the samples reanalyzed.

Acceptance Criteria
ICV

<u>Element</u>	<u>Control Limits (ppb)</u>	<u>True Value (ppb)</u>
Aluminum	4500 - 5500	5000
Antimony	360 - 440	400
Arsenic	360 - 440	400
Barium	360 - 440	400
Beryllium	36 - 44	40
Boron	360 - 440	400
Cadmium	36 - 44	40
Calcium	4500 - 5500	5000
Chromium	360 - 440	400
Cobalt	360 - 440	400
Copper	360 - 440	400
Iron	4500 - 5500	5000
Lead	360 - 440	400
Magnesium	4500 - 5500	5000
Manganese	360 - 440	400
Molybdenum	360 - 440	400
Nickel	360 - 440	400
Potassium	4500 - 5500	5000
Selenium	360 - 440	400
Silicon	360 - 440	400
Silver	360 - 440	400
Sodium	9000 - 11000	10000
Strontium	360 - 440	400
Thallium	360 - 440	400
Tin	360 - 440	400
Titanium	360 - 440	400
Vanadium	360 - 440	400
Zinc	360 - 440	400

Acceptance Criteria
CCV

<u>Element</u>	<u>Control Limits (ppb)</u>	<u>True Value (ppb)</u>
Aluminum	45,000 – 55,000	50,000
Antimony	450 – 550	500
Arsenic	450 – 550	500
Barium	450-550	500
Beryllium	45 – 55	50
Boron	450 – 550	500
Cadmium	450 – 550	500
Calcium	90,000 – 110,000	100,000
Chromium	450 – 550	500
Cobalt	450 – 550	500
Copper	450 – 550	500
Iron	45,000 – 55,000	50,000
Lead	450 – 550	500
Magnesium	90,000 – 110,000	100,000
Manganese	450 – 550	500
Molybdenum	450 – 550	500
Nickel	450 – 550	500
Potassium	22,500 – 27,500	25,000
Selenium	450 – 550	500
Silicon	450 – 550	500
Silver	450 – 550	500
Sodium	90,000 – 110,000	100,000
Strontium	450 – 550	500
Thallium	450 – 550	500
Tin	450 – 550	500
Titanium	450 – 550	500
Vanadium	450 – 550	500
Zinc	450 – 550	500

8.7.4 ICB/CCB

The ICB and CCB are blank solutions. The ICB must be run immediately after the ICV. A CCB must be run immediately after each CCV. The absolute value of the ICB and CCB measurements should be less than or equal to the PQL.

For DOD projects, the value of the ICB and CCB measurements should be less than one half of the PQL.

<u>Element</u>	<u>PQL (ppb)</u>
Aluminum	200
Antimony	20
Arsenic	20
Barium	5
Beryllium	2
Boron	15
Cadmium	6
Calcium	1000
Chromium	5
Cobalt	5
Copper	10
Iron	150
Lead	10
Magnesium	250
Manganese	5
Molybdenum	5
Nickel	10
Potassium	250
Selenium	20
Silicon	50
Silver	3
Sodium	2500
Strontium	5
Thallium	30
Tin	25
Titanium	25
Vanadium	10
Zinc	20

8.7.5 ICSA/ICSAB

The ICSA and ICSAB must be run at the beginning and end of each analysis run or at a minimum of twice per eight hour shift. ICSAB must be run immediately following ICSA. ICSA contains interferences. ICSAB contains analytes plus interferences. The ICSA analytes results must be within absolute value of their reporting limits. If any element is outside this limit, then the analysis is void for that element. The ICSAB measurements must be within 20% of the true values. If any element is outside this limit, then the analysis is void for that element. The problem must be corrected and the element should be reanalyzed.

For DOD projects, the value of the ICSA measurements should be less than one half of the PQL.

Acceptance Criteria
ICSA

<u>Element</u>	<u>Control Limits (ppb)</u>	<u>True Value (ppb)</u>
Aluminum	400000 – 600000	500000
Calcium	400000 – 600000	500000
Iron	160000 – 240000	200000
Magnesium	400000 – 600000	500000

Acceptance Criteria
ICSAB

<u>Element</u>	<u>Control Limits (ppb)</u>	<u>True Value (ppb)</u>
Aluminum	400000 – 600000	500000
Antimony	480 – 720	600
Arsenic	80 – 120	100
Barium	400 – 600	500
Beryllium	400 – 600	500
Boron	800 – 1200	1000
Cadmium	800 – 1200	1000
Calcium	400000 – 600000	500000
Chromium	400 – 600	500
Cobalt	400 – 600	500
Copper	400 – 600	500
Iron	160000 – 240000	200000
Lead	40 - 60	50
Magnesium	400000 – 600000	500000
Manganese	400 – 600	500
Molybdenum	800 – 1200	1000
Nickel	800 – 1200	1000
Potassium	3500 – 6500	5000
Selenium	40 - 60	50
Silver	160 – 240	200
Sodium	4000 – 6000	5000
Strontium	800 – 1200	1000
Thallium	80 – 120	100
Tin	800 – 1200	1000
Titanium	800 – 1200	1000
Vanadium	400 – 600	500
Zinc	800 – 1200	1000

8.7.6 Linear Dynamic Range (LDR)

The LDR (Linear Dynamic Range) is to verify linearity of each element. The LR must be run at least once during analysis. Any element results above the LR must be diluted and reanalyzed. Specific acceptance range $\pm 10\%$ of the true value will be used. The LR will be verified with three standards every three months.

8.7.7 PQL (LOWCHECK)

The PQL (LOWCHECK) is to verify linearity at the reporting limit. The PQL (LOWCHECK) is prepared at approximately 5 times the MDL. The PQL must be run at least once during analysis and the acceptance range of $\pm 30\%$ will be used. QSM clients, the acceptance range of $\pm 20\%$ will be used.

8.7.8 BLK

The BLK is laboratory digested blank. If any element concentration in the digested blank is above the PQL, then all the samples associated with that blank which have concentrations greater than the PQL and less than ten times the blank concentration must be re-digested and reanalyzed for that element. If any element concentration in the digested blank is less than the negative of the PQL, then all samples associated with that blank must be reanalyzed.

Prep. Blank criteria for DOD projects must meet QSM requirements: Any samples associated with a Prep. Blank that fail following criteria shall be re-digested and re-analyzed, except when the sample analysis results in a non-detect.

- if one-half the PQL is exceeded, and the concentration exceeds 1/10 of the measured concentration of any sample in the associated preparation batch or
- If the BLK concentration is greater than the 1/10 of the specified regulatory limit.
- If the concentration of common laboratory contaminants exceed the PQL

NOTE : If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifier.

8.7.9 BKS

The BKS are digested control samples. The BKS measurements must be within control limits. If any element concentration in the BKS is outside the control limits, then all the samples associated with that BKS must be re-digested and reanalyzed. The same spiking levels for the BKS are used for the MS.

8.7.10 Duplicates

One duplicate must be analyzed for each matrix type in each group of samples. If an element concentration is greater than or equal to five times the PQL, then the % RPD should be 20%.

$$\%RPD = \frac{\text{sample} - \text{dup}}{\frac{\text{sample} + \text{dup}}{2}} \times 100$$

If the duplicate falls outside the criteria, it must be noted on the raw data and written in the case narrative.

8.7.11 Matrix Spike and Matrix Spike Duplicate

The MS or MS/MSD sample analyses are designed to provide information regarding the digestion and methodology used for analysis. One matrix spike or MS/MSD samples are prepared for each matrix type in each batch of 20 samples. If the spike recovery for an element is outside $\pm 20\%$ (for QSM clients, and if the sample concentration corresponding to the spiked sample is less than four times the spike added, then it should be noted in the case narrative and in the raw data. The acceptance limits for MS/MSD is $\pm 20\%$ RPD.) For purposes of calculating the % spike recovery, sample results less than the instrument detection limit/reporting limit, should be assumed to be zero.

<u>Element</u>	<u>Spiking Levels</u>	
	<u>Aqueous (ppb)</u>	<u>Solid (ppb)</u>
Aluminum	5000	10000
Antimony	50	100
Arsenic	50	100
Barium	500	1000
Beryllium	25	50
Boron	500	1000
Cadmium	50	100
Calcium	5000	10000
Chromium	250	500
Cobalt	250	500
Copper	250	500
Iron	5000	10000
Lead	500	1000
Magnesium	5000	10000
Manganese	500	1000
Molybdenum	250	500
Nickel	250	500
Potassium	5000	10000
Selenium	50	100
Silicon	5000	10000
Silver	50	100
Sodium	5000	10000
Strontium	500	1000
Thallium	50	100

Tin	250	500
Titanium	1000	2000
Vanadium	250	500
Zinc	500	1000

NOTE: The DUP/SPIKE or MS/MSD requirements are subject to change to suit various client requirements.

8.7.12 Post Digestion Spike

A post digestion spike is analyzed for each batch of digested samples. The post spike concentration should be at 10-100 times the MDL. The post digestion spike can be analyzed on a diluted sample if the sample requires dilution. The criterion for post digestion spike is 75-125%. The criterion is not applicable when the spike addition is insignificant. (i.e. sample concentration is greater than 4x of the spike addition)

8.7.13 Serial Dilution

Transfer 2mLs of sample to 8mLs of flame water. One serial dilution is prepared for one sample of each matrix type in each group of samples. If the element concentration is fifty times the reporting limit or greater, then the % difference between the serial dilution and the sample should be 10%.

$$\% \text{ Difference} = \frac{\text{sample} - \text{dilution}}{\text{sample}} \times 100$$

8.7.14 Internal Standard

The internal standard should be approximately 40ppm. When internal standard is outside control limits of 50% the analysis will be repeated. The problem must be corrected and all samples reanalyzed.

8.7.15 Interelement Correction factor determination (IEC)

OVERVIEW:

Inter-element interference's are false spectral signals arising from other elements in the sample besides the analyte. By measuring the apparent false signal of interfering elements at known concentrations, corrections factors can be determined and applied to unknown samples. These are determined every six months or when routine interference check samples reveal the need for further adjustments.

PROCEDURE:

Set up, profile, and calibrate the ICP. Introduce clean single element standards at the linear range concentrations. Determine the correction factor using the following equation:

$$(eq. 1) \quad k_1 = \frac{\text{apparent concentration of analyte}}{\text{known concentration of interferent}}$$

IEC's are then verified by introducing solutions of the interferences to determine the effectiveness of the IECs at canceling the apparent concentrations. IEC's are entered into the software controlling the ICP for application on unknown samples.

REPORTING:

After all inter-element correction factors have been determined IEC's are entered onto CLP forms 11A and 11B.

8.7.17 Hardness by Calculation

The preferred method for determining hardness is to calculate the results of separate determinations of calcium and magnesium.

$$\text{Hardness, mg equivalent CaCO}_3/\text{L} = 2.497 [\text{Ca, mg/L}] + 4.118 [\text{Mg, mg/L}]$$

8.8 Instrument Shut Down

8.8.1 Aspirate flame water for several minutes.

8.8.2 Under Setup menu, select F7 (shutdown). This shuts off the torch and pump windings, goes through a cool down period of 90 seconds before shutting off the water recirculator and gas flows.

8.8.3 After the peristaltic pump stops, disengage the top two cartridges, leaving the tension setting untouched. The bottom cartridge supplies tension to the rinse reservoir and should be kept on to prevent back flowing of the rinse water.

8.8.4 Leave the circuit breaker on the RF power unit behind the instrument on at all times.

8.8.5 Leave the nitrogen purge gas on at all times.

8.9 Trouble shooting and corrective action

8.9.1 Problem: Stable plasma will not start.

Action: Make sure the pump tubing is clamped down and that there are no leaks in the tubing or spray chamber. Make sure the drain line is submerged under 6 inches of water in the drain waste vessel. Press reset on instrument, and there is

- 8.9.2 Problem: Profile peak position greater than 0.3 units from zero position.
- Action: ICP may not have reached purge or thermal equilibrium. Check the nitrogen purge gas flow, replace nitrogen dewar if empty or low pressure prevents sufficient purging. If purge is sufficient, try re-profiling and recalculate spectrum shifter position. Set new vernier position and verify profile. Record new vernier position in daily maintenance log.
- 8.9.3 Problem: ICV fails for an element.
- Action: (a) The instrument will need to be re-calibrated. (b) The profile may have drifted beyond 0.3 units from zero position. (c) The sample introduction system may have deteriorated since calibration indicating the nebulizer tip, pump windings, etc. may need cleaning or replacement. (d) The internal standard may have run out. (e) the ICV solution or sample introduction system may have been contaminated, perform additional rinse, refill ICV/CCV, and rerun to verify. (f) The calibration standards may need to be remade. (g) IEC's have changed.
- 8.9.4 Problem: ICB fails for an element.
- Action: (a) The instrument will need to be re-calibrated. (b) The profile may have drifted beyond 0.3 units from zero position. (c) The sample introduction system may have deteriorated since calibration indicating the nebulizer tip, pump windings, etc. may need cleaning or replacement. (d) The internal standard may have run out. (e) the ICB solution or sample introduction system may have been contaminated, perform additional rinse, refill ICB/CCB, and rerun to verify. (f) The calibration standards may need to be remade. (g) The torch is dirty and will need to be cleaned.
- 8.9.5 Problem: ICSA or ICSAB fails for an element.
- Action: (a) The spectrum shifter may need to be re-profiled, (b) the Interelement correction files may need to be changed or (c) new background points may need to be selected and new IEC performed.

9.0 SAFETY

9.1 Equipment

- Lab coat
- Safety glasses
- Gloves

9.2 Potential hazards

9.2.1 All samples and solutions are maintained 5% HCl and may contain high concentrations of metals as well. Safety glasses should be worn to protect the eyes from acid splashes. Gloves and lab coats should be worn to protect the hands and skin from spills or splatter. If any solution comes in contact with the skin, wash the area immediately with plenty of water and notify a supervisor. If any solution is splashed in the eyes, flush immediately and thoroughly in an eyewash basin and contact a supervisor immediately.

9.2.2 The ICP uses high voltage electricity and generates an RF field, so there is a potential risk of electrocution if shielding is circumvented. The TJA ICP has numerous safety interlocks to shut off power to the RF coils if there is a break in the shielding around the coils, if the supply of cooling water is lost, or if there is a loss of argon pressure.

10.0 WASTE MANAGEMENT

10.1 After analysis sample digestates must be held for six months, after which they are disposed of in accordance with the Hazardous Waste Disposal Procedure. For procedure and methods used for disposal refer to Standard Operating Procedures D.1 and D.2.

10.2 All other non-hazardous solutions may be washed down the drain with copious amounts of water.

11.0 POLLUTION PREVENTION

11.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

12.0 REPORTING REQUIREMENTS

12.1 The ICP run log must be filled out for each day's operations. The method, date & time of start and end of each run, standard sequence and sources, QC sequence and sources, and sequence of samples analyzed and date for preparation batch. In addition, each run must be recorded according to work orders, fraction numbers, corresponding elements completed, and analyst's initials.

12.2 The results reported on the raw data are in units of ug/L. To convert raw results of soil (solid) samples, multiply by the samples final volume (1 L) divided by the weight in grams. Then correct the results for solids if required. The final units are mg/kg.

Example for solid sample

$$\frac{1 \text{ ug/L raw result} \times 1\text{L}}{1.0 \text{ g sample}} = 0.1 \frac{\text{ug}}{\text{g}} = \frac{\text{mg}}{\text{Kg}}$$

- 12.3 If any maintenance is performed, routine or non-routine, the Maintenance log for the affected ICP shall be filled out if service people conduct maintenance on the instrument. The field service report is filed in a binder.
- 12.4 All solutions made must be entered in Solutions manager program and a print out is kept in a notebook in the laboratory.
- 12.5 Include copy of digestion log with weights and final volume for reviewer.
- 12.6 Include short narrative on why the run was needed, what problems were encountered, what actions were taken to correct them, and any future actions that will be needed.

13.0 METHOD PERFORMANCE

- 13.1 Per digestion methods MDL limits are obtained by digestion seven spiked replicates the same way as samples and analyzing them. MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. Precision and accuracy studies are performed once a year at a minimum.

14.0 METHOD DETECTION LIMIT

- 14.1 Method detection limits for this method are listed in the GPL Laboratory Method Detection Limit and Reporting Limit table.

15.0 DEFINITIONS

- 15.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

SOP No: H.12

Title: Cold Vapor Analysis for Mercury in Accordance with SW846 Methods 7470A and 7471B.

1.0 SCOPE AND APPLICATION

1.1 The method detailed in this procedure explains the analysis of mercury in water, soil, sediment and TCLP extracts, by the Manual Cold Vapor Technique in accordance with SW846 methods 7470A and 7471B.

2.0 METHOD SUMMARY

2.1 To quantify both organic and inorganic forms of mercury in water, TCLP extracts, soil and sediment. The sample holding time is 28 days from sample collection. Sample digests containing mercury concentrations greater than the highest calibration standard will be diluted.

3.0 SAFETY

3.1 Gloves, safety glasses, and lab coat must be worn when performing any aspect of the mercury digestion or analysis process.

3.2 Mercury vapor is toxic. Caution should be taken during all phases of the digestion and analytical process. Care should be exercised so that mercury has no chance to be absorbed through the skin or inhaled.

3.3 The acids used to digest mercury are used full strength. Extreme care should be taken so that acid does not spill or splash. Transport samples and empty bottles on one of the 3 high concentration room spill carts. Transport acid using the 4 liter plastic carrying buckets.

3.4 Additional safety equipment includes mercury sponges, acid neutralization media, apron, a mercury scrubber, and face shield.

4.0 INTERFERENCES

- 4.1 Contaminants in the solvents, reagents, glassware and other sample processing hardware. These contaminants lead to discrete artifacts or to elevate baseline in gas chromatograms. All of these materials routinely must be demonstrated by running laboratory method blanks. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of the interferences will vary from source to source.

5.0 EQUIPMENT AND SUPPLIES

5.1 Mercury Digestion Equipment

- 300mL BOD bottles with glass stoppers
- hot plate
- hot water bath
- VWR Calibra M23
- VWR Calibra M27
- 100mL graduated cylinders
- 100mL volumetric flask (Class A)
- 200mL volumetric flask (Class A)
- balance capable of accurately measuring 0.200 grams ($\pm .001g$)
- squirt bottle filled with deionized water
- Parafilm squares for mixing standards in volumetric flasks
- thermometer traceable to NIST, alcohol based.
- Bottle top dispensers
- Fisher brand Finnpipette M9
- three 125mL plastic bottles with caps for ICV sub, ICV and Hg sub
- one 250mL plastic bottle with cap fo Hg SPIKE

5.2 Leeman Labs Automated Mercury Analyzer, Model HYDRA AA

5.3 Air source capable of delivering 1 liter of air per minute, argon source.

6.0 REAGENTS AND STANDARDS

- Concentrated Nitric Acid, Trace Metals Grade.
- Concentrated Sulfuric Acid, Trace Metals Grade.
- Concentrated Hydrochloric Acid, Trace metals Grade.
- Potassium Permanganate solution, 5% w/v. Dissolve 200g Potassium Permanganate to a final volume of 4L of deionized water.
- Potassium Persulfate solution, 5% w/v. Dissolve 100g Potassium Persulfate to a final volume of 2L of deionized water.

- Stock Mercury solution, 100µg/mL, traceable to NBS standard reference materials. (Source: Absolute Standards, Hamden, CT).
- Stannous Chloride (10%). Dissolve 200g of Stannous Chloride to 2L of 10% HCl. [Leeman requires the use of 10% Stannous Chloride in 10% HCl, DO NOT SUBSTITUTE Stannous Sulfate or Sulfuric Acid as described in the EPA method.]
- 10% Hydrochloric Acid. To a 20L HDPE carboy, add 10L deionized water, then add 2000mL of concentrated Hydrochloric Acid. Add deionized water to 20L final volume, cap container and mix. Record initials, date of preparation, and lot number of acid on side of carboy.
- Deionized water ASTM Type II or equivalent.
- Sodium chloride - Hydroxylamine Hydrochloride solution - Dissolve 240g of Sodium Chloride and 240g of Hydroxylamine Hydrochloride in deionized water and dilute to 2L (Hydroxylamine Sulfate may be used in place of Hydroxylamine Hydrochloride).
- ICV Solution(second source), 100µg/mL, traceable to NBS standard reference materials (Source: CPI International)
- 50% Aqua Regia digestion solution. To a 2.5 L Glass bottle, add 750mL DI water. Slowly add 750mL of concentrated Hydrochloric Acid, then add 250mL of DI water, followed by 250mL of concentrated Nitric Acid and mix well. Pour 50% Aqua Regia into plastic side bottle with bottle-top dispenser for use.

7.0 SAMPLE COLLECTION AND PRESERVATION

- 7.1 The holding time for mercury is 28 days.
- 7.2 Samples may be collected in either plastic or glass containers for aqueous samples, at least 500mL should be collected. For soil/solid samples, at least 5grams should be collected.
- 7.3 All samples should be stored at 4°C ± 2°C.

8.0 CALIBRATION AND STANDARDIZATION

- 8.1 2.0ppm Hg intermediate stock in 2% HNO₃ - Pipette 2.0mL of 100ppm Hg stock standard into a 100mL volumetric flask. Add 2% HNO₃ to a final volume of 100mL.
 - 8.2 0.1ppm (100ppb) Hg working solution in 2% HNO₃ - Pipette 10mL of 2.0ppm Hg intermediate stock standard into a 200mL volumetric flask. Add 2% HNO₃ to final volume of 200mL.
 - 8.3 1.0ppm Hg ICV intermediate solution in 2%HNO₃ - Pipette 1.0mL of 100ppm Hg stock standard into a 100mL volumetric flask. Add 2% HNO₃ to a final volume of 100mL.
- 0.1ppm Hg ICV working solution in 2% HNO₃ (see 8.5 and 8.10)

Standards for Water Samples and sample QC.

- 8.4 First add 100mls of DI water to six BOD bottles. Then with a pipette remove the appropriate amount of DI water then replace with equal amount of 0.1ppm working standard directly into the BOD bottle using the following volumes 0, 0.2, 0.5, 1.0, 5.0 and 10.0mL for calibration. The 0 standard is used for calibration, the ICB and CCB. The 0.2ppb standard is used for calibration and the reporting limit. The 5.0ppb standard is used for calibration and the continuing calibration verification standard. All other for mentioned standards are used for calibration only.
- 8.5 ICV (3.0ppb) – To BOD bottle, add 100mL of DI water using a graduated cylinder. Using a Pipette remove 3.0mL of DI water then add 3.0mL of Hg ICV working solution (0.1 ppm) into BOD bottle.
- 8.6 Preparation Blank (BLK) - Add 100mL DI water using graduated cylinder into BOD bottle.
- 8.7 Laboratory Control Sample (BKS) - Add 100mL DI water using graduated cylinder to BOD bottle. Using a pipette remove 1.0mL of DI water then Spike with 1.0mL of 0.1ppm working standard.
- 8.8 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) (1.0ppb) - For both MS and MSD samples, add 100mL of the selected QC sample using a graduated cylinder to a BOD bottle. Using a pipette, remove 1.0mL of sample. Next, Spike the sample with 1.0mL of 0.1ppm working standard. For TCLP samples, remove 11.0 mL of DI water, then add 10mL of sample, and then 1.0mL of 0.1ppm working standard.

Standards for Soil Samples and sample QC.

- 8.9 First add 10mL of DI water to four BOD bottles then remove with a pipette the following volumes of DI water 0, 0.2, 0.5 and 1.0 (ml), then replace with equal amounts of 0.1ppm working standard. For 5.0 standard add 5mL DI water then 5mL of 0.1ppm standard. For standard 10.0 add 10mL of working standard only. The 0 standard is used for calibration, the ICB and CCB. The 0.2ppb standard is used for calibration and the reporting limit. The 5.0ppb standard is used for calibration and the continuing calibration verification standard. All other for mentioned standards are used for calibration only.
- 8.10 ICV (3.0ppb) – To a BOD bottle pipette 3.0mL of 0.1ppm ICV working solution and 7.0mL of DI water.
- 8.11 Preparation Blank -(BLK) Pipette 10.0mL of DI water.
- 8.12 Laboratory Control Sample (BKS) - Pipette 7.0mls of DI water and Spike it with 3.0mL of 0.1ppm working standard.
- 8.13 Matrix spike (MS) and matrix spike duplicate (MSD) (3.0ppb)-For both MS and MSD samples, pipette 7mL of DI water to the BOD bottle. Using a pipette Spike the sample with 3.0mL of 0.1ppm working standard.

9.0 METHOD DETECTION LIMIT

- 9.1 Method detection limits for this method are listed in the GPL Laboratory Method Detection Limit and Reporting Limit table.

10.0 METHOD PERFORMANCE

- 10.1 The MDL concentrations listed in the GPL MDL table are generally obtained using organic-free reagent water. Results were also obtained by extracting seven spiked replicates the same way as the samples and analyzing them. MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. Precision and accuracy studies are performed once a year at a minimum. Single operator precision, overall precision and method accuracy were found to be directly related to the concentration of the parameter.

11.0 PROCEDURE (See Figure 1 for flow chart)

11.1 Preliminary Preparations.

- 11.1.1 Read workorders and test sheets for assigned samples, noting which samples are selected for duplicate and spike analysis. Review special instructions and submit sample request form to sample receiving for assigned samples.
- 11.1.2 Obtain samples from sample receiving and verify the identity of each sample. Record in the Mercury Digestion Log Form the date of digestion, analyst initials, and make an entry for each sample assigned (workorder-sample-fraction) using one line for each sample, standard, quality control check standard, blank, duplicate, spiked sample, and laboratory control sample.
- 11.1.3 Prepare BOD bottles for each entry by writing with permanent ink on the outside of the bottle the workorder number, the sample number, and fraction, using "MD", "MS" and "MSD" for Matrix Duplicate, Matrix Spike and Matrix Spike Duplicate.

11.2 Sample Digestion for Waters/TCLP Extracts and Water Standards

- 11.2.1 In the metals digestion lab measure 100.0mL of water or leachate sample into a clean, 100mL graduated cylinder and transfer sample into a clean, labeled 300mL BOD bottle. Stopper the BOD bottle, replace onto spill cart. For TCLP samples, measure out 90mL of DI water in a clean graduated cylinder and transfer that into a clean BOD bottle. Next, shake the closed TCLP bottle, and then pipette 10mL of the TCLP sample into the BOD bottle. Stopper the BOD bottles which hold the TCLP samples as well.
- 11.2.2 In the hood, to each bottle, add 5.0mL of concentrated Sulfuric Acid, 2.5mL of concentrated Nitric Acid, and 15mL of 5% Potassium Permanganate solution. Mix sample after each addition. After the first 3 reagents have been added let the samples sit for 15 minutes. If a sample

does not retain a purple color after sitting for 15 minutes, add additional Potassium Permanganate in 15mL increments until the sample is able to sit for 15 minutes without losing color. Do not add more than 32mL of Potassium Permanganate to any sample. Record the final amount of permanganate added into the digestion log.

Note: If additional permanganate is added, the final volume must be changed accordingly on the bench sheet for correct final calculation of results.

11.2.3 Add 8mL of 5% Potassium Persulfate solution to each sample mix

11.2.4 Make sure to cover the water bath with the heat retention plastic balls. Once all required reagents are added, place the BOD bottles gently into the water bath. Depending upon how low the temperature drops by the addition of the BOD bottles depends upon what the new set point should be changed to in order to heat the bottles to the 92°C-98°C range. The analyst should make these judgment calls each day for each digestion. Make sure that the temperature probe is not touching the bottom of the water bath, yet is still below the water line for an accurate reading. Temperature should be checked with a NIST traceable thermometer. Heat at temperature for 2 hours.

11.2.5 Remove samples from water bath and allow samples to cool to room temperature. Add 6mL of Sodium Chloride Hydroxylamine Hydrochloride solution and swirl bottle to mix so that all traces of permanganate will be reduced. [Caution: the addition of this solution produces an effervescence which could cause back-pressure build up and force out the glass stopper. After adding the hydroxylamine to the sample, let the pressure vent off by removing the glass stopper periodically.]

11.2.6 Samples are ready for analysis.

11.3 Sample Digestion for Soils/Sediments and Soil Standards

11.3.1 Use an analytical balance to measure a representative 0.600-1.00g, or three separate scoops of approximately 0.2 grams each of soil, or sludge, Laboratory Control Sample, solid or sediment sample into a 300ml BOD bottle. Record measurement to 3 significant figures. Stopper the BOD bottle. When all samples have been measured, return the unused portion of sample to sample receiving area. Set MS and MSD bottles aside for spiking.

11.3.2 In the hood, add 10mL 50% Aqua Regia digestion solution to each BOD bottle. Add 10mL of distilled water to each BOD bottle in order to rinse any remaining sample residue and to cover the entire sample. Add the correct amount of water for the day's digestion before hand. Make sure to cover the water bath with the heat retention plastic balls. Once all required reagents are added, place the BOD bottles gently into the water bath. Depending upon how low the temperature drops by the addition of the BOD bottles depends upon what the new set point should be changed to in order to heat the bottles to the 92°C - 98°C range. The analyst should make

these judgment calls each day for each digestion. Make sure that the temperature probe is not touching the bottom of the water bath, yet is still below the water line for an accurate reading. Temperature should be checked with a NIST traceable thermometer. Heat at temperature for 2 minutes.

11.3.3 Remove bottles from bath and allow to cool. Add 15mL of Potassium Permanganate then 40mL of distilled water to each bottle. Make sure to cover the water bath with the heat retention plastic balls. Once all required reagents are added, place the BOD bottles gently into the water bath. Depending upon how low the temperature drops by the addition of the BOD bottles depends upon what the new set point should be changed to in order to heat the bottles to the 92°C - 98°C range. The analyst should make these judgment calls each day for each digestion. Make sure that the temperature probe is not touching the bottom of the water bath, yet is still below the water line for an accurate reading. Temperature should be checked with a NIST traceable thermometer. Heat at temperature for 30 minutes.

11.3.4 Remove bottles and allow samples to cool to room temperature. Add 50.0mL DI water. Add 6mL of Sodium Chloride Hydroxylamine Hydrochloride solution to reduce the excess permanganate.

11.4 Running, Tuning and Calibration of Instrument HYDRA AA

Soil, tissue and thick non-aqueous liquid samples can share standards. Water, Extracts and thin Non-Aqueous Liquid samples can share standards. These two groups cannot share standards and must be analyzed separate from each other.

11.4.1 Maintenance Check: pump tubing (for worn down areas), reductant level, waste vessel (waste drain tubing should never be below the surface of the liquid waste), rinse level (rinse bottle should be filled before software is started), dry the little piece of tubing leading from the Liquid/Gas Separator to the Dehydrator (use compressed nitrogen), look for any blockage in all of the tubing (usually found where one piece of tubing connects with another), make sure that the printer has enough paper for the run

11.4.2 Double click on the "WinHg" icon on the desktop, this will open th WinHg Runner. Select the "Protocol" and "Dataset" to be used for the run. Click on the "Database" icon and also the "Rack Editor" icon.

11.4.3 In the WinHg Database, click on the "Cal Curve" tab. The click on the "Calib Coeffs" and "New Cal" buttons to clear the graph and allow for a new set of standards to be used for the run.

11.4.4 In the WinHg Rack Editor, enter cup identification information in both the "sample ID" and "extended ID" columns. In the fifth column, enter the codes for checking the ICV, ICB, CRA, CCV, and CCB. C2 for the ICV, C3 for the ICB, C4 for the CRA, C5 for the CCV, and C6 for the CCB. In the row for the first cup in the fifth column, place CP after the check codes. (Example: C2 C3 C4 C5 C6 CP) Every 10th sample should have a CCV and CCB

action code in its fifth column. (Example: C5 C6) The fifth column for the last sample should also contain the action codes for the CCV and the CCB. If a sample is diluted by any amount, make a mark in the extended ID column attached to the end of the extended ID. (Example: 01-001-1/1:10 for a times 10 dilution.) Save the rack as the year first, then month, then date. (Example: 051117 for November 17th, 2005) If there is more than one rack prepared in a day, save them with letters descending from A. (Example: 051117A and 051117B for racks saved after the first one on November 17th, 2005)

- 11.4.5 Click on the WinHg Runner window. Click on the "Main" tab. Under the Printer Control panel, check to make sure that the Next Page number is at 1 and that the Lines Waiting number is at 0. If there are no Lines Waiting and the Next Page number is not 1, click on the "Reset" button next to it. If there are Lines Waiting, click on the "Form feed" button. This information might be needed for a past run. If this is the case, click on the "Reset" button once it is done printing. Lines Per Page should be set no higher than 63 and no less than 50.
- 11.4.6 Click on the "Standard" tab. Click the buttons for the standards and triplicate repetitions. S1 for the 0.0 standard, S2 for the 0.2 standard, S3 for the 0.5 standard, S4 for the 1.0 standard, S5 for the 5.0 standard, S6 for the 10.0 standard. Rep1, Rep2 and Rep3 for repetition 1, 2 and 3. Load the standards rack into the correct slot with all the standards in it.
- 11.4.7 Click on the "Sample" tab. Click on the "Start New Batch" button and click okay when prompted. No Batch ID is required. Then click on the WinHg Database window. Click on the "Report" tab. Make sure that only the new Batch is selected in the Batch List panel. Under the Record panel, there should only be a number under the "Seq" column and under the "Rec" column next to it should be BAT.
- 11.4.8 Return to the WinHg Runner window. In the Autosampler Run panel, select the correct Rack to be used (Example: 051117), then which samples in that Rack by selecting the first cup in the Rack to be tested under Start Cup (Example: 1) and the last cup in the Rack to be tested under End Cup (Example: 25). The Cups Per Rack should be 44.
- 11.4.9 Click on the "Control" tab. Click on in the Hg Lamp panel to start warming up the instrument's mercury lamp. After the lamp has warmed up sufficiently, attach rinse and reductant tubing to appropriate bottles and put the pump cassettes on. Turn on both the Gas and Pump by clicking the appropriate "On" buttons. Once the liquids have passed through the instrument for about one minute, click the "Adjust" button. This will take about two minute to complete. When prompted, click "Okay" and then click on the "Utility" tab. Under the Action menu, select DAQ Test and then click the "Do Action" button. Record the reading it gives under Results for the Sample and the Hg Lamp Volt. The Sample reading should be between 700,000 and 1,720,000. The Hg Lamp Volt reading should be between 5.000 and 9.500 V.

- 11.4.10 Next, go back to the "Control" tab and once again turn "On" the Gas and Pump. Next, move the tubing from the small 10% HCl bottle and place it into the 10% Stannous Chloride bottle. Let the instrument run like this for no less than 1 minute and no more than 5 minutes.
- 11.4.11 Click on the "Standard" tab. Click on the "Std Auto" button. This will run standards 1-6 in triplicate if you have prepared properly. Make sure that the WinHg Database is on the "Cal Curve" tab screen. The allowed %RSD for the 2nd standard is 20%. The allowed %RSD for the 3rd standard is 10%. Standards 4 through 6 have a %RSD maximum of 5%. If Rho is greater than 0.998000, the deviation of the blank standard is within 0.1 ug/L and the other standards are within 5% of their true values, click the "Accept" button and then the "Print" icon. The 2nd standard is exempt from the 5% variance. All of these requirements must be met for the curve to be acceptable to use.
- 11.4.12 Go back to the WinHg Runner window. Click on the "Sample" tab. Click on the "Run Auto" button.
- 11.4.13 When the run is over, turn off the Hg Lamp and move the tubing from the reductant bottle to the extra 10% HCl rinse bottle. If the last page has not been printed out yet, click on the "Form feed" button in the "Main" tab. Then click the "Reset" button after the last page has printed in the Printer Control panel. Click on the "Report" tab in the WinHg Database window. Click on the "Generate Report" button. Under Format, select PRN file. Click the "..." button for where to save the PRN file. Save the file on the T drive via the Metals/hg pathway. Save file as Hg and date. (Example: HG051117 for November 17th, 2005) If multiple files are needed in one day name them in the same method that multiple racks are saved in. Once the file is saved, click the "Generate" button. The "Total Lines Generated" should be one more than the number under "Included In Report" in the WinHg Database window.
- 11.4.14 Allow the Pump and Gas to run for several minutes to clean the instrument's tubing and sterilize it. After a reasonable amount of time, remove the tubing from the spare 10% HCl bottle and the Rinse bottle. Let the instrument draw in air for about 5 minutes, then turn off the Gas and Pump. Remove the pump cassettes. Close all windows to shut off program.

Trouble Shooting Guide. Refer to Leeman Labs Manual.

12.0 DATA ANALYSIS AND CALCULATION

- 12.1 The linear regression analysis is being used to generate the initial calculation line. The equation used in calculating the curve is: $y = mx + b$
- 12.2 The linear regression analysis is being used to generate the initial calculation line.

- 12.3 The results reported on the raw data are in units of ug/L. To convert raw results of soil (solid) samples, multiply by the samples final volume (0.1L) divided by the weight in grams. Then correct the results of solids if required. The final units are mg/kg.

Example for solid sample:

$$\frac{1 \text{ ug/L raw result} \times 0.1\text{L}}{.6\text{g}} = 1.67 \frac{\text{ug}}{\text{g}} = \frac{\text{mg}}{\text{kg}}$$

- 12.4 In addition to the accurate reporting and handling of raw data, there are records that need to be maintained for internal auditing purposes.

12.4.1 Before analysis is completed, record on the raw data the date of analysis, the analyst, the Mercury standard's source, lot number, and GPL ID, the ICV's number, and GPL ID, the LCSS's number and GPL ID, a list of the cases included in the run, and the instrument upon which the analysis was performed. Note the source for the spike used in the matrix spike sample and % Recovery for the spike in the raw data.

12.4.2 Complete the Bench Sheet for Mercury with the following information: Instrument ID, Date of analysis, Analyst name and signature, Time analysis was initiated, case and SDG, work order numbers, sample sequence run, dilution factors and comments.

12.4.3 Complete the Instrument Use Log with the following information: Date, Method, Case, Analyst and Comments.

12.4.4 If maintenance or repair was required on the instrument, all relevant information must be recorded in the Maintenance Log. This Log will be maintained with all field service reports for each instrument.

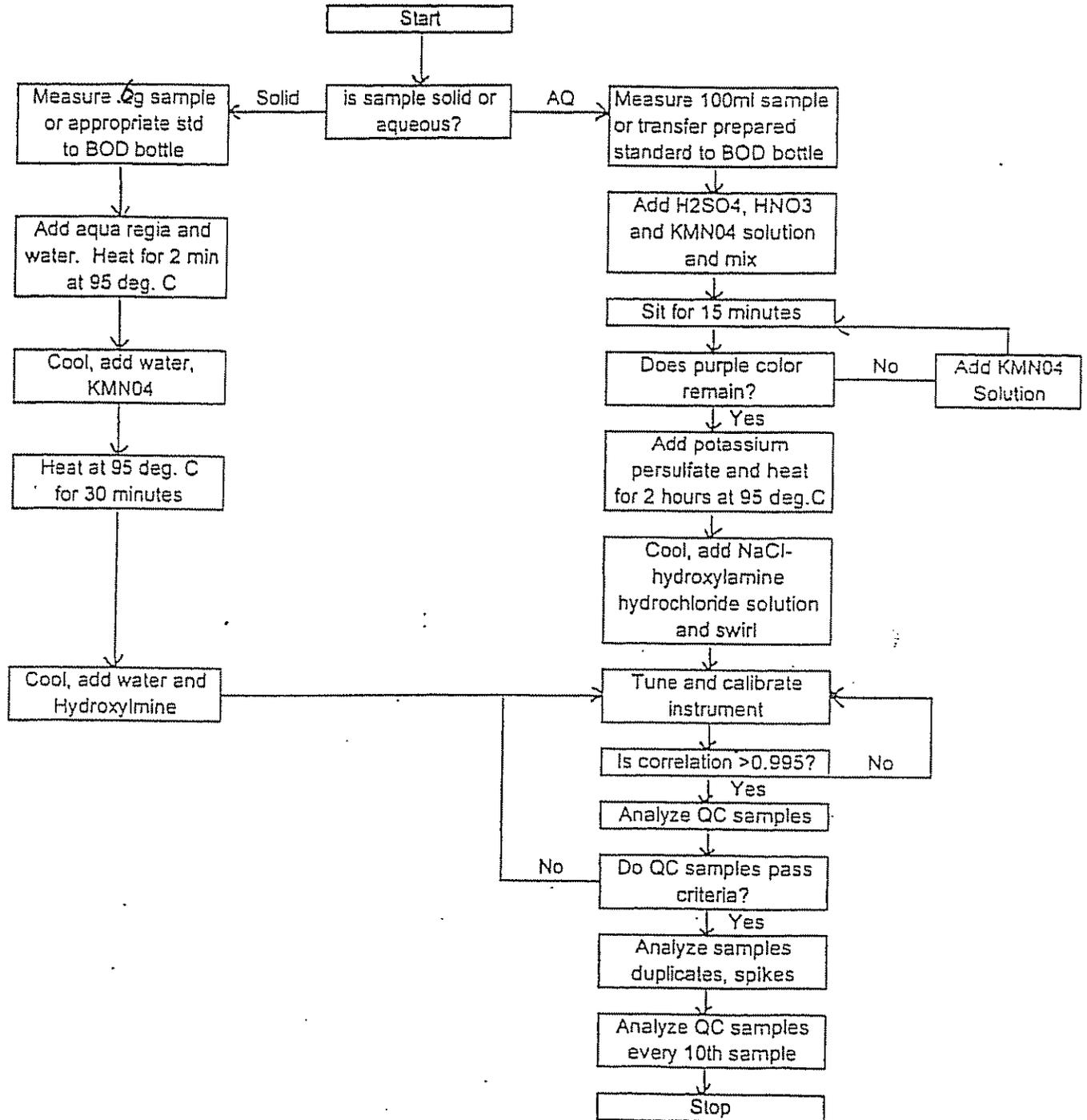
13.0 QUALITY CONTROL

- 13.1 Any results that exceed the highest calibration standard are diluted and reanalyzed. (LR = highest calibration standard)
- 13.2 The mercury auto-analyzer is capable of detecting parts per trillion levels of mercury when optimized. However, the laboratory reports lowest standard concentration level.
- 13.3 Calibration standards must be prepared fresh daily from stock standards. The deviation of each standard must be within 5% of the standard's concentration or the run must be terminated and the instrument re-calibrated. The 5% criteria does not apply to calibration standard of the CRQL 0.2ppb. The correlation coefficient must be $\geq .995$ or the run must be terminated and the instrument re-calibrated.
- 13.4 Initial Calibration Verification Check solution. An ICV solution is prepared from an independent standard as the calibration standards are prepared. This solution is then digested with the samples and is the 1st check standard to be run. If recovery

is outside 90-110% window, the run terminates. Recalibrate and rerun the ICV and ICB.

- 13.5 Continuing Calibration Check solution. The middle standard is used as the CCV solution. Window of acceptance for CCV is 80-120%. The CCV is run once every 10 samples.
 - 13.6 Initial and Continuing Blank Solution. A blank solution, separate from the calibration blank, is digested with the standards and analyzed at after each ICV and CCV. Criteria is absolute value of +/- 0.2ug/L. The CCB is run after the CCV.
 - 13.7 PQL solution. A solution of 0.2ug/L Hg is prepared as calibration standards are being prepared and is analyzed at the beginning of the run following the ICB.
 - 13.8 Preparation Blanks are digested exactly as the samples. The same type of container, utilization of the same amount of reagents, and are assigned to the same digestion batch. The blanks are placed in sequence every twenty samples. Criterion is absolute value of +/- 0.2ug/L. For DOD project, the criterion is one half of the Reporting limit.
 - 13.9 Laboratory Control sample. A blank solution is spiked at 1.0ug/L to check the spiking procedure and the digestion process. Acceptance criteria is 80 –120%.
 - 13.10 Spike Sample. 0.6 – 1.0g of sample is spiked with 1.0mL of 0.1ppm Hg standard for water, 3.0mL for soil before addition of reagents or digestion. One spike is prepared every 20 samples of the same matrix. If the matrix spike is outside 75 -125% (80 – 120% for QSM clients) criteria, a post digestion spike must be analyzed (sec 13.11). It also must be noted in the case narrative.
 - 13.11 Post digestion spike. This is performed if the matrix spike is outside the criteria. The post digestion spike should fall within the midrange of the calibration curve and should be within 75-125%.
 - 13.12 Duplicate. One duplicate every twenty samples will be performed. The duplicate is prepared before addition of any reagents or digestion.
- 14.0 POLLUTION PREVENTION
- 14.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.
- 15.0 DEFINITIONS
- 15.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.
- 16.0 REFERENCES
- SW846 method 7470A and 7471A.
 - Leeman Labs HYDRA AA Manual, Leeman Labs, Inc., Lowell, MA

Figure 1
Procedure Flow Chart



SOP No: H.21

Title: Acid Digestion of Soil, Sludge, Sediment, and Other Solid Waste Samples for ICP and ICPMS by SW846 Method 3050B.

Scope: The method detailed in this procedure is performed to prepare soil, sediment, sludge and other solid samples for quantitation of certain metallic analytes using Inductively Coupled Plasma (ICP) and ICPMS, in accordance with SW846 method 3050B.

1.0 PURPOSE

1.1 The method detailed in this procedure is used to prepare solid waste samples for analysis using an Inductively Coupled Plasma (ICP) and (ICPMS). The sample holding time before extraction is 180 days. Samples must be stored in refrigerator at 4°C until time of preparation. The elements to be analyzed using this procedure are:

ICP Elements

Ag	K
Al	Na
As	Mg
Ba	Mn
Be	Ni
Ca	Pb
Cd	Sb
Co	Se
Cr	Tl
Cu	V
Fe	Zn

This method is also applicable to other metals (B, Mo, Sr, Sn, Ti, Te, Zr).

2.0 REFERENCES

- SW846 method 3050B revision 2, December 1996.

3.0 EQUIPMENT AND SUPPLIES

- 150ml beakers/or hot block vessels
- Analytical balance accurate to 0.001 grams
- 100ml volumetric flasks (Class A)
- Hot plate, or hot block capable of maintaining temp. of 90-95 degrees C.
- Whatman No. 41 filter paper
- 125ml sample bottle (plastic)
- Watch glass, ribbed
- Plastic disposable funnels
- Thermometer, calibrated, NIST traceable
- Fume hood
- Pipettors (calibrated) VWR Calibra M23 & M24
- Bottle top dispensers used to add all reagents
- Teflon coated spatula

4.0 REAGENTS

- Concentrated Nitric Acid - trace metals grade
- 1:1 Nitric Acid - trace metals grade
- Concentrated Hydrochloric Acid - trace metals grade
- Metals Standards - (ICP SPK 1,2,3) commercially prepared NBS traceable metals standards with documented concentrations, including impurities and expiration dates. (Vendor: High Purity Standards, Charleston, SC). The spiking solutions cannot be used past the expiration date located on the label.
- 30% Hydrogen Peroxide
- Grade and quality of water required is ASTM Type II water (ASTM D1193): Water must be monitored for changes in conductivity by laboratory staff and is currently provided by a laboratory pure water system.

5.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 5.1 All sample containers must be collected in glass or polyethylene containers.
- 5.2 All samples must be iced or refrigerated at 4 degree C (+/- 2) from the time of collection until digestion. The maximum holding time for metals is 180 day.

6.0 PROCEDURE

6.1 Sample Digestion Procedure

- 6.1.1 Mix the sample thoroughly to achieve homogeneity using a spatula. For each digestion procedure, weigh (to the nearest .01g) 1-2g portion of sample and transfer to a beaker/vessel.

Note: A separate sample shall be dried for percent solids determination. See SOP "Percent Solids Determination Procedure".

- 6.1.2 Label two beakers/vessels, one as a sample and the second as a duplicate.
- 6.1.3 Label one beaker/vessel as matrix spike. To matrix spike beaker/vessel add 0.2mL each of spiking solution (ICP SPK1,2,3) after addition of 10mls of 1:1 nitric acid.
- 6.1.4 Take one beaker/vessel and label as prep blank (BLK). A purified solid matrix is used.
- 6.1.5 Take beaker/vessel and label as BKS. To BKS beaker/vessel add 0.2mL each of spiking solution (ICP SPK1,2,3) after addition of 10mls of 1:1 nitric acid. A purified solid matrix is used.
- 6.1.6 To all beakers/vessels, add 10ml of 1:1 nitric acid (HNO_3), mix the slurry, and cover with a watch glass. Heat the sample on a hot plate in the fume hood to 95°C ($\pm 5^\circ\text{C}$) and reflux for 10 minutes without boiling. Allow the sample to cool, add 5ml of concentrated HNO_3 , replace the watch glass, and reflux for 30 minutes. Repeat the concentrated HNO_3 addition until no brown fumes are given off by the sample. Maintain a covering of solution over the bottom of the beaker/vessel. Check the temperature achieved during the digestion with a beaker containing 50ml ASTM type II water. Record temperature in digestion log. Using a ribbed watch glass either allow the solution to evaporate to approximately 5mL without boiling.
- 6.1.7 After the sample has cooled, add 2ml of 30% hydrogen peroxide (H_2O_2) and cover with watch glass. Return the vessels to the block digester to warm to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides, and cool the vessels. Continue to add 2ml + 3ml of 30% H_2O_2 with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 7ml 30% H_2O_2 .) Continue heating the solution at 95°C ($\pm 5^\circ\text{C}$) for 30 minutes without boiling. Maintain a covering of solution over the bottom of the vessels at all times.

6.1.8 Add 10ml of concentrated HCl, return the covered beaker to the hot plate, and heat for an additional 15 minutes at 95°C ($\pm 5^\circ$). After cooling, filter through Whatman No. 41 filter paper (or equivalent) using disposable funnel and dilute in a 100ml volumetric flask with Type II water. The diluted sample has an approximate acid concentration of 10% (v/v) HCl and 5% (v/v) HNO₃. Transfer the sample diluted sample to a 125mL plastic sample bottle labeled with the work order, fraction, and date of digestion. Date of digestion may be put on the box of digestates instead of on each bottle. The sample is now ready for analysis.

7.0 METHOD PERFORMANCE

7.1 Per digestion method MDL limits are obtained by digestion of seven spiked replicates in the same way as samples and analyze them. MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. Precision and accuracy studies are performed once a year at a minimum.

8.0 METHOD DETECTION LIMIT

8.1 Method detection limits for this method are listed in the GPL Laboratory Method Detection Limit and Reporting Limit table.

9.0 DEFINITIONS

9.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

10.0 QUALITY CONTROL

10.1 Troubleshooting and corrective action.

10.1.1 The temperature should be maintained between 90-95°C on each hot plate / hot block. The temperature of the hot plate / hot block is documented on the bottom of the digestion log.

10.1.2 ICP operators should report to his/her supervisor and lab manager any recoveries outside warning limits for LCS samples for analytes being determined or preparation blanks are above control limits. Sample recoveries for any element which are outside of the control established limits for the laboratory control sample or contaminated preparation blanks are deemed unacceptable. The digestion batch must be re-digested for those analytes. Document the incident on a re-digestion form and submit to supervisor.

11.0 SAFETY

11.1 Safety equipment required

- Fume hood - minimum flow of 100 linear feet/minute
- Safety glasses
- Safety gloves (unpowdered)
- Lab apron
- Face shield, if necessary

11.2 Potential hazards

The most hazardous chemical acids that laboratory personnel are likely to encounter are strong acids such as Hydrochloric Acid and Nitric Acid (HNO₃). Special handling requirements.

- 11.3 Analysts should always read the label on the bottle. Chemicals require handling with care to include wearing adequate garments for skin protection. Also, acids should be handled only under a ventilated hood.

12.0 POLLUTION PREVENTION

- 12.1 GPL Laboratory operates in a safe manner to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. For more details on pollution prevention, refer to GPL SOP D.5.

13.0 DISPOSAL REQUIREMENTS

- 13.1 Acid wastes should be placed into the acid waste bottle, which is located in the metals digestion lab. Any remaining samples should be returned to sample control. More details concerning disposal characteristics and procedures can be located in the SOP "Laboratory Waste Handling and Storage Procedure".

14.0 REPORTING REQUIREMENTS

- 14.1 Documentation to include Work Orders and Metals Preparation and Sample Description log, must be submitted to the Metals Supervisor with the Digestion Technician's initials and the preparation date documented on each form for each case.

Sample Description must be filled out and should contain the following information for aqueous digestates. The fields for color and clarity, before and after digestion, must be completed. The following descriptive terms are recommended:

Color - red, blue, yellow, green, orange, violet, white, colorless, brown, gray, black

Clarity - clear, cloudy, opaque

Texture - coarse, medium, fine

Note any significant changes that occur during sample preparation (i.e., emulsion formation) in the Comments section. Enter any sample-specific comments concerning the analyte results in the comments section. If ICP analysis is required, use color of ICP digests for sample description.

Metals Preparation and Sample Description Log Forms (Figure 1) must be documented completely by the Digestion Technician during digestion and include the date of digestion, work order number, digestion technician signature, supervisor initials, identification of method used, Lab Sample ID, sample matrix (soil/water), amount of sample used in digestion and final volume of sample, identification of the matrix spiking solution used, the amount used, and identifications of any reagents used during the digestion.

UNCONTROLLED DOCUMENT
AND
BUSINESS CONFIDENTIAL

SOP No: J.4

Title: Percent Solids Determination Procedure

Scope: This Standard Operating Procedure describes the method used for the determination of percent solids in soils, sediments, and sludges

1.0 PURPOSE

- 1.1 The method detailed in this procedure is used to determine the percent solids in soils, sediments, and sludges. The percent solids data is used to convert sample results from wet weight based analytical concentrations to reportable dry weight based analytical concentrations.

2.0 REFERENCES

- USEPA SOW OLM04.3/ASTM D2216

3.0 EQUIPMENT AND SUPPLIES

- Weighing dishes
- Drying oven maintained at 103-105°C
- Dessicator(s)
- Analytical balance accurate to 0.0001g
- Or Analytical balance with serial port connection and PC

4.0 PROCEDURE (manual)

- 4.1 Determine the weight of a pre-dried (105°C) weighing dish to the nearest 0.0001g. Record the weight on the data sheet in Column labeled: Wt. of Dish (g).
- 4.2 Add 5-10g of soil, sediment, or sludge sample to the weighing dish. Determine the weight of the sample and weighing dish to the nearest 0.0001g. Record the value on the data sheet in Column labeled: Wet Sample + Dish.
- 4.3 Place the weighing dish into the drying oven, which is maintained at 103-105°C. Record the time the samples were placed into the drying oven on the data sheet.

- 4.4 Allow the sample to dry at least 12 but not more than 24 hours in the drying oven. Record the time the samples were removed from the drying oven on the data sheet.
- 4.5 If sample dried less than 12 hours, it must be demonstrated that constant weight is attained. Constant weight must be defined as a loss in weight of no greater than 0.01g between the start weight and final weight of the last cycle. Constant weight data must be recorded for a minimum of two repetitive weigh/dry/dessicate/weight cycles with a minimum of 1hour drying time in each cycle.
- 4.6 Remove the weighing dish from the oven and allow it to cool in a dessicator.
- 4.7 Record dessicator temperature and % humidity on the data sheet.
- 4.8 When the weighing dish has cooled, weigh the sample dish to the nearest 0.0001g. Record this value in Column labeled: Dry Sample + Dish (g).
- 4.9 Calculations

$$\% \text{ Solids} = \frac{\text{Sample dry weight}}{\text{Sample wet weight}} \times 100$$

Where:

Sample dry weight = (weight of dry sample + dish) – weight of dish

Sample wet weight = (weight of wet sample + dish) – weight of dish

5.0 PROCEDURE (automated calculation)

- 5.1 Open Solids program on PC. The login information for this program is the same as your Xenco login. Click on File, then New Batch, then choose the appropriate analytical method.
- 5.2 A box will appear with batch information to be filled in. You must fill in the analyst's name (drop down menu), the oven temperature in and out, and the dessicator temperature and humidity. Date/time information is automatically entered. (The information for the date/time and temperature out, and the dessicator readings will be corrected before the dry sample results are entered.) Click OK to bring up the sample backlog.
- 5.3 Click the check box beside all the samples that are to be analyzed in the backlog, and then click OK. This will put the samples into the analysis spreadsheet. To create a duplicate of a sample, click on the Lab Sample ID to highlight, then click edit, then create duplicate.
- 5.4 Place a pre-dried, labeled dish on the analytical balance. Highlight the "dish" box on the spreadsheet corresponding to the sample to be analyzed. When the reading becomes stable, hit enter on the PC keyboard. The weight will be entered on the spreadsheet in the highlighted box.

- 5.5 Add between 5-10g of wet soil to the dish on the balance. Click on the "wet dish/smp" box, and hit enter. Remove the dish and sample and place it in the drying oven. Continue for the rest of the samples in the batch.
- 5.6 When all the initial dish and wet sample weights have been entered on the spreadsheet, click Edit on the top menu, then "update xenco". The data will be transferred into a Xenco analytical batch. Print the preliminary batch data from the Edit menu, and record the Xenco batch number on it.
- 5.7 Follow steps 4.4 through 4.7 above.
- 5.8 When the samples are cooled, reopen the Solids program. Click on File from the top menu, the "open from Xenco". Choose the Xenco batch number for your samples.
- 5.9 Choose Edit from the top menu, then "batch info". Enter the correct date/time and Temp out for the drying oven, and the correct dessicator readings.
- 5.10 Place a dried sample/dish on the analytical balance. Choose the appropriate "dry dish/smp" box on the spreadsheet, and hit enter. The percent solids for the sample will be calculated by the software, using the same equation as in 4.9 above. The %RPD for the sample/duplicate pairs will also be calculated.
- 5.11 When all dry samples have been entered, click on Edit from the top menu, and choose "update Xenco". The Xenco batch originally created will be updated with the final results. Print the final results spreadsheet for the Edit menu, and tape a copy in the %Solids logbook.

6.0 QUALITY CONTROL

- 6.1 Duplicate percent solids determinations must be performed for every 20 samples per batch and matrix.
- 6.2 Check the humidity level of the dessicator. The humidity level should not exceed 20%. All % solids exceeding 20% humidity levels will be reset.
- 6.3 All data must be recorded on the data recording sheets in the Percent Solids Logbook.

7.0 SAFETY

- 7.1 Sample drying should take place in a hood or other well ventilated area.
- 7.2 Care shall be taken in the use of the drying oven as with any heated device.

8.0 DISPOSAL REQUIREMENTS

- 8.1 Details concerning disposal characteristics and procedures can be located in the SOP D.1 "Laboratory Waste Handling and Disposal Procedure".

9.0 DEFINITIONS

- 9.1 For definition of terminologies used in this document, refer to GPL Laboratories SOP G.14.

UNCONTROLLED DOCUMENT
AND
BUSINESS CONFIDENTIAL

SOP No: M.5

Title: Volatile Organics - Method 8260B

1.0 SCOPE AND APPLICATION

- 1.1 This method is used to identify and quantify purgeable compounds in waste, water, soil, and various types of waste samples using GCMS methodology. The compounds listed below include the most common requested volatile parameters.

Target Compound List

Dichlorodifluoromethane
Chloromethane
Vinyl Chloride
Bromomethane
Chloroethane
Trichlorofluoromethane
1,1-Dichloroethene
1,1,2-Trichloro-1,2,2-Trifluoroethane
Acetone
Carbon Disulfide
Methyl Acetate
Methylene Chloride
Trans-1,2-Dichloroethene
Methyl tert-Butyl Ether
1,1-Dichloroethane
cis-1,2-Dichloroethene
2-Butanone
Chloroform
1,1,1-Trichloroethane
Cyclohexane
Carbon Tetrachloride
Benzene
1,2-Dichloroethane
Trichloroethene
Methylcyclohexane
1,2-Dichloropropane

Bromodichloromethane
Cis-1,3-Dichloropropene
4-Methyl-2-Pentanone
Toluene
Trans-1,3-Dichloropropene
1,1,2-Trichloroethane
Tetrachloroethene
2-Hexanone
Dibromochloromethane
1,2-Dibromoethane
Chlorobenzene
Ethylbenzene
Xylenes (total)
Styrene
Bromoform
Isopropylbenzene
1,1,2,2-Tetrachloroethane
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
1,2-Dibromo-3-chloropropane
1,2,4-Trichlorobenzene

Appendix IX List

** Acetonitrile
** Acrylonitrile*
Allyl Chloride
** 2-Chloro-1,3-butadiene
Dibromomethane
trans-1,4-Dichloro-2-butene
p-Dioxane*
Ethyl methacrylate
Isobutyl alcohol
Methacrylonitrile
Methyl iodide
Methyl methacrylate
Pentachloroethane
Propionitrile*
1,1,1,2-Tetrachloroethane
Hexachloroethane
1,2,3-Trichloropropane
1,1-Dichloropropylene
1,4-Dioxane
** Acrolein
Bromochloromethane

* Poor purging efficiency / **poor performers resulting in high EDL.

Additional Compounds

2, 2 - Dichloropropane
1, 3 - Dichloropropane
Bromobenzene
n - Propylbenzene
2 - Chlorotoluene
4 - Chlorotoluene
1, 3, 5 - Trimethylbenzene
tert - Butylbenzene
1, 2, 4 - Trimethylbenzene
sec - Butylbenzene
4 - Isopropyltoluene
n - Butylbenzene
Hexachlorobutadiene
Naphthalene
1, 2, 3 - Trichlorobenzene
Iodomethane

TABLE 1

<u>Comp No.</u>	<u>Compound Name</u>	<u>Quantitation Ion</u>
1)	Dichlorodifluoromethane	85
2)	Chloromethane	50
3)	Vinyl Chloride	62
4)	Bromomethane	94
5)	Chloroethane	64
6)	Trichlorofluoromethane	101
7)	1,1-Dichloroethene	96
8)	1,1,2-Trichloro-1,2,2-Trifluoroethane	101
9)	Acetone	43
10)	Carbon Disulfide	76
11)	Methyl Acetate	43
12)	Methylene chloride	84
13)	trans-1,2-Dichloroethene	96
14)	Methyl tert-Butyl Ether	73
15)	1,1-Dichloroethane	63
16)	Cis-1,2-Dichloroethene	96
17)	2-Butanone	43
18)	Chloroform	83
19)	1,1,1-Trichloroethane	97
20)	Cyclohexane	56
21)	Carbon Tetrachloride	117
22)	Benzene	78
23)	1,2-Dichloroethane	62
24)	Trichloroethene	130
25)	Methylcyclohexane	83
26)	1,2-Dichloropropane	63
27)	Bromodichloromethane	83
28)	cis-1,3-Dichloropropene	75
29)	4-Methyl-2-pentanone	43
30)	Toluene	91
31)	trans-1,3-Dichloropropene	75
32)	1,1,2-Trichloroethane	97
33)	Tetrachloroethene	164
34)	2-Hexanone	43
35)	Dibromochloromethane	129
36)	1,2-Dibromoethane	107
37)	Chlorobenzene	112
38)	Ethylbenzene	106
39)	Xylene (total)	106
40)	Styrene	104
41)	Bromoform	173
42)	Isopropylbenzene	105
43)	1,1,2,2-Tetrachloroethane	83

44)	1,3-Dichlorobenzene	146
45)	1,4-Dichlorobenzene	146
46)	1,2-Dichlorobenzene	146
47)	1,2-Dibromo-3-Chloropropane	75
48)	1,2,4-Trichlorobenzene	180

*Poor performers.

2.0 SUMMARY OF METHOD

- 2.1 The volatile compounds are introduced into a gas chromatograph by the purge and trap method.
- 2.2 An inert gas is bubbled through the solution at ambient temperature (at elevated temperatures for soil samples), and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is heated to elute the components, which are detected with a mass spectrometer.

3.0 INTERFERENCES

- 3.1 Interferences purged from the samples will vary considerably from source to source, depending upon the particular sample or extract being tested. The analytical system, however, should be checked to ensure freedom from interferences and contamination. All samples and QC samples must be examined for the possibilities of contamination or carryover. If high concentrations of analyte(s) are found in a sample, the next sample(s) on the sequence batch should be checked for possible carryover. Concentrations comparable to the highest levels of the calibration range are considered a potential source of carryover. Usually those compounds that are quantified against the second and third internal standards (applicable to volatiles) may cause carryover. After the analysis of a sample containing high concentration of volatile organic compounds, one or more blanks should be analyzed to check for cross-contamination. Alternatively, if the sample immediately following the high concentration sample does not contain the organic compounds present in the high level sample, freedom from contamination has been established. If the sample is suspected to have carryover, re-analysis should be performed. The ALS (Automatic Liquid Sampler) position that contained the sample with the high levels of contaminants needs to be marked and cleaned. The next sample loaded in that position needs to be monitored for any possibilities of carryover. The contaminated position and corrective action performed must be documented on the injection log. Contamination generated by non-target compounds also needs to be monitored. The level of carryover by non-target compounds should be compared to the internal standards. The significance of the non-target

compound carryover should be evaluated by the supervisor to determine the overall impact on the sample results.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph

Hewlett-Packard 5890 Gas Chromatographic Systems (GC) are employed for execution of this method. The GCs are complete with temperature programming capabilities, and all required accessories such as columns, gases, cooling valves and syringes. Sample introduction is accomplished through the use of purge and trap systems.

4.2 Purge and Trap System

The Tekmar 2000/3000 purge and trap system is coupled with the gas chromatographic system. The system is equipped with a Tekmar 2016 sixteen position autosampler, and a sample heating device for soil samples.

The purge and trap device consists of three separate modules. The sample purger, the trap and the desorber. The purging chamber accepts glass vessels capable of holding at least 5-ml of liquid samples or 5 grams of soil samples. The glass tubes are stored in an oven at 105°C to prevent contamination from organic vapors.

The trap utilized by the laboratory is 25 cm long and has an inside diameter of 0.105 in. Traps are marketed as VOCARB 3000 by vendors such as Supelco, and consist of graphitized carbon.

A complete description of the inner workings of this purge and trap model is available in the Tekmar 2000/3000 manual.

4.3 Mass Spectrometer

The HP-5970 quadruple Mass Selective Detector is used as the detection device for Method 8260B. This system is capable of scanning from 35-260amu every one second or less, using 70 volts (nominal) electron energy in the EI mode. After proper tuning, the system produces a mass spectrum that meets all the criteria in Table 4 when 50ng of 4-Bromofluorobenzene (BFB) are injected into the gas chromatograph.

4.4 Data System

Each GC/MS system is attached to a dedicated 486 or pentium based computer equipped with Enviroquant software for automated acquisition and processing. The software generates total and extracted ion profiles of each compound and is capable of performing library searches on spectra using a full EPA/NIH Mass

Spectral Data Library. Each system is attached to an internal laboratory computer network for additional data processing, storage and archiving.

4.5 Column

Megabore column, 60 meters in length, with a 0.53 mm internal diameter and 2.0 micron film thickness is used.

4.6 Microsyringes - 10ul, 25ul, 100ul, 250ul, 500ul, and 1000ul.

4.7 Luerlock syringe - 5 and 25ml gas tight.

5.0 REAGENTS AND STANDARDS

5.1 Organic free reagent water.

5.2 Stock solutions.

5.3 Methanol, High purity grade, B & J Brand for Purge and Trap analysis.

6.0 SAMPLE COLLECTION AND PRESERVATION

6.1 Water samples may be collected in glass containers having a total volume of at least 40mL with a teflon-lined septum and an open top screw-cap. Soil samples may be collected in glass containers or closed end tubes (e.g., brass sleeves) in sufficient quantity to perform the analysis. Headspace should be avoided. The specific requirements for site sample collection are outlined by the Region.

6.2 For collection of water samples, the containers must be filled in such a manner that no air bubbles pass through the sample as the container is being filled. Seal the vial so that no air bubbles are entrapped in it.

6.3 Water samples are preserved to a pH of 2 at the time of collection.

6.4 All samples must be iced or refrigerated at 4°C ($\pm 2^\circ\text{C}$) from the time of collection until analysis.

6.5 All water and soil samples must be analyzed within 14 days of collection.

7.0 METHOD DETECTION LIMIT

7.1 Method detection limits for this method are listed in the GPL Laboratory Method Detection Limit and Reporting Limit official book.

8.0 METHOD PERFORMANCE

8.1 The MDL concentrations listed in the GPL MDL book are generally obtained using organic-free reagent water. Results were also obtained by extracting seven spiked replicates the same way as the samples and analyzing them. MDL is defined as the minimum concentration of a substance that can be measured and a reported with 99% confidence that the value is above zero. Precision and accuracy studies are performed once a year at a minimum. Single operator precision, overall precision and method accuracy were found to be directly related to the concentration of the parameter.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Calibration

Five concentration levels are analyzed for each analyte and surrogate standards. A minimum five point curve is then created after all 5 points are analyzed. Calibration levels are analyzed at 5, 20, 50, 100, 150 and/or 200ug/l for all analytes and surrogate standards except for 2-Chloroethyl vinyl ether which is analyzed at 40, 100, 200, 300 and 400ug/l.

9.2 Instrumentation Parameters

The following set of operating conditions exist for method 8260B.

Electron energy:	70 volts (nominal)
Mass range:	35-260amu
Scan time:	Not to exceed 1 second/scan
Initial column temperature:	10° C
Initial column holding time:	4 min
Column temperature program:	8° C/minutes
Final column temperature:	190° C
Final column holding time:	1 min
Injector temperature:	220° C
Source temperature:	According to manufacturer's specifications
Transfer line temperature:	280° C (Preset to manufacturer)
Carrier gas:	Helium at about 8ml/min

9.3 Tuning

9.3.1 At the beginning of each day, each GC/MS system is injected with 50ng of BFB and tuned to meet the criteria listed in Table 4. The analysis must not commence unless the criteria are met. This requirement must be met for each 12 hour interval.

9.3.2 One of the following approaches is used to evaluate the BFB tune.

- One scan at the apex without background subtraction.
- Three scans (the apex peak scan and the scan immediately preceding and following the apex) are acquired and averaged.
- Use the mean of the apex and the preceding of the following scans.
- Use the average across the entire peak.

9.4 Calibration Requirements

Once tuning requirements are met, the initial or continuing calibration check must be established. The generation of response factors is the next step in establishing the calibration requirements for method 8260B. The instructions for determining the response factors are as follows:

9.4.1 Tabulate the area response of the quantitation ions (see Table 1) against the concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard with the retention time closest to the compound being measured. The RF is calculated as follows:

$$RF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)}$$

where:

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_{is} = Concentration of the specific internal standard.

C_x = Concentration of the compound being measured.

The average RF must be calculated for each compound. A system performance check should be completed before the calibration curve is used. Five compounds, the System Performance Check Compounds, or SPCCs, are checked for a minimum average response factor. These compounds are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene (see Table 3). The minimum relative response factors for these compounds should be 0.1, 0.1, 0.1, 0.3 and 0.3. These compounds typically have RFs respectively, of 0.4-

0.6 and are used to check compound instability and check for degradation caused by contaminated lines or active sites in the system. Initial calibration is analyzed at a minimum of 5 levels. These levels are 5, 20, 50, 100, 150 and/or 200ug/l. Acrolein and acrylonitrile have higher initial calibration levels.

	<u>Levels</u>					
	<u>5ppb</u>	<u>20ppb</u>	<u>50ppb</u>	<u>100ppb</u>	<u>150ppb</u>	<u>200ppb</u>
Acrolein	25	100	250	500	750	1000
Acrylonitrile	25	100	250	500	750	1000
2 - Chloroethyl Vinyl Ether	20	80	200	400	600	800

Using the RFs from the initial calibration, calculate the percent relative standard deviation (% RSD) for all compounds.

$$\% \text{ RSD} = \frac{\text{SD} \times 100}{\bar{x}}$$

where:

- RSD = relative standard deviation
- X = mean of 5 initial RFs for a compound
- N = Number of calibration points
- SD = standard deviation of average RFs for a compound
- X_i = Response factor for each point

$$\text{SD} = \frac{\sum_{i=1}^N (X_i - \bar{X})}{N - 1}$$

9.4.2 After analyzing the initial calibration standard, average response factor RF should be calculated for each compound. The percent relative standard deviation %RSD should be equal or less than 15% for each compound. However, Calibration Check Compounds (CCC's) must have a %RSD less than or equal to 30%. It is likely that some analytes may exceed the 15% acceptance limit for the %RSD. In those instances the following steps should be considered:

- If the %RSD is greater than 15%, the analyst should review the results (area counts, response factors, standard concentrations) for those analytes to ensure that the problem is not associated with a single standard. If so, that standard should be reanalyzed and RSD recalculated. Replacing of standard may be necessary in some cases.

- It may also be necessary to narrow the calibration ranges to achieve a better linearity. High standard may saturate the column or/and the detector and need to be dropped from the calibration curve accordingly. Similarly poor purging compounds that exhibited erratic chromatographic behavior in the lowest calibration point could also be reviewed and dropped if necessary. The changes to the upper end of the calibration will affect the need to dilute samples above that range, while the change to the lower calibration will affect the sensitivity and elevate the reporting limit of the method. When considering dropping one or two points to narrow the calibration range, a minimum of five points is required for curve to be acceptable.

9.4.2.1 After visual inspection of the calibration points for each analytes and for those analytes that did not meet the %RSD criteria, at the discretion of the analyst it should consider to employ a regression equation that does not through the origin or a quadratic (second order) model. Linearity is presumed acceptable if the correlation coefficient (r) is equal to or greater than 0.995.

9.4.2.2 If linear regression curve did not match the response being observed, quadratic equation should be considered as an option. A quadratic model requires six standard. Linearity is presumed acceptable if the correlation coefficient (r^2) is equal to or greater than 0.99.

9.4.2.3 Prior to use for sample analysis, the acceptability of initial calibration curve must be verified through analysis of calibration verification (ICV) solution obtained from second source. Calibration verification analysis should meet the same acceptance criteria used for continuing (daily) calibration.

9.4.3 Continuing (Daily) Calibration

The initial calibration curve for each compound of interest must be checked and verified once every 12 hours of analysis time. This is accomplished by analyzing a 50ppb calibration standard and verifying if all compound of interest meet the acceptance criteria for daily calibration. Daily calibration standard analysis must meet the relative response factor criteria for SPCC and non SPCC % difference criteria of calibration check compounds (CCC) and non CCC. If SPCC and non SPCC criteria listed in Table 3 are not met, the system must be evaluated and corrective action must be taken before sample analyses begins. Potential problem include standard mixture degradation, injection port inlet contamination, contamination of front end of the analytical column, and active sites in the column or chromatographic system. After the criteria for relative response factor (RRF) are met, calculate the % difference of each compound to check the validity of the initial calibration.

Calculate the % difference using the following equation:

$$\% \text{Difference} = \frac{\text{RRF}_c - \text{RRF}_i}{\text{RRF}_i} \times 100$$

where:

RRF_c = Relative response factor from continuing calibration standard

RRF_i = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria

If the 20 percent difference for each CCC and non-CCC (except 8 poor purging or performing compounds that could have %D less than or equal to 40%) did not meet, corrective action should be taken. After corrective action, if the source of the problem cannot be determined, a new five point calibration MUST be generated. This criteria must be met before sample analysis begins. The internal standard response and retention times in the calibration check standard must be evaluated immediately after or during data acquisition. Tables 2 and 3 list SPCC and CCC compounds.

9.4.4 When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations, raw data output showing the results of manual integration and notation of date and initials of person performing the task.

9.5 Retention Time and Area Change

9.5.1 If the retention time of any internal standard of a sample or QC sample change by more than 30 seconds from the daily calibration verification standard or if the daily calibration verification standard change by more than 30 seconds from mid level standard of the latest initial calibration corrections must be made. Affected samples must be reanalyzed after problem is corrected.

9.5.2 If internal standard area in the samples changes by a factor two (-50% to +100%) from the calibration standard verification, corrections must be made. After corrections reanalysis of samples analyzed while the system was malfunctioning is required. Area of the daily calibration standard verification must not change by a factor of two (-50% to +100%) from that in the midpoint standard of the most recent initial calibration.

10.0 DATA ANALYSIS AND CALCULATION

10.1 Qualitative Analysis

10.1.1 The compounds listed in Section 1 shall be identified by an analyst by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the identification.

10.1.1.1 Relative retention time of the sample and standard must agree ± 0.05 .

10.1.1.2 Correspondence of the sample component and standard component mass spectra.

10.1.2 The requirements for qualitative verification by comparison of mass spectra are as follows:

All ions present in the standard mass spectra at a relative intensity greater than 10% must be present in the sample spectrum. All ions specified must agree within $\pm 20\%$ between the standard and sample spectra. Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for.

10.2 Quantitative Analysis

When a compound is identified, quantification is based on the integrated abundance of the primary characteristic ion and the response and amount of the corresponding internal standard.

$$\text{Concentration (ug/L)} = \frac{(A_x)(I_{Is})(D)}{(A_{Is})(\text{Avg RF})(V_o)}$$

$$\text{Concentration (ug/kg)} = \frac{(A_x)(I_{Is})(D)}{(A_{Is})(\text{Avg RF})(W)(S)}$$

where:

- A_x = Area of primary ion
- I_s = Amount of internal standard, ug/L
- A_{is} = Area of ion for internal standard
- Avg RF = Mean relative response factor for compound measured
- V_o = Volume of sample purged, L
- D = Dilution factor
- W = Weight in Kg
- S = % Solid

10.3 For non-TCL components, a library search may be executed. Up to 20 non-target organic compounds shall be tentatively identified when required.

Guidelines for making tentative identification:

10.3.1 Ions greater than 10% of the most abundant ion should be present in the sample spectrum.

10.3.2 Relative intensities of the major ions should agree within $\pm 20\%$.

10.3.3 Molecular ion present in the reference spectrum should be present in sample spectrum.

.1.1 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

11.0 PROCEDURE

11.1 Sample Preparation

11.1.1 For sample preparation and introduction of close system purge and trap and extraction of soil and water volatile compounds to the mass spectrometer, refer to GPL SOP M.7.

11.2 Water Samples

11.2.1 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

11.2.2 BFB tuning criteria and daily GC/MS calibration criteria must be met before analyzing samples.

11.2.3 Adjust the purge gas (Helium) flow rate to approximately 40ml/min on the purge and trap device.

11.2.4 Remove the plunger from a 5ml syringe. Open the sample and carefully pour the sample into the syringe barrel to just short of overflowing. Compress the sample. Vent any residual air while adjusting the sample volume to 5.0ml.

11.2.5 Add 5.0ul of surrogate spiking solution and 5ul of internal standard solution (50ppm each) through the valve bore of the syringe and load samples into the sample tube. Immediately after loading the sample, verify the pH of the samples using narrow range pH paper. Sample vials should not be opened to verify pH prior to loading into the purge unit.

11.2.6 Lab Control Spikes (LCS) is analyzed per batch or every 20 samples and at the same frequency as the method blank. LCS is spiked with all target

compound of interest. LCS compound must meet the criteria outlined in the Quality Control section 12.7.

11.2.7 For the matrix spike analysis, add 5ul of the matrix spike solution (50ppm) to the sample to be purged. This will yield a final concentration of 50ug/L (ug/kg for soils) in the final sample. Matrix spike QC sample must contain all target analytes of interest.

11.2.8 The samples are analyzed with appropriate dilution when the concentration level of any analyte exceeds the calibration range. Dilutions are made in the 5ml gas tight luer lock syringe by adding reagent water. Calculate the volume of reagent water needed for the dilution and add into a 5ml syringe. Dilution of samples should result in analysis with the highest concentration target analyte in the upper half of the calibration range. Using a suitable syringe, add the exact volume of sample into the reagent water in the 5ml syringe. Add 5ul of Internal Standard & Surrogate and analyze as discussed above. If the dilute sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution.

11.2.9 The spiked sample is injected into a purging tube attached to the Purge & Trap device. Purge the sample for 11 minutes and desorb the contents of the trap at 200° for 2 minutes into the GC/MS.

11.2.10 Low Concentration Method: This method is applicable to water samples containing low levels of contaminants. The detection limits are generally improved by analyzing larger sample volume (25ml). The detection limits of compounds with poor purging efficiency (such as ketones) do not significantly improve when low concentration method is utilized. The low concentration analytical procedure is virtually the same as low level water analysis except for concentration of the calibration, surrogate and internal standards. The following concentration levels and ranges are analyzed:

Calibration	- .5, 1, 10, 20, 40, 60ug/L
Surrogates	- .5, 1, 10, 20, 40, 60ug/L
Internal Standards	- 10ug/L
Continuing Calibration	- 10ug/L

Acceptance criteria for the initial and continuing calibrations is the same as the normal –5mL purge. Criteria for percent RSD and difference in the initial and continuing calibrations are described in section 8.4. Minimum response factor for the poor purging or poor performers is 0.010.

11.2.11 Cleaning of purge and trap cells.

Upon completion of volatile analysis, the purge and trap cells are disconnected from the ALS. The purge and trap cells are cleaned with deionized water and mild soap, and rinsed with deionized water. The purge and trap cells are then baked in an oven at 105° for at least four hours. Upon completion of baking, the purge and trap cells are placed in a container covered with aluminum foil.

11.3 Sediment/Soil and Waste Samples

11.3.1 Low Concentration method: Applicable for samples containing individual compounds of <1mg/kg. All granular/porous waste/sediment/soil samples can be analyzed using this method.

Weigh 5g of the sample into a glass sparge tube and record the weight to the nearest 0.1g. Add reagent water to a 5ml luerlock type syringe and adjust the volume to 5ml. Add 5ul of internal standard and 5ul of surrogate standard to the water. Connect the sparge tube with the sample to the purge & trap device and add the spiked water to the sample. Heat the sample to 40°C ±1°C and purge the sample for 11 minutes. Desorb the trap at 200°C for 2 minutes into the GCMS. If the concentration level of any analyte exceeds the calibration range and is below 1mg/kg, the sample should be analyzed by weighing 1g of the sample. If the concentration level of any analyte is higher than 1mg/kg, then the high concentration method should be used.

11.3.2 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

11.3.3 BFB tuning criteria and daily GC/MS calibration criteria must be met before analyzing samples.

11.3.4 Adjust the purge gas (Helium) flow rate to approximately 40ml/min on the purge and trap device.

11.3.5 Weigh 5.0 grams of the soil sample into a tared purge device. Use a top loading balance. Note and record the actual weight to the nearest 0.10g. Quickly connect the device to the purge and trap system.

11.3.6 Prepare a soil method blank which consists of a 5g of a purified solid matrix to reagent water with 5ul of surrogate and 5.0ul of internal standard solution (50ppm each). Add the solution through the valve pore of the syringe into the soil sample tube.

11.3.7 Lab Control Spikes (LCS) is analyzed per batch or every 20 soil/sediment samples and at the same frequency as the method blank. LCS is spiked with all target compound of interesting in a purified solid matrix. LCS

compound must meet criteria specified in the Quality Control section 12.7.

11.3.8 For the matrix spike analysis, add 5ul of the matrix spike solution (50ppm) to the sample to be purged. This will yield a final concentration of 50ug/L (ug/kg for soils) in the final sample. Matrix spike QC sample must contain all target analytes of interest.

11.3.9 High Concentration Method: The method is based on extracting the soil/sediment with methanol. Weigh 4gm of the sample into a 20ml vial using a top loading balance. Record the weight to the nearest 0.1g. Using a pipet, add 9.0ml methanol; then add 1.0ml of the surrogate spiking solution into the vial. Close the cap and shake for 2 minutes. Pipet approximately 1ml of the extract to a GC vial for storage, using a disposable pipet. Transfer approximately 1ml of the method blank to a separate GC vial for each set of samples. The standards, LCS and blanks should contain 100ul of a solvent to simulate the sample condition. 100ul of the extract is added to reagent water collected in a 5ml luerlock syringe. 5ul each of Internal Standard is added to the syringe. Proceed with the analysis as outlined in the water sample analysis. If further dilution is needed, a smaller extract volume is used and the same procedure is repeated.

A separate method blank containing 100ul methanol should be analyzed prior to a high concentration sample.

11.3.10 For matrix spike in the high concentration sediment/soil samples, 8.0ml of methanol, 1.0ml of surrogate spike solution, and 1.0ml of spike solution is added to a 4g sample. Cap and shake for 2 minutes. Add a 100ul aliquot of this extract to 5ml of organic free reagent water for purging.

11.3.11 Samples extracted through Toxicity Characteristic Leachate Procedure by zero headspace extractor are analyzed by this method. Only those compounds designated by the TCLP method are accounted for. Samples are analyzed at 10 times dilution in order to minimize the effects of acetic acid on the chromatography and purge and trap systems.

12.0 QUALITY CONTROL

12.1 Prior to initiating on going data, it is necessary that the GC/MS meets BFB abundance criteria (Table 4) every twelve (12) hour period for the method.

12.2 When twelve (12) hours have elapsed since the initial tune, the GC/MS must be returned to continue the analysis.

- 12.3 Internal standard responses and retention times in the calibration checks, blanks and samples must be evaluated. Retention time variation of any internal standard in the calibration standard must be less than 30 seconds from that in the midpoint standard in the calibration standard. All other samples and QC samples must be compared to the daily CCV and should not vary by more than or equal to 30 seconds. If this criteria exceeds samples that did not meet the criteria should be re-injected.
- 12.4 Surrogate recoveries must be evaluated by determining whether the concentrations fall within internal QC limits. If surrogate recoveries falls outside the established QC limits, then analyze the sample that fail the criteria.
- 12.5 A matrix spike and spike duplicate must be performed every tune or 20 samples, whichever comes first, at the concentration equal to mid-level calibration (50ug/l). If matrix spike QC recoveries does not meet established acceptance criteria, the analyst and the manager must assess the batch to determine whether the spike results are attributable to matrix affect, or the result of other problems in analytical process. If all batch QC elements which are not affected by matrix are in control (e.g., method blank, LCS) the poor recovery may be attributed to matrix affect. If the native concentration is low, and the MS/MSD recoveries confirm matrix interferences, dilute the MS/MSD and reanalyze. TCLP samples are spiked with TCLP target compounds at 50ug/l concentration.
- 12.6 A method blank must be performed for each 12 hour time period.
- 12.6.1 A method blank for volatile analysis must contain less than or equal to $\frac{1}{2}$ of the reporting limits. Any method blank must comply with the following criteria:
- If the concentration of target analyte in the method blank is greater than MDL but less than the reporting limit, rinse the purging apparatus with two portions organic free reagent water and reanalyze the method blank. After corrective action, if low level contamination is still detected, associated sample data may be reported with qualifier.
 - If the concentration of any target analyte in the method blank is greater than the reporting limit, it may be necessary to wash the purging device with a soap solution, rinse it with organic free reagent water, and then dry the purging device in an oven at 105°C. No sample should be analyzed before the contamination is eliminated.
- 12.7 Lab Control Spikes (LCS) is analyzed per batch or every 20 samples and at the same frequency as the method blank. LCS is spiked with all target compound of interest. Statistical control limit are based on 20 data points. Data points used in the data set must not be selectively included or excluded.

Each LCS must be evaluated against the Control Limits and Marginal Exceedance limit. If any LCS is outside the Control Limit, it should be also compared to the Laboratory Marginal Exceedance Limits to ensure that it does not exceed. If a single analyte in the LCS exceeded the Marginal Exceedance Limit, the LCS had failed and corrective action needs to be initiated. If the LCS has more than the allowable number of Marginal Exceedance, the LCS has failed and corrective action needs to be initiated.

The corrective action for failed LCS is based on professional judgment in conjunction with matrix spike and surrogate recoveries in the same batch. If after checking the associated QC's it was determined by the section supervisor or manager that the LCS had failed, all affected samples associated with the out of control LCS should be reprocessed and re-analyzed.

If samples cannot be reprocessed due to lack of sample volume or the holding times has lapsed, the results should be reported with appropriate flag. The report case narrative must include a discussion of the failed LCS, and its impact on the data quality.

When LCS is spiked with large number of analytes, the laboratory should add up total number of exceedances for the LCS based on the number of analyte spiked in the LCS. The total of exceedance should be compare with the allowable number from the following chart.

Since a large amount of GPL's clients most requested compound list are fairly long (> 20 compounds), the laboratory will spike all the reportable compounds as specified in the clients project. At a minimum, 16 compounds from the list will be selected and used to verify if the LCS meets the required QC requirements. However, the laboratory should ensure that all compounds in the target list are used to verify the LCS criteria over a period of two years.

Number of analyte in the LCS	Allowable Number of Marginal Exceedances of LCS
< 11	0
11-30	1
31-50	2
51-70	3
71-90	4
>90	5

Since a large amount of GPL's clients most requested compound list are fairly long (> 20 compounds), the laboratory will spike all the reportable compounds as specified in the clients project. At a minimum, 16 compounds from the list will be selected and used to verify if the LCS meets the required QC requirements. However, the laboratory shall ensure that all compounds in the target list are used to verify the LCS criteria over a period of two years.

12.8 QC limits for MS/MSD, LCS and surrogates are established semi-annually for internal use.

12.9 For DOD projects, DOD QSM control limits policy shall be followed for MS/MSD, LCS and surrogates.

13.0 MS SIM ANALYSIS

13.1 When requested, low concentration target analytes can be recovered using Selective Ion Monitoring (SIM) mode. Selective Ion Monitoring (SIM) mode is a data acquisition technique in which only a few selected ion fragments are monitored in order to obtain maximum selectivity. Standard spike volumes for surrogates, internal standards, matrix spikes, and laboratory control samples are dependent on the reporting limit requested.

13.2 Five calibration standard levels are analyzed at concentrations dependent on the reporting limit requested. All Method 8260B QC criteria applies to the 8260 SIM analysis.

14.0 SAFETY

14.1 Safety glasses, laboratory coats, and latex gloves must be worn.

14.2 Due to the toxicity or carcinogenicity of each reagent, each chemical compound should be treated as a potential health hazard.

14.3 Material Safety Data Sheets (MSDS) can be found on the procurement bookshelves located in the administrative area.

14.4 Standard preparation should be handled under a hood.

15.0 POLLUTION PREVENTION

15.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

16.0 WASTE MANAGEMENT

16.1 Several wastes that GPL generates can be handled in a fairly routine manner. The process of describing the method for waste disposal of chemicals including standards and reagent solutions, and process waste, and samples is described in Standard Operating Procedures D.1 and D.2.

17.0 DEFINITIONS

17.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

18.0 REFERENCES

- Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Revision 3, December 1996.
- DOD Quality System Manual, Final Version 3, January 2006

TABLE 2
CALIBRATION CHECK COMPOUNDS

- 1 - 1,1-Dichloroethene
- 2 - Chloroform
- 3 - 1,2-Dichloropropane
- 4 - Toluene
- 5 - Ethylbenzene
- 6 - Vinyl chloride

TABLE 3
SYSTEM PERFORMANCE CHECK COMPOUNDS

- 1 - Chloromethane
- 2 - 1,1-Dichloroethane
- 3 - Bromoform
- 4 - 1,1,2,2-Tetrachloroethane
- 5 - Chlorobenzene

All other compounds must meet a minimum RRF of 0.050

TABLE 4

BFB KEY ION ABUNDANCE CRITERIA

<u>Mass</u>	<u>Ions Abundance Criteria</u>
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

SOP No: N.6

Title: Method 3520C, Continuous Liquid-Liquid Extraction for Pesticide/PCBs

1.0 SCOPE AND APPLICATION

- 1.1 This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics such as organochlorine pesticides and PCBs from water, wastewater, groundwater and TCLP extracts. Extracts produced by this method may be analyzed by Methods 8081A and 8082.
- 1.2 This method also describes concentration techniques suitable for preparing the extract for the appropriate method.

2.0 DEFINITIONS

- 2.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

3.0 SUMMARY OF METHOD

- 3.1 A measured volume of sample, usually 1 liter, is placed into a continuous liquid-liquid extractor, adjusted, if necessary, to a specific pH and extracted with organic solvent for 18 ± 2 hours. The extract is dried, concentrated and exchanged into a solvent compatible with the step being employed.

4.0 INTERFERENCES

- 4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifact and/or interferences to sample analysis. All materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

5.0 APPARATUS AND MATERIALS

- Continuous Liquid-Liquid Extractor (Equipped with joints and snapping clips)
- Drying Column (A glass funnel with pyrex glass wool at the neck of the funnel, half filled with dry Na₂SO₄)
- Kuderna-Danish (K-D) Apparatus
 - Concentrator tube: 10ml, graduated (Kontes K-570050-1025 or equivalent). A ground-glass stopper is used to prevent evaporation of extracts.
 - Evaporator flask: 500ml (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs.
 - Snyder column: Three-ball macro (Kontes K-503000-0121 or equivalent)
- Solvent extracted Boiling Chips, approximately 10/40 mesh (silicon carbide or equivalent)
- Heated water Bath, with concentric ring cover, capable of temperature control ($\pm 5^{\circ}\text{C}$). The bath should be used in a hood.
- Glass vials, 2ml capacity, with Teflon-lined crimp cap
- pH Indicator Paper covering pH 2 to pH 12
- Rheostat controlled Heating Mantle
- 1.0ml Gas-Tight Syringe
- One (1) Liter Class A Graduated Cylinder
- Pyrex Glass Wool
- 1g florisil cartridge
- Large disposable glass pipets

6.0 REAGENTS

- Reagent Water (water in which an interferant is not observed at the method detection limit of the compounds of interest).
- Sodium Hydroxide Solution, 10N
(ACS) Dissolve 40g NaOH in reagent water and dilute to 100ml.

- Sodium Sulfate (ACS) Granular, Anhydrous (purified by heating at 400°C for 4 hours in a shallow tray)
- Sulfuric Acid Solution (1:1)
Slowly add 50ml of H₂SO₄ (sp. gr. 1.84) to 50ml of reagent water.
- Methylene Chloride (B & J GC2 grade or equivalent)
- Hexane (Pesticide grade)
- Acetone (pesticide grade)

7.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

- 7.1 Samples are collected in 1 liter amber glass bottles which have been precleaned and certified to be free of target analytes. Samples are shipped in coolers packed in ice and stored at the lab in refrigerators at 2-6°C until extraction. The holding time for extraction is 7 days from sample collection.

8.0 PROCEDURE

- 8.1 Place a few boiling stones in a 500ml collecting flask and assemble continuous extractor.
- 8.2 Add 400ml of methylene chloride to the extractor.
- 8.3 Using a Class A graduated cylinder, measure out 1 liter of sample and transfer it to the continuous extraction. Rinse the sample bottle with 20-30ml methylene chloride and add to the extractor. Add 1.0ml of surrogate spiking solution. Check the pH of the sample using wide range pH paper and adjust the pH to 7 using 1:1 H₂SO₄ or 10N NaOH. Use 1.0 liter of deionized water to another extraction for the method blank preparation and analysis.
- 8.4 For the sample in each analytical batch selected for spiking, measure two additional 1.0 liter aliquots. Add 1.0ml of the matrix spiking standard.
- 8.5 Add sufficient methylene chloride to the extractor to ensure proper operation and extraction for 18 ± 2 hours.
- 8.6 Allow to cool, then detach and label the boiling flask. The extract is then ready to be dried and concentrated.
- 8.7 Assemble a Kuderna-Danish (K-D) Concentrator by attaching a 10ml concentrator tube to a 500ml evaporation flask.
- 8.8 Dry the extract by passing it through a drying column containing about 10cm anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Rinse the flask which contained the solvent extract with 20-30ml of methylene chloride and add to the column to complete the quantitative transfer.

- 8.9 Add one or two clean boiling chips to the flask and attach a three ball Snyder Column. Pre-wet the Snyder Column by adding approximately 1ml of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (90-100°C) until the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration. When the apparent volume of liquid reaches about 10ml, remove the K-D apparatus from the water bath and perform a solvent exchange by adding 50ml of hexane through the top of Snyder column. Continue concentration in the water bath until the volume of extract reaches approximately 5ml.
- 8.10 Take the K-D off the water bath and place on a rack and allow to cool. Disconnect the concentrator tube and adjust the volume to 10.0ml with hexane.
- 8.11 If florisil cleanup is required, carefully homogenize the 10ml extract and take 1.0ml for cleanup.
- 8.11.1 Florisil cartridge cleanup
- Attach the vacuum manifold to a vacuum pump with a trap installed between the manifold and vacuum source. Place 1 florisil cartridge into vacuum manifold for each sample extract.
 - Wash each cartridge with hexane/acetone (90:10) v:v. Pass at least 5ml of hexane/acetone solution through the cartridge while pulling a vacuum, adjust the vacuum so that the flow rate is approximately equal through each cartridge. DO NOT LET THE CARTRIDGES GO DRY AFTER THEY HAVE BEEN WASHED.
 - After the cartridges have been washed, release the vacuum, and place a rack containing labeled 20ml vials inside the manifold. Care must be taken to ensure that the solvent line from each cartridge is in the appropriate cartridge as the manifold top is replaced.
 - Once the flasks are in place, restore vacuum to the manifold and place 1ml of the sample extract into the cartridge.
 - Elute the cartridge with 8ml of hexane/acetone (90:10), then rinse the cartridge with two 1ml portions of hexane.
 - Shut off the vacuum, take off the manifold top and remove the 20ml vials and place in the nitrogen blowdown manifold and blow the extract down to 1.0ml.

- Pipet the extract into labeled 1ml vials using a disposable pasteur pipet, taking care to rinse the sides of the 20ml vial with extract to ensure a quantitative transfer of extract. The 1ml vial is then transferred for GC analysis.

9.0 QUALITY CONTROL

9.1 Method Blank

- 9.1.1 The method blank is a volume of deionized, laboratory water carried through the entire extraction. The method blank volume must be equal to 1.0 liter or the sample volumes being extracted.
- 9.1.2 Frequency of Method Blanks
Method blank extraction must be performed per each batch of samples, not to exceed 20 samples per batch.

9.2 Matrix Spike/Matrix Spike Duplicate

9.2.1 MS/MSD Frequency of Extraction

Matrix spike and matrix spike duplicate extraction must be performed per each batch of 20 samples or less. If the batch of samples are received periodically, then MS/MSD are extracted every 14 days.

9.3 Matrix Spiking Solutions

9.3.1 Method 8081A matrix spikes

Matrix spike is prepared in acetone and 1.0ml is added to the designated samples. The mixture contains the following compounds at the given concentrations:

4,4'-DDD	0.5ppm
4,4'-DDE	0.5ppm
4,4'-DDT	0.5ppm
Aldrin	0.5ppm
Alpha-BHC	0.5ppm
Alpha-Chlordane	0.5ppm
Beta-BHC	0.5ppm
Delta-BHC	0.5ppm
Dieldrin	0.5ppm
Endosulfan I	0.5ppm
Endosulfan II	0.5ppm
Endosulfan Sulfate	0.5ppm
Endrin	0.5ppm
Endrin Aldehyde	0.5ppm
Endrin Ketone	0.5ppm
Gamma-Chlordane	0.5ppm
Heptachlor	0.5ppm

Heptachlor Epoxide	0.5ppm
Lindane (Gamma-BHC)	0.5ppm
Methoxychlor	0.5ppm

LCS spiking levels are the same as those used to spike samples.

9.4 Surrogate Spike (SS)

Each sample, matrix spike, and blank are spiked with surrogate compounds prior to extraction. The concentration in the following table exhibits the concentration in the spiking solution.

<u>Compounds</u>	<u>Concentration Added</u>
Decachlorobiphenyl	1.0ml of 0.2ug/ml
TCMX	1.0ml of 0.2ug/ml

9.5 Lab Control Spike (LCS)

Blank spikes are extracted at the same frequency as matrix spikes using deionized water as the matrix.

9.6 PCBs Spike (Method 8082)

When PCBs are only needed, follow the same extraction procedure as Pest/PCBs. For matrix spike and LCS QC runs use 1.0ml of AR1260 and AR1016 spiking solution of the concentration of 10.0 and 5.0ug/ml, respectively.

10.0 SAFETY

10.1 Safety glasses, lab coat, and gloves should be worn at all times during extraction.

10.2 Label reagents, spikes, surrogate solutions by identifying the solution, concentration, date, chemist's initials, and shelf life.

10.3 Waste Disposal

10.3.1 Chlorinated solvent waste should be put into a polyethylene safety can designated for methylene chloride. This will be disposed of by the hazardous waste coordinator.

10.3.2 Water waste can be poured down the drain after neutralizing it using H₂SO₄ or NaOH depending upon the pH of the waste.

11.0 WASTE MANAGEMENT

- 11.1 Several wastes that GPL generates can be handled in a fairly routine manner. The process of describing the method for waste disposal of chemicals including standards and reagent solutions, and process waste, and samples is described in Standard Operating Procedure D.1 and D.2.

12.0 POLLUTION PREVENTION

- 12.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

13.0 REFERENCES

- Test Methods For Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition.

SOP No: N.7

Title: Soil Extraction for Pesticides/PCBs Compounds by Method 3550B
(Sonication Extraction)

1.0 APPLICATION AND SCOPE

- 1.1 This method is a procedure for extracting organochlorine pesticide and PCB compounds from solids such as soils, sludges, and wastes. Extracts are suitable for analysis by methods 8081A and 8082.
- 1.2 Method 3550B describes both high level and low level extraction procedures. This SOP is based on the low level version of method 3550.

2.0 SUMMARY OF METHOD

- 2.1 A 30g sample is mixed with anhydrous sodium sulfate to form a free-flowing powder. Mixture is extracted with solvent three times using sonication. The extract is separated from the sample by filtration. The extract is ready for cleanup following concentration.

3.0 INTERFERENCES

- 3.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. These materials must be demonstrated to be free from interferences under the conditions of the analysis of analyzing method blanks.

4.0 APPARATUS AND MATERIALS

- 4.1 Mortar Model 155 Grinder, Fisher Scientific Company, or an equivalent brand and model.
- 4.2 Ultrasonic cell disrupter, Heat Systems - Ultra Sonic, Inc., Model W-385 (475 watt) Sonicator or equivalent (power wattage must be a minimum of 375 with pulsing capability and No. 200 1/2" Tapped Disrupter Horn) plus No. 207 3/4" Tapped Disrupter Horn, and No. 419 1/8" Standard Tapped Microtip Probe (where applicable).

- 4.3 Pasteur Glass Pipets, Disposable, 1ml
- 4.4 Beakers, 400ml
- 4.5 Filtration Apparatus
 - Conical funnel
 - Filter paper: Whatman No. 41 or equivalent
- 4.6 Kuderna-Danish (K-D) Apparatus
 - 4.6.1 Concentrator tube: 10ml graduated (Kontes K-570050-1025 or equivalent).
 - 4.6.2 Evaporator Flask: 500ml (Kontes K-570001-0500 or equivalent).
 - 4.6.3 Snyder Column: Three-ball macro (Kontes K-503000-0121 or equivalent).
- 4.7 Solvent extracted Boiling Chips, approximately 10/40 mesh (Silicon Carbide or equivalent).
- 4.8 Heated Water Bath, with concentric ring cover, capable of temperature control ($\pm 5^{\circ}\text{C}$). The bath should be used in a hood.
- 4.9 Top-loading Analytical Balance, capable of accurately weighing 0.01 grams.
- 4.10 12ml Vials and Caps
- 4.11 Glass Funnel
- 4.12 Nitrogen evaporation device equipped with a water bath that can be maintained at 35-40°C.
- 4.13 Spatula, Stainless steel or Teflon
- 4.14 Syringe, 1ml
- 4.15 Large disposable glass pipets
- 4.16 20ml scintillation vials with caps

5.0 REAGENTS

- 5.1 Sodium Sulfate: Anhydrous and reagent grade, heated at 400°C for 4 hours, cooled in a desiccator, and stored in a glass bottle. Baker anhydrous powder, catalog #73898, or equivalent.
- 5.2 Methylene chloride: acetone (1:1, V:V)

- 5.3 Methylene chloride (B&J GC2 grade or equivalent)
- 5.4 Hexane (pesticide quality or equivalent).
- 5.5 Concentrated sulfuric acid (reagent grade)
- 5.6 Florisil cartridges (0.5g packing B&J 9104 or equivalent)

6.0 PROCEDURE

6.1 Sample Handling and Storage

- 6.1.1 Samples are collected in appropriately sized glass bottles which have been pre-cleaned and certified to be free of target analytes. Samples are shipped in coolers packed with ice and samples must be refrigerated at 2-6°C upon arrival. Holding time for soil samples is 14 days from the collection date. Samples must be brought to room temp before extraction begins. Spare containers (if provided) should be used for matrix spike and re-extractions (if needed).
- 6.1.2 Sediment/soil samples: Decant and discard any water layer on a sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

- 6.2 The following step should be performed to avoid loss of the more volatile extractables. Weigh approximately 30g of sample into a 400ml beaker. Record the weight to be nearest 0.01g. Add 1ml of surrogate standards to all samples, spikes, and blanks. For the sample in each analytical batch selected for spiking, add 1.0ml of the matrix spiking standard. The surrogates and spiking solutions must be added before any processing of the sample and QC begins. Nonporous or wet samples (gummy or clay type) lacking a free-flowing sandy texture must be mixed with 10-20g of anhydrous sodium sulfate using a spatula. The sample should be free-flowing at this point. Then immediately add 100ml of 1:1 methylene-chloride: acetone.

6.3 Sonicator tuning procedure

The sonicator is to be tuned weekly, at a minimum, and whenever any of the components are changed or, at the discretion of the analyst. The following procedure is to be followed for tuning.

- 6.3.1 Turn the power output knob to zero.
- 6.3.2 Turn the sonicator mode switch to continuous mode.
- 6.3.3 Turn the sonicator power switch on.

- 6.3.4 Press and hold the sonicator tune switch on with one hand for the Tekmar sonicator; use the other hand to turn the power output knob or the sonicator tuning knob as follows. For the Misonix sonicators, the tune switch will stay on by itself.
- 6.3.5 Slowly turn the power output knob toward 10 while watching the output power meter reading. When the meter reading reaches 50%, turn the tuning knob until the minimum meter deflection is observed. The minimum deflection can be determined by observing when the needle starts to go back up when turning the tuning knob in either direction. The tuning knob for the Tekmar sonicator and the Misonix sonicators is on the front panel of the generator.
- 6.3.6 Proceed again with step (6.5.5.) until reaching output level 10 (output level 5 for microtips), and adjust the tuning knob to achieve the minimum deflection. At this point, the sonicator is considered tuned and operational provided that minimum deflection is achieved and the sonicator power output meter reads less than 25% at the minimum.
- 6.3.7 If the tuning criteria can't be met, check for loose cable connections or loose horn to converter connection and tighten as necessary. Retune. If the tuning criteria still can't be met, try replacing horn and converter with backup unit. Retune. If the tuning criteria still can't be met, notify the supervisor immediately so that repairs and/or a loaner can be obtained. Place a sign on the sonicator stating "Out of order, do not use".
- 6.3.8 Release the sonicator tune switch, switch the sonicator mode switch to pulsed mode and switch the sonicator off.
- 6.3.9 Record the tuning in the appropriate sonicator maintenance logs and weekly checklist.
- 6.4 Place the bottom surface of the tip of the #207 3/4" disrupter horn about 1/2" below the surface of the solvent, but above the sediment layer.
- 6.5 Sonicate for 3 minutes with the output control knob set at 10 and the mode switch set on pulse and the percent-duty cycle knob set at 8-10. DO NOT use a microtip probe.
- 6.6 Decant and filter extracts through Whatman No. 41 filter paper using filtration or centrifuge and decant extraction solvent.
- 6.7 Repeat the extraction two more times with two additional 100ml portions of solvent. Decant off the extraction solvent after each sonication. On the final sonication, pour the entire sample into the conical funnel and rinse with extraction solvent.
- 6.8 Assemble a Kuderna-Danish (K-D) Concentrator by attaching a 10ml concentrator tube to a 500ml evaporative flask.

- 6.9 Dry the extract by passing it through a drying funnel filled with anhydrous Sodium Sulfate. Collect the dried extract in a K-D Concentrator. Wash the extractor flask and Sodium Sulfate Column with 100-125ml of extraction solvent to complete the quantitative transfer.
- 6.10 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder Column. Pre-wet the Snyder Column by adding about 1ml methylene chloride to the top. Place the K-D apparatus on a hot water bath (90-100°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-15 minutes. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 10ml, remove the K-D apparatus and add 50ml of Hexane through the top of Snyder column and continue concentration until liquid volume reaches about 5ml.
- 6.11 Remove the Snyder Column and rinse the flask and its lower joints into the concentrator tube with 1-2ml of Hexane. Adjust the volume of extract to 10.0ml with hexane and proceed with any cleanups, if required.
- 6.12 If PCB's only are to be quantitated, an acid cleanup may be done as follows: Transfer the 10mL extract into a 20mL scintillation vial, add 5mL concentrated sulfuric acid, cap tightly and shake vigorously for 2 minutes. Let the layers settle and centrifuge if necessary. If the extract (top layer) is still cloudy repeat the cleanup as many times as necessary by pipetting the extract into a clean vial and adding a fresh volume of acid. If extracts are to be used for separate pesticide and PCB analysis, split the extract by using 5mL for the acid cleanup and the remaining 5mL save for subsequent florisil cleanup, if needed.
- 6.13 If florisil cleanup is required, carefully homogenize the 10ml extract and take 1.0ml for cleanup.
- 6.13.1 Florisil cartridge cleanup
- Attach the vacuum manifold to a vacuum pump with a trap installed between the manifold and vacuum source. Place 1 florisil cartridge into vacuum manifold for each sample extract.
 - Wash each cartridge with hexane/acetone (90:10) v:v. Pass at least 5ml of hexane/acetone solution through the cartridge while pulling a vacuum, adjust the vacuum so that the flowrate is approximately equal through each cartridge. DO NOT LET THE CARTRIDGES GO DRY AFTER THEY HAVE BEEN WASHED.

- After the cartridges have been washed, release the vacuum, and place a rack containing labeled 20ml vials inside the manifold. Care must be taken to ensure that the solvent line from each cartridge is in the appropriate cartridge as the manifold top is replaced.
- Once the flasks are in place, restore vacuum to the manifold and place 1ml of the sample extract into the cartridge.
- Elute the cartridge with 8ml of hexane/acetone (90:10), then rinse the cartridge with two 1ml portions of hexane.
- Shut off the vacuum, take off the manifold top and remove the 20ml vials and place in the nitrogen blowdown manifold and blow the extract down to 1.0ml.
- Pipet the extract into labeled 1ml vials using a disposable pasteur pipe, taking care to rinse the sides of the 20ml vial with extract to ensure a quantitative transfer of extract. The 1ml vial is then transferred for GC analysis.

7.0 QUALITY CONTROL

7.1 Method Blank

7.1.1 A method blank is purified solid matrix (NA₂SO₄) for soil samples, carried through the entire extraction. The method blank weight must be approximately equal to the sample weights being processed.

Method blank extraction must be performed per each batch of 20 samples or less.

7.2 Matrix Spike/Matrix Spike Duplicate

7.2.1 Matrix Spike/Matrix Spike Duplicate frequency of extraction

Matrix spike and matrix spike duplicate extraction must be performed per each batch of 20 samples or less. If the batch of samples are received periodically, then MS/MSD are extracted every 14 days.

7.3 Matrix Spiking Solution is prepared in acetone and 1.0ml is added to the designated samples. The mixture contains the following compounds at the given concentrations:

Analyte	Conc.	Analyte	Conc.
4,4'-DDD	0.5PPM	Endosulfan II	0.5PPM
4,4'-DDE	0.5PPM	Endosulfan sulfate	0.5PPM
4,4'-DDT	0.5PPM	Endrin	0.5PPM

Aldrin	0.5PPM	Endrin Aldehyde	0.5PPM
Alpha-BHC	0.5PPM	Endrin Ketone	0.5PPM
Alpha-Chlordane	0.5PPM	Gamma-Chlordane	0.5PPM
Beta-BHC	0.5PPM	Heptachlor	0.5PPM
Delta BHC	0.5PPM	Heptachlor Epoxide	0.5PPM
Dieldrin	0.5PPM	Lindane (Gamma-BHC)	0.5PPM
Endosulfan I	0.5PPM	Methoxychlor	0.5PPM

Aroclors 1016 and 1260 are added when samples are extracted for PCBs only, at a concentration of 5.0ppm and 10.0ppm respectively.

7.4 Surrogate Spike

Each sample, matrix spike, matrix spike duplicate and blank is spiked with surrogate compounds prior to extraction. The concentration in the following table exhibits the concentration in the spiking solution.

Compounds	Concentration Added
Decachlorobiphenyl	1.0ml of 0.2ug/ml
TCMX	1.0ml of 0.2ug/ml

7.5 Lab Control Spikes (LCS)

Blank spikes are extracted at the same frequency as matrix spikes using Na₂SO₄ as the matrix. LCS spiking levels are the same as matrix spike concentrations. If only PCBs are needed LCS is spiked with AR1260 and AR1016 only. Aroclors LCS level are the same as matrix spike concentrations.

8.0 SAFETY

8.1 Safety glasses, lab coat, and gloves should be worn at all times during extraction.

8.2 Label reagents, spikes, surrogate solutions with solution type, concentration, date, chemist's initials and shelf life.

9.0 POLLUTION PREVENTION

9.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

10.0 WASTE MANAGEMENT

10.1 Several wastes that GPL generates can be handled in a fairly routine manner. The process of describing the method for waste disposal of chemicals including standards and reagent solutions, and process waste, and samples is described in Standard Operating Procedures D.1 and D.2.

11.0 DEFINITIONS

11.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

12.0 REFERENCES

12.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition.

Uncontrolled Copy

SOP No: N.11

TITLE: Method 3520C Continuous Liquid-Liquid Extraction for Semivolatile Organics

1.0 SCOPE AND APPLICATION

- 1.1 This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics from water, wastewater, groundwater and TCLP extracts. Extracts produced by this method may be analyzed by Methods 8270.
- 1.2 This method also describes concentration techniques suitable for preparing the extract for the appropriate method.

2.0 DEFINITIONS

- 2.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

3.0 SUMMARY OF METHOD

- 3.1 A measured volume of sample, usually 1 liter, is placed into a continuous liquid-liquid extractor, adjusted to specific pHs and extracted with organic solvent for 18 ± 2 hours for each pH. The extracts are dried and concentrated.

4.0 INTERFERENCES

- 4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifact and/or interferences to sample analysis. All materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

5.0 APPARATUS AND MATERIALS

- 5.1 Continuous Liquid-Liquid Extractor (Equipped with joints and snapping clips).
- 5.2 Drying Column (A glass funnel with pyrex glass wool at the neck of the funnel, half filled with dry Na₂SO₄).

5.3 Kuderna-Danish (K-D) Apparatus

- Concentrator tube: 10ml, graduated (Kontes K-570050-1025 or equivalent). A ground-glass stopper is used to prevent evaporation of extracts.
- Evaporator flask: 500ml (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs.
- Snyder column: Three-ball macro (Kontes K-503000-0121 or equivalent).

5.4 Solvent extracted Boiling Chips, approximately 10/40 mesh (silicon carbide or equivalent).

5.5 Heated water Bath, with concentric ring cover, capable of temperature control ($\pm 5^{\circ}\text{C}$). The bath should be used in a hood.

5.6 Glass vials, 2ml capacity, with Teflon-lined crimp cap.

5.7 Narrow range pH Indicator Paper covering pH 0 to pH 6 and pH 11 to pH 13.

5.8 Rheostat controlled Heating Mantle.

5.9 1.0ml Gas-Tight Syringe.

5.10 One (1) Liter Class A Graduated Cylinder.

5.11 Pyrex Glass Wool.

5.12 Large disposable glass pipets.

6.0 REAGENTS

6.1 Reagent Water (water in which an interferant is not observed at the method detection limit of the compounds of interest).

6.2 Sodium Hydroxide Solution, 10N.

(ACS) Dissolve 40g NaOH in reagent water and dilute to 100ml.

6.3 Sodium Sulfate.

(ACS) Granular, Anhydrous (purified by heating at 400°C for 4 hours in a shallow tray).

6.4 Sulfuric Acid Solution (1:1).

Slowly add 50ml of H_2SO_4 (sp. gr. 1.84) to 50ml of reagent water.

6.5 Methylene Chloride (B & J GC2 grade or equivalent).

7.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

7.1 Samples are collected in 1 liter amber glass bottles which have been precleaned and certified to be free of target analytes. Samples are shipped in coolers packed with ice and stored at the lab in refrigerators at 2-6°C until extraction. The holding time for extraction is 7 days from sample collection.

8.0 PROCEDURE

- 8.1 Place a few boiling stones in a 500ml collecting flask and assemble continuous extractor.
- 8.2 Add 400ml of methylene chloride to the extractor.
- 8.3 Using a Class A graduated cylinder, rinse with methylene chloride and measure out 1 liter of sample, add 1.0ml surrogate spiking solution, transfer the content to the continuous extractor. Rinse out the sample bottle with 20-30ml methylene chloride and add to the extractor. Check the pH of the sample using wide range pH paper and adjust the pH to less than 2 using 1:1 H₂SO₄. Use 1.0 liter of deionized water to another extractor for the method blank preparation and analysis.
- 8.4 For the sample in each analytical batch selected for spiking, measure two additional 1.0 liter aliquots. Add 0.5 ml of the matrix spiking standard to each aliquot.
- 8.5 Add sufficient methylene chloride to the extractor to ensure proper operation and extract for 18 ± 2 hours.
- 8.6 Allow to cool. Adjust the pH of the remaining aqueous layer to greater than 11 using 10N NaOH. Extract the sample for 18 ± 2 hours. The extract is then ready to be dried and concentrated.
- 8.7 Assemble a Kuderna-Danish (K-D) Concentrator by attaching a 10ml concentrator tube to a 500ml evaporation flask.
- 8.8 Dry the extract by passing it through a drying column containing about 10cm anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Rinse the flask which contained the solvent extract with 20-30ml of methylene chloride and add to the column to complete the quantitative transfer.
- 8.9 Add one or two clean boiling chips to the flask and attach a three ball Snyder Column. Pre-wet the Snyder Column by adding approximately 1ml of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (65-70°C) until the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to

complete the concentration. When the apparent volume of liquid reaches about 10ml, remove the K-D apparatus from the water bath.

8.10 Disassemble K-D Apparatus and place the concentrator tube in a warm water bath (35°C) and evaporate the solvent volume to 1.0ml using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

9.0 QUALITY CONTROL

9.1 Method Blank

9.1.1 The method blank is a volume of deionized, laboratory water carried through the entire extraction. The method blank volume must be equal to 1.0 liter or the sample volumes being extracted.

9.1.2 Frequency of Method Blanks

Method blank extraction must be performed per each batch of samples, not to exceed 20 samples per batch.

9.2 Matrix Spike/Matrix Spike Duplicate

9.2.1 MS/MSD Frequency of Extraction

Matrix spike and matrix spike duplicate extraction must be performed per each batch of 20 samples or less. If the batch of samples are received periodically, then MS/MSD are extracted every 14 days.

9.3 Matrix Spiking Solutions

9.3.1 Matrix spikes

Matrix spike is prepared in acetone and 0.5ml is added to the designated samples. The mixture contains the following compounds at the given concentrations.(attachment 1 & 2).

LCS spiking levels are the same as those used to spike samples.

9.4 Surrogate Spike (SS)

Each sample, matrix spike, and blank are spiked with 1ml of the surrogate solution prior to extraction. The concentration in the following table exhibits the compounds and their concentration in the spiking solution.

Phenol-d5	200ug/ml
2,4,6-Tribromophenol	200ug/ml
2-Fluorophenol	200ug/ml
Nitrobenzene-d5	100ug/ml

Terphenyl-d14	100ug/ml
2-Fluorobiphenyl	100ug/ml

9.5 Lab Control Spike (LCS)

Blank spikes are extracted at the same frequency as method blank using deionized water as the matrix.

10.0 SAFETY

10.1 Safety glasses, lab coat, and gloves should be worn at all times during extraction.

10.2 Label reagents, spikes, surrogate solutions by identifying the solution, concentration, date, chemist's initials, and shelf life.

10.3 Waste Disposal

10.3.1 Chlorinated solvent waste should be put into a polyethylene safety can designated for methylene chloride. This will be disposed of by the hazardous waste coordinator.

10.3.2 Water waste can be poured down the drain after neutralizing it using H_2SO_4 or NaOH depending upon the pH of the waste.

11.0 WASTE MANAGEMENT

11.1 Several wastes that GPL generates can be handled in a fairly routine manner. The process of describing the method for waste disposal of chemicals including standards and reagent solutions, and process waste, and samples is described in Standard Operating Procedures D.1 and D.2.

12.0 POLLUTION PREVENTION

12.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

13.0 DEFINITIONS

13.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

14.0 REFERENCES

- Test Methods For Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Revision 3, December 1996.
- Navy Installation Restoration Laboratory Quality Assurance Guide, February 1996.
- USACE Shell Document, Version 1.0, November 1998.

ATTACHMENT 1

<u>ANALYTE</u>	<u>CONC</u>	<u>UNITS</u>
1,2,4 -Trichlorobenzene	200.0	ug/ml
1,2 -Dichlorobenzene	200.0	ug/ml
1,3-Dichlorobenzene	200.0	ug/ml
1,4 -Dichlorobenzene	200.0	ug/ml
2,4,5-Trichlorophenol	200.0	ug/ml
2,4,6-Trichlorophenol	200.0	ug/ml
2,4-Dichlorophenol	200.0	ug/ml
2,4 -Dimethyphenol	200.0	ug/ml
2,4 -Dinitrophenol	200.0	ug/ml
2,4 -Dinitrotoluene	200.0	ug/ml
2,6 -Dinitrotoluene	200.0	ug/ml
2-Chloronaphthalene	200.0	ug/ml
2 -Chlorophenol	200.0	ug/ml
2 -Methylnaphthalene	200.0	ug/ml
2 -Nitroaniline	200.0	ug/ml
2 -Nitrophenol	200.0	ug/ml
3,3'-Dichlorobenzidine	200.0	ug/ml
3 -Nitroaniline	200.0	ug/ml
4,6 -Dinitro-2Methylphenol	200.0	ug/ml
4 -Bromophenyl-Phenyl Ether	200.0	ug/ml
4 -Chloro-3-Methylphenol	200.0	ug/ml
4 -Chloroaniline	200.0	ug/ml
4 -Chlorophenyl Phenyl Ether	200.0	ug/ml
4 -Nitroaniline	200.0	ug/ml
4 -Nitrophenol	200.0	ug/ml
Acenaphthene	200.0	ug/ml
Acenaphthylene	200.0	ug/ml
Aniline	200.0	ug/ml
Anthracene	200.0	ug/ml
Azobenzene	200.0	ug/ml
Benzidine	200.0	ug/ml
Benzo(A)Anthracene	200.0	ug/ml
Bezo(A)Pyrene	200.0	ug/ml
Benzo(B)Fluoranthene	200.0	ug/ml
Benzo(G,H,I) Perylene	200.0	ug/ml
Benzo(K)Fluoranthene	200.0	ug/ml
Benzoic Acid	200.0	ug/ml
Benzyl Alcohol	200.0	ug/ml
Bis(2-Chloroethoxy)Methane	200.0	ug/ml

ATTACHMENT 2

<u>ANALYTE</u>	<u>CONC</u>	<u>UNITS</u>
Bis (2-Chloroethyl)Ether	200.0	ug/ml
Bis(2-Chloroisopropyl)Ether	200.0	ug/ml
Bis(2-Ethylhexyl)Phthalate	200.0	ug/ml
Butyl Benzyl Phthalate	200.0	ug/ml
Carbazole	200.0	ug/ml
Chrysene	200.0	ug/ml
Di-N-Butylphthalate	200.0	ug/ml
Di-N-Octylphthalate	200.0	ug/ml
Dibenz(A,H)Anthracene	200.0	ug/ml
Dibenzofuran	200.0	ug/ml
Diethylphthalate	200.0	ug/ml
Dimethylphthalate	200.0	ug/ml
Fluoranthene	200.0	ug/ml
Fluorene	200.0	ug/ml
Hexachlorobenzene	200.0	ug/ml
Hexachlorobutadiene	200.0	ug/ml
Hexachlorocyclopentadiene	200.0	ug/ml
Hexachloroethane	200.0	ug/ml
Indeno(1,2,3-CD)Pyrene	200.0	ug/ml
Isophorone	200.0	ug/ml
M-Cresol	200.0	ug/ml
N-Nitroso-Di-N-Propylamine	200.0	ug/ml
N-Nitrosodimethylamine	200.0	ug/ml
N-Nitrosodiphenylamine	200.0	ug/ml
Naphthalene	200.0	ug/ml
Nitrobenzene	200.0	ug/ml
O-Cresol	200.0	ug/ml
P-Cresol	200.0	ug/ml
Pentachlorophenol	200.0	ug/ml
Phenanthrene	200.0	ug/ml
Phenol	200.0	ug/ml
Pyrene	200.0	ug/ml
Pyridine	200.0	ug/ml

SOP No: N.12

Title: Soil Extraction for Semivolatile Organics by Method 3550B (Sonication Extraction)

1.0 APPLICATION AND SCOPE

- 1.1 This method describes the procedure for extracting non-volatile and semi-volatile organic compounds from solids such as soils. Extracts are suitable for analysis by method 8270.

2.0 SUMMARY OF METHOD

- 2.1 Method 3550 describes both a high level and low level technique. This SOP is based on the low level version of method 3550B
- 2.2 A 30g sample is mixed with anhydrous sodium sulfate to form a free-flowing powder. This is solvent extracted three times using sonication. The extract is separated from the sample by filtration. The extract is ready for cleanup following concentration.

3.0 INTERFERENCES

- 3.1 Solvents, reagent, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. These materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

4.0 APPARATUS AND MATERIALS

- 4.1 Mortar Model 155 Grinder, Fisher Scientific Company, or an equivalent brand and model.
- 4.2 Ultrasonic cell disrupter, Heat Systems – Ultra Sonic, Inc., Model W-385 (475 watt) Sonicator or equivalent (power wattage must be a minimum of 375 with pulsing capability and No. 200 1/2" Tapped Disrupter Horn) plus No. 207 3/4" Tapped Disrupter Horn, and No. 419 1/8" Standard Tapped Microtip Probe.

- 4.3 Pasteur Glass Pipets, Disposable, 1ml
- 4.4 Beakers, 400ml
- 4.5 Filtration Apparatus
 - Conical funnel
 - Filter paper: Whatman No. 41 or equivalent
- 4.6 Kuderna-Danish (K-D) Apparatus
 - 4.6.1 Concentrator Tube: 10ml graduated (Kontes K-570050-1025 or equivalent)
 - 4.6.2 Evaporator Flask: 500ml (Kontes K-570001-0500 or equivalent).
 - 4.6.3 Snyder Column: Three-ball macro (Kontes K-503000-0121 or equivalent).
- 4.7 Solvent extracted Boiling Chips, approximately 10/40 mesh (Silicon Carbide or equivalent)
- 4.8 Heated Water Bath, with concentric ring cover, capable of temperature control ($\pm 5^{\circ}\text{C}$). The bath should be used in a hood.
- 4.9 Top-loading Analytical Balance, capable of accurately weighing 0.001 grams.
- 4.10 2ml and 12ml Vials and Caps
- 4.11 Glass Funnel
- 4.12 Nitrogen evaporation device equipped with a water bath that can be maintained at 30-35°C
- 4.13 Spatula, Stainless steel or Teflon
- 4.14 Syringe, 1ml
- 4.15 Large disposable glass pipets

5.0 REAGENTS

- 5.1 Sodium Sulfate; Anhydrous and reagent grade, heated at 400°C for 4 hours, cooled in a desiccator, and stored in a glass bottle. Baker anhydrous powder, catalog #73898, or equivalent.
- 5.2 Methylene Chloride: acetone (1:1, V:V)

5.3 Methylene Chloride: (B&J GC2 grade, or equivalent)

6.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

6.1 Samples are collected in appropriately sized glass bottles which have been precleaned and certified to be free of target analytes. Samples are shipped in coolers packed in ice and stored at the lab in refrigerators at 2-6°C until extraction. The holding time for extraction is 14 days from sample collection.

7.0 PROCEDURE

7.1 Sample Handling

7.1.1 Sediment/soil samples: Decant and discard any water layer on a sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

7.2 The following step should be performed rapidly to avoid loss of the more volatile extractables. Weigh approximately 30g of sample into a 400ml beaker. Record the weight to the nearest 0.01g. Add 1ml of surrogate standards to all samples, spikes, and blanks. For the sample in each analytical batch selected for spiking, add 0.5ml of the matrix spiking standard. The amount added for the surrogate compounds should result in a final concentration of 3300µg/Kg of each base/neutral analyte and 6600µg/Kg of each acid analyte in the extract to be analyzed. All surrogate and spiking solutions must be added to the samples before any processing occurs. To ensure an efficient extraction, all samples must be dried by mixing with 60g of anhydrous sodium sulfate using a spatula. The sample should be free-flowing at this point. Immediately add 100ml of 1:1 methylene-chloride: acetone.

7.3 Sonicator Tuning Procedure

The sonicator is to be tuned weekly, at a minimum, and whenever any of the components are changed or, at the discretion of the analyst. The following procedure is to be followed for tuning.

7.3.1 Turn the power output knob to zero.

7.3.2 Turn the sonicator mode switch to continuous mode.

7.3.3 Turn the sonicator power switch on.

7.3.4 Press and hold the sonicator tune switch on with one hand for the Tekmar sonicator; use the other hand to turn the power output knob or the sonicator tuning knob as follows. The tuning switch stays on by itself on the Misonix sonicators. The tuning knob for the Tekmar sonicator and Misonix sonicators is on the front panel of the generator.

- 7.3.5 Slowly turn the power output knob toward 10 while watching the output power meter reading. When the meter reading reaches 50%, turn the tuning knob until the minimum meter deflection is observed. The minimum deflection can be determined by observing when the needle starts to go back up when turning the tuning knob in either direction.
- 7.3.6 Proceed again with step (6.5.5) until reaching output level 10 (output level 5 for microtips), and adjust the tuning knob to achieve the minimum deflection. At this point, the sonicator is considered tuned and operational provided that minimum deflection is achieved and the sonicator power output meter reads less than 25% at the minimum.
- 7.3.7 If the tuning criteria can't be met, check for loose cable connections or loose horn to converter connection and tighten as necessary. Retune. If the tuning criteria still can't be met, try replacing horn and converter with backup unit. Retune. If the tuning criteria still can't be met, notify the supervisor immediately so that repairs and/or a loaner can be obtained. Place a sign on the sonicator stating "Out of order, do not use".
- 7.3.8 Release the sonicator tune switch, switch the sonicator mode switch to pulsed mode and switch the sonicator off.
- 7.3.9 Record the tuning in the appropriate sonicator maintenance logs and weekly checklist.
- 7.4 Place the bottom surface of the tip of the #207 3/4" disrupter horn approximately 1/2" below the surface of the solvent, above the sediment layer.
- 7.5 Sonicate for 3 minutes with the output control knob set at 10, the mode switch set on pulse, and the percent-duty cycle knob set at 50%. DO NOT use a microtip probe.
- 7.6 Decant and filter extracts through Whatman No. 41 filter paper, or centrifuge and decant extraction solvent.
- 7.7 Repeat the extraction two more times using two additional 100ml portions of solvent. Decant and filter the extraction solvent after each sonication. On the final sonication, pour the entire sample into the conical funnel and rinse with extraction solvent.
- 7.8 Assemble a Kuderna-Danish (K-D) Concentrator by attaching a 10ml concentrator tube to a 500ml evaporative flask.
- 7.9 Dry the extract by passing it through a drying column containing approximately 10cm of anhydrous Sodium Sulfate. Collect the dried extract in a K-D Concentrator. Wash the extractor flask and Sodium Sulfate Column with 100-125ml of extraction solvent to complete the quantitative transfer.

- 7.10 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder Column. Pre-wet the Snyder Column by adding approximately 1ml methylene chloride to the top. Place the K-D apparatus in a hot water bath (65-70°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-15 minutes. At the proper rate of distillation the balls of the column will actively chatter, however, the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1ml, remove the K-D apparatus and allow to drain and cool for at least 10 minutes.
- 7.11 Remove the Snyder Column and rinse the flask and lower joints into the concentrator tube with 1-2ml of methylene chloride. The extract may be further concentrated and adjusted to 1.0ml using methylene chloride.
- 7.12 Transfer the concentrated extract to a clean 2ml vial. Seal the vial with a Teflon-lined lid and mark the level on the vial. Label with the sample number and fraction and store in the dark at 4°C.

8.0 QUALITY CONTROL

8.1 Method Blank

8.1.1 A method blank for a soil sample consists of purified solid matrix (Na_2SO_4) which is carried through the entire extraction. The method blank weight must be approximately equal to the sample weights being processed.

8.1.2 Method Blank

A method blank extraction must be performed with each batch (samples of the same or similar matrix prepared together) not to exceed twenty samples excluding QC sample).

8.2 Matrix Spike/Matrix Spike Duplicate

8.2.1 Matrix Spike/Matrix Spike Duplicate frequency of extraction

A matrix spike and matrix spike duplicate must be performed once:

- each 20 field samples in a batch, or
- each 14 calendar day period during which field samples in a batch are received.

Matrix spike QC sample must contain all target analytes of interest.

8.3 Surrogate Spike (SS)

Each sample, matrix spike, and blank are spiked with 1ml of the surrogate solution prior to extraction. The concentration in the following table exhibits the compounds and their concentration in the spiking solution.

Phenol-d5	200ug/ml
2,4,6-Tribromophenol	200ug/ml
2-Fluorophenol	200ug/ml
Nitrobenzene-d5	100ug/ml
Terphenyl-d14	100ug/ml
2-Fluorobiphenyl	100ug/ml

8.4 Laboratory Control Sample

LCS extraction must be performed with each batch of samples processed. The LCS is spiked with the same analytes at the same concentrations as the matrix spike sample.

9.0 SAFETY

9.1 Safety glasses, lab coat, and gloves should be worn at all times during extraction.

9.2 Label reagents, spikes, surrogate solutions with solution type, concentration, date, chemist's initials and shelf life.

10.0 POLLUTION PREVENTION

10.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

11.0 WASTE MANAGEMENT

11.1 Several wastes that GPL generates can be handled in a fairly routine manner. The process of describing the method for waste disposal of chemicals including standards and reagent solutions, and process waste, and samples is described in Standard Operating Procedures D.1 and D.2.

12.0 DEFINITIONS

12.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

13.0 REFERENCES

- Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, Revision 2, December 1996.

- Navy Installation Restoration Laboratory Quality Assurance Guide, February 1996.

- USACE Shell Document, Version 1.0, November 1998.

ATTACHMENT 1

<u>ANALYTE</u>	<u>CONC</u>	<u>UNITS</u>
1,2,4-Trichlorobenzene	200.0	ug/ml
1,2-Dichlorobenzene	200.0	ug/ml
1,3-Dichlorobenzene	200.0	ug/ml
1,4-Dichlorobenzene	200.0	ug/ml
2,4,5-Trichlorophenol	200.0	ug/ml
2,4,6-Trichlorophenol	200.0	ug/ml
2,4-Dichlorophenol	200.0	ug/ml
2,4-Dimethylphenol	200.0	ug/ml
2,4-Dinitrophenol	200.0	ug/ml
2,4-Dinitrotoluene	200.0	ug/ml
2,6-Dinitrotoluene	200.0	ug/ml
2-Chloronaphthalene	200.0	ug/ml
2-Chlorophenol	200.0	ug/ml
2-Methylnaphthalene	200.0	ug/ml
2-Nitroaniline	200.0	ug/ml
2-Nitrophenol	200.0	ug/ml
3,3'-Dichlorobenzidine	200.0	ug/ml
3-Nitroaniline	200.0	ug/ml
4,6-Dinitro-2Methylphenol	200.0	ug/ml
4-Bromophenyl-Phenyl Ether	200.0	ug/ml
4-Chloro-3-Methylphenol	200.0	ug/ml
4-Chloroaniline	200.0	ug/ml
4-Chlorophenyl Phenyl Ether	200.0	ug/ml
4-Nitroaniline	200.0	ug/ml
4-Nitrophenol	200.0	ug/ml
Acenaphthene	200.0	ug/ml
Acenaphthylene	200.0	ug/ml
Aniline	200.0	ug/ml
Anthracene	200.0	ug/ml
Azobenzene	200.0	ug/ml
Benzidine	200.0	ug/ml
Benzo(A)Anthracene	200.0	ug/ml
Benzo(A)Pyrene	200.0	ug/ml
Benzo(B)Fluoranthene	200.0	ug/ml
Benzo(G,H,I)Perylene	200.0	ug/ml
Benzo(K)Fluoranthene	200.0	ug/ml
Benzoic Acid	200.0	ug/ml
Benzyl Alcohol	200.0	ug/ml
Bis(2-Chloroethoxy)Methane	200.0	ug/ml

ATTACHMENT 2

<u>ANALYTE</u>	<u>CONC</u>	<u>UNITS</u>
Bis(2-Chloroethyl)Ether	200.0	ug/ml
Bis(2-Chloroisopropyl)Ether	200.0	ug/ml
Bis(2-Ethylhexyl)Phthalate	200.0	ug/ml
Butyl Benzyl Phthalate	200.0	ug/ml
Carbazole	200.0	ug/ml
Chrysene	200.0	ug/ml
Di-N-Butylphthalate	200.0	ug/ml
Di-N-Octylphthalate	200.0	ug/ml
Dibenz(A,H)Anthracene	200.0	ug/ml
Dibenzofuran	200.0	ug/ml
Diethylphthalate	200.0	ug/ml
Dimethylphthalate	200.0	ug/ml
Fluoranthene	200.0	ug/ml
Fluorene	200.0	ug/ml
Hexachlorobenzene	200.0	ug/ml
Hexachlorobutadiene	200.0	ug/ml
Hexachlorocyclopentadiene	200.0	ug/ml
Hexachloroethane	200.0	ug/ml
Indeno(1,2,3-CD)Pyrene	200.0	ug/ml
Isophorone	200.0	ug/ml
M-Cresol	200.0	ug/ml
N-Nitroso-Di-N-Propylamine	200.0	ug/ml
N-Nitrosodimethylamine	200.0	ug/ml
N-Nitrosodiphenylamine	200.0	ug/ml
Naphthalene	200.0	ug/ml
Nitrobenzene	200.0	ug/ml
O-Cresol	200.0	ug/ml
P-Cresol	200.0	ug/ml
Pentachlorophenol	200.0	ug/ml
Phenanthrene	200.0	ug/ml
Phenol	200.0	ug/ml
Pyrene	200.0	ug/ml
Pyridine	200.0	ug/ml

**UNCONTROLLED DOCUMENT
AND
BUSINESS CONFIDENTIAL**

Effective Date: August 2007
Version No: 15
Initiated By: GPB
Approved By: GPB
(Signature)

SOP No: P.5
TITLE: SOP for Method SW8270C
GC/MS Analysis of Semivolatile Organics

1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure describes the methodology used to determine the concentration of semivolatile organic compounds in extracts prepared from all types of solid waste matrices, soils, and water. Table 1 indicates compounds typically determined by this method and lists the method reporting limit for each in water or soil. Table 2 lists the Appendix IX compounds which can be determined using 8270C methodologies. Table 3 also lists additional compounds that can be determined using Method 8270C. Table 4 lists the semivolatile PAH compounds analyzed by low concentration.

2.0 SUMMARY OF METHOD

2.1 Prior to using this method, the samples should be prepared for chromatography using the appropriate sample preparation methods.

3.0 INTERFERENCE

3.1 GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation of the samples and initiate corrective action to eliminate the problem.

3.2 Contamination by carryover can occur whenever high-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between each use.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph/Mass Spectrometer

Hewlett Packard 5970 and 5972 MSD System equipped with an autosampler is utilized. The system is a complete instrument composed of two modules: a Controller and a main frame GCMS.

4.1.1 Data System

Each GC/MS system is attached to a dedicated 486 or pentium based computer equipped with Enviroquant software for automated acquisition and processing. The software generates total and extracted ion profiles of each compound and is capable of performing library searches on spectra using a full EPA/NIH Mass Spectral Data Library. Each system is attached to an internal laboratory computer network for additional data processing, storage and archiving.

4.2 Columns

A 30 meter, DB-5ms or equivalent (5% phenyl methyl silicon) column is used.

4.3 Syringe: 10 μ l

5.0 GC CONDITION

5.1 The following GC parameters are suggested:

-	Injection port temp:	250°C
-	Interface temp:	300°C
-	Initial temp:	40°C
-	Initial time:	4 min.
-	Rate at	15°C per min.
-	Final temp:	310°C
-	Final time:	20 min.
-	Equilibrium time:	.5 min.
-	Septum purge flow on at	.5ml/min.
-	Septum purge flow on at	.5 min.
-	Flow rate at about	0.7ml/min.
-	Splitless flow about	50ml/min.
-	Injection type:	"Splitless"
-	Autosampler injection mode:	"Fast"
-	Sample Volume	1 μ l

5.2 GC/MS Condition

5.2.1 Scanning from 35 to 500amu in less than 1 second, using 70 volt (nominal) electron energy in the Electron Impact Ionization mode.

5.2.2 GCMS Tuning - to determine the system performance, 50ng of decafluorotriphenylphosphine (DFTPP) is injected and the mass spectrum is evaluated. Verify that the MS meets standard mass spectral abundance criteria. The tune standard must be analyzed at the beginning of each analytical shift and every 12 hours of continuous analysis. Evaluate the ion abundances using any of the following:

- Use one scan at the apex;
- Use one scan either directly preceding or following the apex;
- Use the mean of the apex and the preceding and following scans;
- Use the average across the entire peak.

Background correction should be employed only for the purpose of correcting for instrument background ions. If any single approach fails, re-inject the DFTPP standard or retune the instrument. The following criteria must be met before any further analysis is performed.

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

The GC/MS tuning standard should also be used to assess GC column performance and injection inertness. Degradation of DDT to DDE and DDD should not exceed 20%. Benzidine and Pentachlorophenol should be present at their normal responses, and no peak tailing should be visible. The acceptance criteria for the peak tailing factor for benzidine is <3.0 and pentachlorophenol is <5.0. For samples received from the State of California, the following documentation will be provided: percent breakdown for DDT and tailing factors for benzidine and pentachlorophenol. The calculation of peak tailing factors is illustrated in Figure 1.

5.3 Calibration

5.3.1 Upon satisfactory completion of DFTPP analysis, six levels of calibration standards at 10,20, 50, 80, 120 and 160ng are analyzed. Calibration standards must contain the following internal and surrogate standards.

5.3.1.1 Internal Standards - the following Internal Standards at 40mg/ml must be present in the Calibration Standard.

- 1,4-dichlorobenzene-d₄
- Naphthalene-d₈
- Acenaphthene-d₁₀
- Phenanthrene-d₁₀
- Chrysene-d₁₂
- Perylene-d₁₂

5.3.1.2 The following Surrogate Standards at 10,20, 50, 80, 120 and 160ng are analyzed.

- Phenol-d₆
- 2-Fluorophenol
- 2,4,6-Tribromophenol
- nitrobenzene-d₅
- 2-fluorobiphenyl
- p-terphenyl-d₁₄

5.4 After analyzing the initial Calibration Standard average RF should be calculated for each compound. The percent relative standard deviation (%RSD) should be equal or less than 15% for each compound. However, Calibration Check Compounds (CCC) must have a % RSD less than or equal to 30%. It is likely that some analytes may exceed the 15% acceptance limit for the %RSD. In those instances the following steps should be considered.

- If the %RSD is greater than 15%, the analyst should review the results (area counts, response factors, standard concentrations) for those analytes to ensure that the problem is not associated with a single standard. If so, that standard should be reanalyzed and RSD recalculated. Replacing of standard may be necessary in some cases.
- It may also be necessary to narrow the calibration ranges to achieve a better linearity. The changes to the upper end of the calibration will affect the need to dilute samples above that range, while the change to the lower calibration will affect the sensitivity and elevate the reporting limit of the method. When considering dropping one or two points to narrow the calibration range, a minimum of five points is required.

After visual inspection of the calibration points for each analytes and for those analytes that did not meet the %RSD criteria, at the discretion of the analyst it should consider to employ a linear regression that does not through the origin or a quadratic (second order) model. A quadratic model requires six standards. Linearity is presumed acceptable if the correlation coefficient (r) is equal to or greater than 0.995 or the coefficient of determination (r^2) is equal to or greater than 0.99.

The relative retention times of each compound in each calibration run should agree within 0.06 relative time units. System performance check compounds must have a minimum RF of 0.05. All other compounds must have minimum RF of 0.01. The CCC and SPCC compounds are listed as follows:

<u>SPCC</u>	<u>CCC</u>
N-nitroso-di-n-Propylamine	Acenaphthene
Hexachlorocyclopentadiene	1,4-Dichlorobenzene
2,4-Dinitrophenol	N-Nitrosodiphenylamine
4-Nitrophenol	Di-n-octylphthalate
	Fluoranthene
	Benzo(a)pyrene
	4-Chloro-3-methylphenol
	2,4-Dichlorophenol
	2-Nitrophenol
	Phenol
	Pentachlorophenol
	2,4,6-Trichlorophenol

- 5.5 Prior to use for sample analysis, the acceptability of initial calibration curve must be verified through analysis of a calibration verification solution obtained from a second source. Calibration verification must meet the same acceptance criteria used for continuing calibration (daily) outlined in Section 5.7.
- 5.6 Internal Standard responses and retention times in the calibration standard, blanks and samples must be evaluated. Retention time variation for any internal standard in the calibration standard must be less than 30 seconds from that in the midpoint standard level of the most recent initial calibration. All other samples and QC samples must be compared to the daily CCV. Variation of the areas for internal standards must not vary by more than a factor of two or -50% to +100%.
- 5.7 A continuing calibration (daily) at 50ng concentration containing all semivolatile analytes, including all required surrogates must be analyzed every 12 hours. The following criteria must be met before sample analysis starts:
- System Performance Check Compounds (SPCCs): A system performance check must be made every 12 hour shift. For each SPCC compound in the daily calibration a minimum response factor of 0.05 must be obtained.
 - All other non SPCC must have RF of 0.01.
 - Percent difference for all CCC should be less than 20%. Non CCC should have less than 20% except up to 8 poor performing compounds could have 40%. If % drift for any poor performing compound is greater than -35% (lost sensitivity) and that compound is present in the sample, the sample should be re-analyzed with acceptable calibration standard.

For DOD projects, non CCCs shall meet less than 25%D criteria.

$$\% \text{Difference} = \frac{\text{RRF}_c - \overline{\text{RRF}}_i}{\overline{\text{RRF}}_i} \times 100$$

where:

RRF_c = Relative response factor from continuing calibration standard

RRF_i = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria

If the % difference criteria for each CCC and non CCC is not met corrective action must be taken, and if after corrective action criteria were not met, a new initial calibration must be generated.

- Retention time for Internal Standards should not drift more than ± 30 seconds compared from that in the mid point standard of the most recent initial calibration.

6.0 METHOD DETECTION LIMITS

6.1 Method detection limits for this method are listed in the GPL Laboratory Method Detection Limit and Reporting Limit table.

7.0 METHOD PERFORMANCE

7.1 The MDL concentrations listed in the GPL MDL/RL table are generally obtained using organic-free reagent water. Results were also obtained by extracting seven spiked replicates the same way as the samples and analyzing them. MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. Precision and accuracy studies are performed once a year at a minimum. Single operator precision, overall precision and method accuracy were found to be directly related to the concentration of the parameter.

8.0 SAMPLE ANALYSIS

8.1 To 1ml of BNA extracts, 20uL of 2000mg/ml internal standard is added to each mix. One ul of this extract is injected into the GCMS using the same parameters as the calibration standard. All samples must be injected within a 12 hour period starting with analysis of DFTPP.

8.2 Qualitative Analysis

8.2.1 Two criteria must be satisfied to verify compound identification.

8.2.1.1 Elution of sample component at the same GC relative retention time (RRT) ± 0.06 units.

8.2.1.2 Correspondence of the sample component and the standard component mass spectrum.

All ions present in the standard mass spectrum at a relative intensity greater than 10% must be present in the sample spectrum. The relative intensity of these ions must agree $\pm 20\%$ between the standard and sample spectra.

8.2.2 Library Search

8.2.2.1 When required, a search may be performed for the purpose of tentative identification. Only after visual comparison of sample spectra with the nearest library searches will the analyst assign a tentative identification. The analyst should use an approach similar to the 5 step identification listed for method SW8270 (pp-20), Dec. 1996.

8.3 Quantitative Analysis

8.3.1 When a compound is identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation is based on the internal standard technique. The internal standard nearest the retention time of that given analyte, shall be used.

9.0 CALCULATIONS

The following information should be used throughout the quantitation process.

9.1 Response Factor

$$RF = \frac{(A_x C_{is})}{(A_{is} C_x)}$$

where:

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 (mg/ml)

C_{is} = Concentration of the specific internal standard (mg/ml)

9.2 % RSD and % Difference

$$\% \text{ RSD} = \frac{\text{Standard Deviation}}{\text{Average RF}} \times 100$$

$$\% \text{ Difference} = \frac{\text{AvgRF}_i(\text{Init. Calib.}) - \text{RF}_c(\text{Cont. Calib.})}{\text{Avg RF}_1} \times 100$$

9.3 Sample Calculation

$$\text{Water Concentration} = \frac{(A_x) (I_s) (V_t) (\text{Dil})}{\text{ug/l} (A_{is}) (\text{Avg RF}) (V_o) (V_i)}$$

$$\text{Soil Concentration} = \frac{(A_x) (I_s) (V_t) (\text{Dil})}{\text{ug/kg} (A_{is}) (\text{Avg RF}) (V_i) (W_s) (D)}$$

where:

I_s = Amount of internal standard injected (ng)

V_t = Volume of total extract

V_o = Initial volume

V_i = Volume of extract injected (ml)

W_s = Weight of sample extracted (G)

D = Percent solids

Dil = Dilution applied

Alternatively, the regression line fitted to the initial calibration may be used for determination of the extract concentration.

10.0 QUALITY CONTROL

10.1 Blank analysis - A method blank is analyzed with each analytical batch to examine the interferences and contamination from extraction and analysis. Concentration of the target compounds in the blank must be less than or equal to ½ of the reporting limit. If the above criteria are not met, re-injection or re-extraction of the blank and the samples associated with the blank if sufficient sample exists and holding times have not elapsed.

- 10.2 Surrogate analysis - Surrogate standard determinations are performed on all samples and blanks.

For DOD projects, the QSM control limits policy shall be followed. First, the project-specific limits will be used if available. If not, QSM limits and policy shall be used, if available. Other wise, the internal limits shall be used.

Policy for non-DOD projects:

- 10.2.1 If any surrogate recoveries in the method blank in either the BN or acid fraction are outside the surrogate spike recovery limits, corrective action should be taken. First, re-inject the blank - if no improvement is observed, reextraction should be considered. The blank and all samples associated with the blank should be re-extracted if enough sample exists and holding time have not elapsed.
- 10.2.2 If more than one BN or acid fraction in the sample are outside the surrogate spike recovery limits, corrective action should be taken. First, re-inject the sample. If the surrogate recoveries fail to meet the criteria after re-analysis, re-extract the sample. If the surrogate compound recoveries meet acceptance criteria in the re-extraction/reanalysis, submit data from the re-extraction/reanalysis only if it met holding times. If not, submit both data.
- 10.2.3 If any surrogate in the sample has a recovery limit less than 10 percent, first re-analyze the sample. If sample does not meet recovery limit, re-extract and re-analyze the sample if enough sample exists and holding time have not elapsed.
- 10.2.4 If the surrogate compound recoveries fail to meet the acceptance criteria in the re-extraction/reanalysis sample, then submit data from both analyses.
- 10.2.5 If the surrogate recoveries in the sample have been affected by obvious matrix interferences and reanalysis of the extract may harm the analytical system, report the data and explain in the case narrative.
- 10.3 Matrix Spikes - A matrix spike and matrix spike duplicate are analyzed with each batch of up to 20 samples of the same matrix processed together. MS/MSD recoveries should be within the in-house established control limits. Control limits are established based on minimum 20-30 historical data points that span between 6 months to a year. The results from the MS/MSD normally would not be used to determine the validity of the entire batch. The poor performers in the spike may indicate a problem with sample composition and shall be reported to the client as such.

If matrix spike recovery does not meet the Control Limit criteria, the supervisor must asses the data to determine whether the spike results are attributed to a

matrix affect, or the results of other problem in the analytical process. If all QC elements, which are not affected by the sample matrix, are in control (e.g. Method Blank, LCS, Calibration check), and if there is no evidence the spiking may have been improperly performed, the poor spike recovery may be attribute to matrix affect. In this case corrective action is not required.

If any of the batch QC elements which are not affected by sample matrix are out of control, or if there is any evidence that spiking may have been improperly performed, the matrix spike sample must be reprocess. MS/MSD pair are spiked the same as the LCS components.

For DOD projects, DOD QSM matrix spike control limits policy shall be followed.

10.4 Lab Control Sample

10.4.1 A laboratory control sample is extracted and analyzed routinely with each extracted batch. Calculated concentrations are compared with the amount added and results are used to demonstrate that the laboratory process for sample preparation and analysis is in control. Generation of in house statistical control limits must be based on minimum 30 data points. Data set used to generate control limits must have been generated using the same analytical procedure and data sets must not be selectively included or excluded.

Each analyte in the LCS must be evaluated against the Control Limits and Marginal Exceedance limit. If any LCS is outside the Control Limit, it should be also compared to the Laboratory Marginal Exceedance Limits to ensure that it does not exceed. If a single analyte in the LCS exceeded the Marginal Exceedance Limit, the LCS had failed and corrective action needs to be initiated.

If the LCS has more than the allowable number of Marginal Exceedance, the LCS has failed and corrective action needs to be initiated.

The corrective action for failed LCS is based on professional judgment in conjunction with matrix spike and surrogate recoveries in the same batch. If after checking the associated QC's it was determined by the section supervisor or manager that the LCS had failed, all affected samples associated with the out of control LCS should be reprocessed and re-analyzed.

If samples cannot be reprocessed due to lack of sample volume or the holding times has lapsed, the results should be reported with appropriate flag. The report case narrative must include a discussion of the failed LCS, and its impact on the data quality.

When LCS is spiked with large number of analytes, the laboratory should add up total number of exceedances for the LCS based on the number of analyte spiked in the LCS. The total of exceedance should be compare with the allowable number from the following chart.

Number of analyte in the LCS	Allowable Number of Marginal Exceedances of LCS
< 11	0
11-30	1
31-50	2
51-70	3
71-90	4
>90	5

Since a large amount of GPL's clients most requested compound list are fairly long (> 20 compounds), the laboratory will spike all the reportable compounds as specified in the clients project. At a minimum, 16 compounds from the list will be selected and used to verify if the LCS meets the required QC requirements. However, the laboratory should ensure that all compounds in the target list are used to verify the LCS criteria over a period of two years.

10.5 Internal Standard Recoveries and Retention times - when internal standard recoveries (-50 to +100%) or retention time requirements, (0.06 RRT) are not satisfied, sample is reinjected and reanalyses results are submitted if they are acceptable. If both analyses are out of criteria, then both results are submitted.

10.6 Sample Collection and Preservation.

10.6.1 Water samples may be collected in 1L (or one quart) amber glass container. Soil samples may be collected in glass containers or closed end tubes.

10.6.2 All samples must be iced or refrigerated at 4°C (± 2°C) from the time of collection until extraction.

10.6.3 Extracts of water and soil/sediment samples must be analyzed within 40 days following extraction.

10.7 Dilutions are performed on samples when the concentrations of target analytes exceed the calibration range. Additional IS must be added to the diluted extract to maintain the required IS concentration. Dilution of extracts should result in analysis with the highest concentration target analyte in the upper half of the calibration range.

10.8 QC limits for MS/MSD, LCS and surrogates are established semi-annually for internal use.

11.0 LOW CONCENTRATION

11.1 When requested, low concentration can be analyzed using a modified 8270C method. Lower detection limit is achieved by analyzing low level standards at 2-4ul injection. Instrument is optimized for high masses and the multiplier is

enhanced to achieve the desired increased sensitivity. The instrument (the source) and injection port need to be scrupulously clean when analyzing low level semivolatile compounds. Sample matrix should be reasonably free of interferences to allow low level detection of semivolatile compounds. Extraction procedures for water and soil matrices are the same as methods 3510, 3520, 3540 and 3550 except for surrogate and matrix spike standards levels added to the sample and quality control samples before extraction. Surrogate standard is added at 1.0ml of 0.5ug/ml of all compounds. Matrix spike and lab control spike consist of 1.0ml of all compounds at 0.5ug/ml concentrations. Both soil and water matrices receive the same amount of surrogate and matrix spike standards. Sample and quality control extracts are spiked with 4.0ug/ml internal standard before injection.

- 11.2 Seven level calibration standards are analyzed at a concentration 0.1, 0.5, 1.0, 2.5, 5.0, 10, and 20ug/ml. All Method 8270C QC criteria's applies to the low level 8270 analysis.

12.0 MS SIM ANALYSIS

- 12.1 When requested, low concentration target analyte can be analyzed using Selective Ion Monitoring (SIM) mode. Selective Ion Monitored (SIM) mode is a data acquisition technique in which only a few selected ion fragments are monitored in order to obtain maximum selectivity. Extraction procedures for water and soil matrices are the same as methods 3510, 3520, 3540 and 3550 except for surrogate and matrix spike standards levels added to the sample and quality control samples before extraction. Surrogate standard is added at 1.0ml of 0.5ug/ml. Matrix spike and lab control spike consist of 1.0ml of all compounds at 0.5ug/ml concentrations. Both soil and water matrices receive the same amount of surrogate and matrix spike standards. Sample and quality control extracts are spiked at 5.0ug/ml internal standard before injection.
- 12.2 Seven level calibration standards are analyzed at a concentration 0.1, 0.5, 1.0, 2.5, 5.0, 10, and 20ug/ml. All Method 8270C QC criteria apply to the SIM analysis.

13.0 SAFETY

- 13.1 Always wear safety glasses or a shield for eye protection, gloves and lab coat.
- 13.2 Observe proper mixing when working with reagents and chemicals. Preparation of samples and standards should be handled under the hood.
- 13.3 A reference file of material data handling sheet is available to all personnel involving in these analyses.

14.0 POLLUTION PREVENTION

- 14.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

15.0 WASTE MANAGEMENT

15.1 Several wastes that GPL generates can be handled in a fairly routine manner. The process of describing the method for waste disposal of chemicals including standards and reagent solutions, and process waste, and samples is described in Standard Operating Procedures D.1 and D.2.

16.0 DEFINITIONS

16.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

17.0 REFERENCES

- Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW846, 3rd Edition, Revision 3, December 1996
- DOD Quality System Manual, Final Version 3, March 2005

TABLE 1

Target Compound List (TCL) and
Contract Required Quantitation Limits (CRQL)*

<u>Semivolatiles</u>	<u>CAS Number</u>	<u>Water ug/L</u>	<u>Quantitation Limits** Low Soil/Sediment^b ug/Kg</u>
Benzaldehyde	100-52-7	10	330
Phenol	108-95-2	10	330
bis (2-Chloroethyl) ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
2-Methylphenol	95-48-7	10	330
bis (2-Chloroisopropyl) ether	108-60-1	10	330
Acetophenone	98-86-2	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
bis (2-Chloroethoxy) methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline***	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
Caprolactam	105-60-2	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene***	77-47-4	10	330
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	20	660
1,1'-Biphenyl	92-52-4	10	330
2-Chloronaphthalene	91-58-7	10	330
2-Nitroaniline***	88-74-4	10	330

TABLE 1 (Cont)

Target Compound List (TCL) and
Contract Required Quantitation Limits (CRQL)*

<u>Semivolatiles</u>	<u>CAS Number</u>	<u>Water ug/L</u>	<u>Quantitation Limits** Low Soil/Sediment^b ug/Kg</u>
Dimethylphthalate	131-11-3	10	330
Acenaphthylene	208-96-8	10	330
2,6-Dinitrotoluene	606-20-2	10	330
3-Nitroaniline***	99-09-2	10	330
Acenaphthene	83-32-9	10	330
2,4-Dinitrophenol***	51-28-5	20	660
4-Nitrophenol***	100-02-7	20	660
Dibenzofuran	132-64-9	10	330
2,4-Dinitrotoluene	121-14-2	10	330
Diethylphthalate	84-66-2	10	330
4-Chlorophenyl-phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	10	330
4-Nitroaniline***	100-01-6	10	330
4,6-Dinitro-2-methylphenol***	534-52-1	20	660
N-nitrosodiphenylamine	86-30-6	10	330
4-Bromophenyl-phenylether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Atrazine***	1912-24-9	10	330
Pentachlorophenol***	87-86-5	20	660
Phenanthrene	85-01-8	10	330
Anthracene	120-12-7	10	330
Carbazole	86-74-8	10	330
Di-n-butylphthalate	84-74-2	10	330
Fluoranthene	206-44-0	10	330
Pyrene	129-00-0	10	330
Butylbenzylphthalate	85-68-7	10	330
3,3'-Dichlorobenzidine***	91-94-1	20	660
Benzo(a)anthracene	56-55-3	10	330
Chrysene	218-01-9	10	330
bis (2-Ethylhexyl) phthalate	117-81-7	10	330
Di-n-octylphthalate	117-84-0	10	330
Benzo(b)fluoranthene	205-99-2	10	330

TABLE 1 (Cont)

Target Compound List (TCL) and
Contract Required Quantitation Limits (CRQL)*

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

^b Medium soil/sediment contract required quantitation limits (CRQL) for semivolatile TCL compounds are 60 times the individual low soil/sediment CRQL.

* Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

** Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment. Calculated on dry weight basis as required by the contract, will be higher.

*** Poor performers/sporadic marginal failures. Poor extraction efficiency, tendency to decompose, or poor chromatographic behavior.

TABLE 2

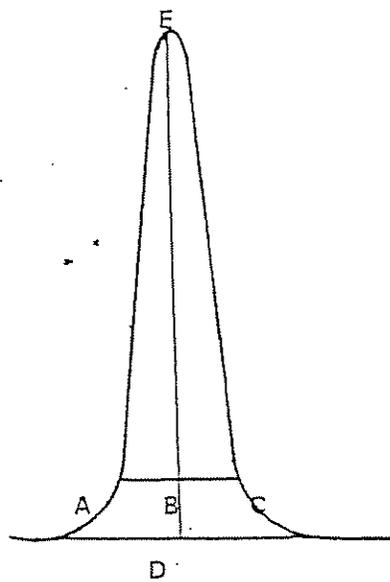
Appendix IX Compounds

1.	2-Acetylaminofluorene	41.	Pyridine
2.	4-Aminobiphenyl	42.	Safrole
3.	Aniline	43.	1,2,4,5-Tetrachlorobenzene
4.	Chlorobenzilate	44.	2,3,4,6-Tetrachlorophenol
5.	m-Cresol	45.	Thionazin
6.	Dimethoate	46.	o-Toluidine
7.	4-Dimethylaminoazobenzene	47.	o,o,o-Triethylphosphorothioate
8.	7,12-Dimethylbenz(a)anthracene	48.	sym-Trinitrobenzene
9.	3,3'-Dimethylbenzidine	49.	Methyl methacrylate
10.	a,a-Dimethylphenethylamine	50.	Ethyl methacrylate
11.	1,3-Dinitrobenzene	51.	Ethyl methane sulfonate
12.	Diphenylamine	52.	o-Anisidine
13.	1,4-Dioxane	53.	p-Cresidine
14.	Famphur	54.	5-Chloro-2-methylaniline
15.	Hexachloropropene	55.	Phthalic anlydride
16.	Isodrin	56.	2,4-Diaminotoluene
17.	Isosafrole	57.	1-Chloronaphthalene
18.	Kepone	58.	Diallate
19.	Methapyrilene hydrochloride	59.	Disulfoton
20.	3-Methylcholanthrene	60.	Methylparathion
21.	Methyl methane sulfonate	61.	Ethylparathion
22.	1,4-Naphthoquinone	62.	4-Aminoazobenzene
23.	1-Naphthylamine	63.	3,3-Dimethoxybenzidine
24.	2-Naphthylamine	64.	Tetraethyldithiopyrophosphate
25.	4-Nitroquinoline-1-oxide	65.	2,6-Dichlorophenol
26.	N-Nitrosodi-n-butylamine	66.	Aramite
27.	N-Nitrosodiethylamine	67.	Hexachlorophene
28.	N-Nitrosodimethylamine		
29.	N-Nitrosomethylethylamine		
30.	N-Nitrosomorpholine		
31.	N-Nitrosopiperidine		
32.	N-Nitrosopyrrolidine		
33.	5-Nitro-o-toluidine		
34.	Pentachlorobenzene		
35.	Pentachloronitrobenzene		
36.	Phenacetin		
37.	1,4-Phenylenediamine		
38.	Phorate		
39.	2-Picoline		
40.	Pronamide		

TABLE 3
ADDITIONAL COMPOUNDS

1. Chloropicrin
2. Malononitrile
3. Thiodiglycol
4. 1,4-Oxathiane
5. 1,4-Dithiane
6. Diisopropyl Methylphosphonic Acid
7. Dimethyl Methylphosphonic Acid
8. 2-Chlorobenzilidenemalononitrile
9. 2-Chloroacetophenone
10. 3-Chloroacetophenone
11. 4-Chloroacetophenone
12. 2-Chlorobenzaldehyde
13. 3-Chlorobenzaldehyde
14. 4-Chlorobenzaldehyde
15. 2-Hydroxyacetophenone
16. 3-Hydroxyacetophenone
17. 4-Hydroxyacetophenone
18. Benzyl Alcohol
19. Benzoic acid***
20. 1,2-Dichlorobenzene
21. 1,3-Dichlorobenzene
22. 1,4-Dichlorobenzene
23. 1,2,4-Trichlorobenzene

Figure 1
Peak Tailing Factors



Peak Tailing Factor = BC/AB

Sample calculation: Peak Height = $DE = 100\text{mm}$
10% Peak Height = $BD = 10\text{ mm}$
Peak Width at 10% Peak Height = $AC = 23\text{mm}$
 $AB = 11\text{ mm}$
 $BC = 12\text{ mm}$
Tailing Factor = $12/11 = 1.1$

SOP No: Q.6
Title: SOP for Method 8081B
Organochlorine Pesticides

1.0 SCOPE AND APPLICATION

1.1 Method 8081B is used to determine the concentration of various organochlorine pesticides in extracts from solid and liquid matrices. Table 1 indicates compounds that may be determined by this method.

TABLE 1

Target Compound

Alpha-BHC	Diallate
Gamma-BHC (Lindane)	Isodrin
Heptachlor	Kepone
Aldrin	Toxaphene
Beta-BHC	Tetrachloro-m-xylene (Sur)
Delta-BHC	Decachlorobiphenyl
Heptachlor Epoxide	
Endosulfan I	
4,4'-DDE	
Dieldrin	
Endrin	
Endosulfan II	
4,4'-DDD	
4,4'-DDT	
Endrin Aldehyde	
Endosulfan Sulfate	
Methoxychlor	
Endrin Ketone	
Alpha-Chlordane	
Gamma-Chloridane	
Hexachlorobenzene	
Hexachlorocyclopentadiene	
Chlorobenryilate	

1.2 The following compounds may also be determined using this method:

<u>Compound Name</u>	<u>CAS Registry No.</u>
Arochlor	15972-60-8
Captafol	2425-06-1
Captan	133-06-2
Chloroneb	2675-77-6
Chloropropylate	99516-95-7
Chlorothalonil	1897-45-6
DCPA	1861-32-1
Dichlone	117-80-6
Dicofol	115-32-2
Etridiazole	2593-15-9
Halowax-1000	58718-66-4
Halowax-1001	58718-67-5
Halowax-1013	12616-35-2
Halowax-1014	12616-36-3
Halowax-1051	2234-13-1
Halowax-1099	39450-05-0
Mirex	2385-85-5
Nitrofen	1836-75-5
PCNB	82-68-8
Perthane	72-56-0
Propachlor	1918-16-17
Strobane	8001-50-1
Trans-Nonarochlor	39765-80-5
Trans-Permethrn	51877-74-8
Trifluralin	1582-09-8

2.0 SUMMARY OF METHOD

2.1 Method 8081A provides gas chromatographic conditions for the detection of ppb levels of certain organochlorine pesticides. Prior to the use of this method, appropriate sample extraction techniques must be used.

3.0 INTERFERENCES

3.1 Interferences by phthalate esters can pose a major problem in pesticide determinations when using the electron capture detector. These compounds generally appear in the chromatogram as large-eluting peaks, especially in the 15% and 50% fraction from cleanups. Avoiding contact with any plastic materials can best minimize interferences from pthalates.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph

Hewlett Packard GC Systems 5890 equipped with autosampler is used. EnviroQuant software systems for data recording and processing is interfaced with the GC system.

4.2 Columns

4.2.1 Dual column connected with a "Y" connector to a single injection port is used. In this mode, material injected is split between the columns and detected by 2 separate ECD detectors.

4.2.2 Column 1: 30 meter, 5% ophenyl DI Methyl Polysiloxane (RTX-CLP) fused silica column is used.

4.2.3 Column 2: 30 meter, 14% cyanopropyl methyl polysiloxane (RTX-CLP2) fused silica column is used.

4.2.4 5 meter guard column (inert) is used at the "Y" connector.

4.3 GC Condition

4.3.1 The following GC parameters are implemented when Pest/PCBs are analyzed:

- Injection port temperature 245°C
- Detectors temperature 300°C
- A tri-ramp temperature program is used

1st Ramp:

Initial temperature	150°C
Initial time	0.5 min
Rate	8°C/min
Final temperature	180°C
Final time	10 min

2nd Ramp:

Initial temperature	180°C
Rate	8°C/min
Final temperature	210°C
Final time	10 min

3rd Ramp:

Initial temperature	210°C
Rate	15°/min
Final temperature	270°C
Final time	8 min

- Equilibrium time 0.75 min
- Septum purge on at 0.75 min at about 2ml/min
- Attenuation 0
- Signal ranges 0
- Splitless purge 50ml/min
- Makeup gas 80ml/min

4.4 Gases

- 4.4.1 Helium ultrapure grade is used as a gas carrier at about 5ml per min at 50° measured at the column end.
- 4.4.2 Make up gas is Argon/Methane (5% Methane) and the flow rate is about 80 ±5ml per min for each detector.

5.0 CALIBRATION

- 5.1 Calibration standards are prepared at five concentration levels through dilution of the stock standards with hexane (refer to standard logbook). Concentrations of the five level calibration are listed on Table 3.

TABLE 3

Compound	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)
4,4'-DDD	5	25	50	75	100
4,4'-DDE	5	25	50	75	100
4,4'-DDT	5	25	50	75	100
Aldrin	5	25	50	75	100
alpha-BHC	5	25	50	75	100
beta-BHC	5	25	50	75	100
Alpha-Chlordane	5	25	50	75	100
Gamma-Chlordane	5	25	50	75	100
Decachlorobiphenyl (CB Surr)	2.5	12.5	25	37.5	50
delta-BHC	5	25	50	75	100
Tetrachloro-m-xylene (Surr)	2.5	12.5	25	37.5	50
Dieldrin	5	25	50	75	100
Endosulfan I	5	25	50	75	100
Endosulfan II	5	25	50	75	100
Endosulfan Sulfate	5	25	50	75	100
Endrin	5	25	50	75	100
Endrin aldehyde	5	25	50	75	100
Endrin ketone	5	25	50	75	100
Gamma-BHC (Lindane)	5	25	50	75	100
Heptachlor	5	25	50	75	100
Heptachlor epoxide	5	25	50	75	100
Methoxychlor	5	25	50	75	100
Toxaphene			500		

- 5.2 Surrogate standards Tetrachloro-m-xylene and Decachlorobiphenyl are used. They are calibrated at levels indicated on Table 3.

6.0 GC ANALYSIS

6.1 Retention time windows

- 6.1.1 Make 3 injections of midpoint standard mixtures and multiplexes throughout the course of a 72-hour period. Calculate standard deviation of the three absolute retention times for each single component standard. For multiresponse products, choose one major peak from the cluster and calculate the standard deviation of the three retention times for that peak.

6.1.1.1 Plus or minus three times the standard deviation of the absolute retention times for each standard will be used to define retention time window. For multiplex products, primarily combination of chromatography pattern and retention times are used.

6.1.1.2 In those cases where the standard deviation for a particular standard is zero, substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.

6.1.1.3 When a new GC column is installed, retention time window must be established.

6.2 Degradation of DDT and Endrin

- 6.2.1 Check for degradation problems by injecting a EVAL MIX containing 4,4'-DDT and endrin. Look for the degradation products of 4,4'-DDT (4,4'-DDE and 4,4'-DDD) and endrin (endrin ketone and endrin aldehyde). Degradation must be less than 15% before sample analysis.

$$\% \text{ Breakdown for DDT} = \frac{\text{Total DDT degradation peak area (DDE + DDD)} \times 100}{\text{Total DDT peak area (DDT + DDE + DDD)}}$$

$$\% \text{ Breakdown for Endrin} = \frac{\text{Total Endrin degradation peak area (endrin aldehyde+endrin ketone)} \times 100}{\text{Total Endrin peak area (endrin+endrin ketone+endrin aldehyde)}}$$

- 6.2.2 At the beginning of each day, break down of Endrin and DDT must be in control before sample analyses should commence. When compounds don't meet the required criteria, as a corrective action for the breakdown, clipping portion of the column and injection port clean up will be performed. Also, to reduce some of the contaminations built up, the gold seal will be replaced.

6.3 Calibration

6.3.1 Five point calibration of pesticides and single point calibration for toxaphene and chlordane are initially analyzed. After analyzing the initial calibration standards, average response factor (CF) should be calculated for each compound. The percent relative standard deviation %RSD should be equal or less than 20% for each compound. It is likely that some analytes may exceed the 20% acceptance limit for the %RSD. In those instances the following steps should be considered:

- If the %RSD is greater than 20%, the analyst should review the results (area counts, calibration factors, standard concentrations) for those analytes to ensure that the problem is not associated with a single standard. If so, that standard should be reanalyzed and RSD recalculated. Replacing of standard may be necessary in some cases.
- It may also be necessary to narrow the calibration ranges to achieve a better linearity. High standard may saturate the column or/and the detector and need to be dropped from the calibration curve accordingly. Similarly poor purging compounds that exhibited erratic chromatographic behavior in the lowest calibration point could also be reviewed and dropped if necessary. The changes to the upper end of the calibration will affect the need to dilute samples above that range, while the change to the lower calibration will affect the sensitivity and elevate the reporting limit of the method. When considering dropping one or two points to narrow the calibration range, a minimum of five points is required for curve to be acceptable.

After visual inspection of the calibration points for each analytes and for those analytes that did not meet the %RSD criteria, at the discretion of the analyst it should be considered to employ a regression equation that does not through the origin or a quadratic (second order – requires six standards) model. Linearity is presumed acceptable if the correlation coefficient (r) is equal to or greater than 0.995 or the coefficient of determination (r^2) is equal to or greater than 0.99.

- 6.3.2 Daily continuing calibration: degradation check, mid-level pesticide, 8081 other compounds, chlordane and toxaphene are analyzed at the beginning of each shift. Percent difference from the mean CF calculated from the ICAL should be within $\pm 15\%$ for all compounds except for surrogate.
- 6.3.3 Sequence continuing standard: mid-level pesticide should be injected between every ten injections of samples and/or QC and at the end of every 12 hours, whichever is more frequent. All samples that were injected after the standard exceeding the criteria of linearity must be re-injected if the initial analyses indicate the presence of specific target analyte that exceeded the criteria. However, if the standard analyzed after a group of samples exhibited a response for an analyte above 15%

limit, and if the analyte was not detected in the specific samples, re-analysis is not necessary. In contrast, if the response of the instrument exhibited 15% below the initial calibration response, then re-injection of sample is necessary (whether an analyte was detected or not-detected). Experience of the chromatographer is an influential factor in the determination of sample re-analysis.

6.3.4 A mid-level calibration verification standard prepared from a different source is injected following the five-point calibration. Recovery range should be within $\pm 20\%$. If acceptance criteria were not met, investigate the problem. Re-injection of a new 5-point initial calibration may be necessary.

6.3.5 When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations, raw data output showing the results of manual integration and notation of date and initials of person performing the task.

6.4 Sample Collection, Preservation and Handling

6.4.1 Sample extracts are preserved by keeping them cooled to 4°C

6.4.2 The holding times for extraction of the samples are 7 days of sampling for water samples, and 14 days of sampling for soil samples. If sample has been extracted outside the holding time, note the aberration on the sample non-conformity report, and notify the manager for further instruction.

6.4.3 Sample must be analyzed within 40 days of the extraction. If sample has been analyzed outside the holding time, note the aberration on the sample non-conformity report, and notify the manager.

6.5 Sample Analysis

6.5.1 Prime the GC column with 1ppm pesticides standard if the instrument has not been used for more than 12 hours followed by an instrument blank.

6.5.2 When all degradation and calibration requirements are met sample analysis may begin. After each batch of 10 runs, linearity should be checked before any more samples are analyzed. Refer to 7.0 for quality control requirements.

6.5.3 Dilution must be made if the response of any compound exceeds the highest calibration standard in the curve.

6.5.4 Peak identification is primarily based on detection on both columns within the established retention time. When results are confirmed using two dissimilar columns, the agreement between the quantitative results should be evaluated after identification has been confirmed. Calculate the relative percent difference (RPD) between the two results using the formula below.

$$RPD = \frac{|R1 - R2|}{(R1+R2)/2} \times 100$$

where R1 and R2 are the results on the two columns and the vertical bars in the equation above indicate the absolute value of the difference. Therefore, the RPD is always a positive value.

6.5.4.1 If one result is significantly higher (e.g., > 40%), check the chromatogram for anomalies. If there is no evidence of chromatographic problem, report the higher result and noted in the case narrative. The experience of the analyst may prove invaluable in determining whether the results are confirmed or not based on retention times and agreement between quantitation results of both columns.

6.5.4.2 When results are confirmed using a second GC column, the higher of the two column results should be reported, unless the column associated with the higher results did not pass the acceptance criteria. In such cases, the lower value that is associated with the column that did pass the criteria may be reported. The experience of the analyst may prove invaluable in determining which results from which column will be reported.

6.5.5 GCMS confirmation may be implemented if the concentration permits.

6.6 Cleanup

6.6.1 If peak detection and identification of pesticides are prevented due to interferences of steroids, esters, ketones, glycerides and some hydrocarbons, the extract may need to undergo florisil cleanup using Method 3620B. If peak detection of pesticides are prevented by sulfur interferences which appear as huge hump at the beginning of the chromatogram, the extract may need to undergo sulfur cleanup (Method 3660B).

6.7 Low Concentration Method

6.7.1 This method is applicable to soil and water samples containing low level contaminants with minimal matrix interferences. To achieve low detection limits, the final volumes of sample extracts are brought to 2.0ml. Concentrations of surrogate and matrix spike standards should be adjusted before extraction in order to produce levels comparable to low level procedure. Quality control and recovery ranges are not expected to be affected by final volume reduction and low level quality control criteria should be applicable to this modified method.

6.8 Calculation

6.8.1 All quantifications are based on external standard calculations.

6.8.1.1 Calculation for calibration factor

$$\text{Calibration factor} = \frac{\text{Total Area of Peak}^*}{\text{Mass injected (nanograms)}}$$

* for multipeak analytes use total areas of all designed peaks for quantitation.

6.8.1.2 Percent difference

$$\text{Percent difference} = \frac{(R_1 - R_2) \times 100}{R_1}$$

R₁ = Calibration factor from first analysis

R₂ = Calibration factor from succeeding analyses

6.8.1.3 The concentration of each analyte in the sample may be determined by calculating the amount of standard injected from the peak response, using the calibration curve or factor determined from 6.7.1.1.

$$\text{Aqueous Concentration (ug/L)} = \frac{[(A_x)(A)(V_t)(D)]}{[(A_s)(V_i)(V_s)]}$$

$$\text{Solid Concentration (ug/kg)} = \frac{[(A_x)(D)(A)(V_t)]}{[(A_s)(W_s)(V_i)]}$$

Where:

A_x = Response for the analyte in the sample, (area or peak height)

A = Amount of standard injected in ng

A_s = Response for the external standard

V_i = Volume of extract injected

D = Dilution factor, if any

V_t = Volume of total extract

V_s = Volume of sample extracted

W_s = Weight of sample extracted

For non-aqueous samples, the unit is µg/kg and dry weight of sample is used for W_s.

6.8.1.4 For DOD projects, when results are confirmed using a second column, the calibration criteria for both columns are the same and must be met in order for the analysis to be valid.

7.0 QUALITY CONTROL

7.1 Required Instrument QC

- 7.1.1 It is required that the % RSD vary by $\leq 20\%$ when 5 point calibration factors are compared.
- 7.1.2 It is required that difference of daily response of a given analyte vary by $\pm 15\%$ when compared to initial responses. If criteria were not met either on the basis of each compound or the average across all compounds, check instrument conditions, re-inject another mid-level calibration standard, and if necessary, analyze a new 5-point calibration.
- 7.1.3 All succeeding standards in an analysis sequence must fall within the daily retention time window established by the first standard of the sequence. If retention time shifted, perform regular maintenance on the instrument and re-inject the mid-level standard. If the system is still unstable, the problem should be corrected before any further samples can be analyzed.
- 7.1.4 Control limits for MS/MSD, LCS and surrogates are established semi-annually for internal use.
- 7.1.5 For DOD projects, recoveries of LCS, MS/MSD and surrogates are evaluated against DOD QSM control limits policy.

7.2 Method Detection Limits

- 7.2.1 Method detection limits for this method are listed in the GPL Laboratory Method Detection Limit and Reporting Limit table.

7.3 Method Performance

- 7.3.1 The MDL concentrations listed in the GPL MDL table are generally obtained using organic-free reagent water. Results were also obtained by extracting seven spiked replicates the same way as the samples and analyzing them. MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. Precision and accuracy studies are performed once a year at a minimum. Single operator precision, overall precision and method accuracy were found to be directly related to the concentration of the parameter.

7.4 Matrix Spikes

- 7.4.1 MS and MSD must be analyzed with each batch of up to 20 samples of the same matrix processed together. If less than twenty samples are analyzed per month, MS/MSD must be analyzed on per month basis. Percent recoveries and Relative Percent Difference (RPD) should be calculated as follows:

$$\text{Matrix Spike \% Recovery} = \frac{\text{SSR}-\text{SR}}{\text{SA}} \times 100$$

Where:

SSR = Spike Sample Results

SR = Sample Results

SA = Spike Added from Spiking Mix

$$\text{RPD} = \frac{\text{D1} - \text{D2}}{(\text{D1} + \text{D2})/2} \times 100$$

D1 = First Sample Value

D2 = Second Sample Value

The MS/MSD is evaluated by comparing the precision of target analytes to the recovery windows established. MS/MSD data evaluation is more complex than method blank or LCS data since MS/MSD measure matrix effect in addition to sample preparation and analysis error. MS/MSD that fail to meet the acceptance criteria would indicate that a potential matrix effect is present. The laboratory must assess the batch to determine whether the spike results are attributable to matrix affect, or the result of another problem in the analytical process. If all the QC batch elements, which are not affected by the sample matrix, are in control (e.g., method blank, LCS), and if there is no evidence that the spiking was not properly performed, the poor spike recovery may be attributed to matrix effect. If the LCS compounds that are not affected by the sample matrix are out of control, and if the same compounds in the MS/MSD are outside control limit, then matrix spiked sample(s) must be re-processed through the entire analytical procedure.

7.5 Lab Control Sample (LCS)

- 7.5.1 A control check sample is extracted and analyzed at frequency as the method blank with each extracted batch. LCS compound must be within the established control limits. Statistical control limit are based on minimum of 20 data points. Data points used in the data set must not be selectively included or excluded. If recovery of LCS compound falls outside the established limit, corrective action must be taken. After corrective action, if LCS analyte recovery is still outside QC acceptance limits, re-extraction of the batch of samples may be necessary if the holding times have not elapsed and/ or refer to supervisor. Should any LCS be out of control, the lab must re-extract and re-analyze for all compounds that had criteria failed. In-house established limits for LCS should be in the range of $\pm 20\%$. When the results of the matrix spikes indicate a matrix problem, the LCS results are used to verify the laboratory performance in a clean matrix.

- 7.5.2 Each LCS must be evaluated against the Control Limits and Marginal Exceedance limit. If any LCS is outside the Control Limit, it should be also compared to the Laboratory Marginal Exceedance Limits to ensure that it does not exceed. If a single analyte in the LCS exceeded the Marginal Exceedance Limit, the LCS had failed and corrective action needs to be initiated.
- 7.5.3 If the LCS has more than the allowable number of Marginal Exceedance, the LCS has failed and corrective action needs to be conceded.
- 7.5.4 The corrective action for failed LCS is based on professional judgment in conjunction with matrix spike and surrogate recoveries in the same batch. If after checking the associated QC's it was determined by the section supervisor or manager that the LCS had failed, all affected samples associated with the out of control LCS should be reprocessed and re-analyzed
- 7.5.5 If samples cannot be reprocessed due to lack of sample volume or the holding times has lapsed, the results should be reported with appropriate flag. The report case narrative must include a discussion of the failed LCS, and its impact on the data quality.
- 7.5.6 When LCS is spiked with large number of analytes, the laboratory should add up total number of exceedances for the LCS based on the number of analyte spiked in the LCS. The total of exceedance should be compare with the allowable number from the following chart.

Number of analyte in the LCS	Allowable Number of Marginal Exceedances of LCS
< 11	0
11-30	1
31-50	2
51-70	3
71-90	4
>90	5

- 7.5.7 Laboratory Control Spike for toxaphene and chlordane should be analyzed quarterly unless it is requested sooner by a client.
- 7.6 Blanks
 - 7.6.1 Blank is extracted and analyzed with each analytical batch. Blanks must be contaminant free. Concentration of any confirmed peak should be less than one half of the reporting limit.

7.7 Surrogates

7.7.1 Percent recoveries for the surrogates determined by plotting percent recoveries of surrogates measured in 20 consecutive blanks. Control limits for each surrogate compound is measured using the following formula:

$$\text{Upper Control Limit (UCL)} = p + 3s$$

$$\text{Lower Control Limit (LCL)} = p - 3s$$

Where p is the mean recovery and s is the standard deviation.

7.7.2 Two surrogates (TCX and DCB) are added to each sample, however, one surrogate can be acceptable for QC determination. Calculate surrogate standard recovery on all samples, and all QC samples. Determine if the recovery is within limits. If recovery is not within limits, re-extract and re-analyze the sample and/or refer to supervisor.

7.7.3 For DOD projects, surrogate recoveries should be evaluated against DOD QSM control limits policy(Appendix DOD-D).

8.0 SAFETY

8.1 Safety glasses for eye protection, laboratory coats for body protection, latex gloves for hand protection.

8.2 Due to the toxicity or carcinogenicity of each reagent, each chemical compound should be treated as a potential health hazard.

8.3 Material safety Data Sheets (MSDS) can be found on the procurement bookshelves located in the company library.

8.4 Preparation of the standard should be handled under a hood.

9.0 POLLUTION PREVENTION

9.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

10.0 WASTE MANAGEMENT

10.1 Several wastes that GPL generates can be handled in a fairly routine manner. The process of describing the method for waste disposal of chemicals including standards and reagent solutions, and process waste, and samples is described in Standard Operating Procedures D.1 and D.2.

11.0 DEFINITIONS

11.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

12.0 REFERENCES

- Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition, Revision 1, December 1996.
- DOD Quality System Manual, Final Version 3, March 2005

SOP No: Q.7

Title: SOP for Method 8082A
PCBs, PCTs and PCB Congeners

1.0 SCOPE AND APPLICATION

1.1 Method 8082A is used to determine the concentrations of various polychlorinated biphenyls (PCBs), polychlorinated terphenyls (PCTs), or as individual PCB congeners in various matrices such as aqueous, solids, oils, products and wipes. Table 1 indicates compounds that may be determined by this method.

TABLE 1

Target Compounds

- 2-Chlorobiphenyl
- 2,3-Dichlorobiphenyl
- 2,2',5-Trichlorobiphenyl
- 2,4',5-Trichlorobiphenyl
- 2,2',5,5'-Tetrachlorobiphenyl
- 2,3',4,4'-Tetrachlorobiphenyl
- 2,2',3,4,5'-Pentachlorobiphenyl
- 2,2',4,5,5'-Pentachlorobiphenyl
- 2,3,3',4',6-Pentachlorobiphenyl
- 2,2',3,4,4',5'-Hexachlorobiphenyl
- 2,2',3,4,5,5'-Hexachlorobiphenyl
- 2,2',3,5,5',6-Hexachlorobiphenyl
- 2,2',4,4',5,5'-Hexachlorobiphenyl
- 2,2',3,3',4,4',5-Heptachlorobiphenyl
- 2,2',3,4,4',5,5'-Heptachlorobiphenyl
- 2,2',3,4,4',5',6-Heptachlorobiphenyl
- 2,2',3,4',5,5',6-Heptachlorobiphenyl
- 2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl
- 2,2',3,3',4,4',5,6-Octachlorobiphenyl
- 2,2',3,3',4,4'-Hexachlorobiphenyl
- 2,2',3,5'-Tetrachlorobiphenyl
- 2,3,4,4',5-Pentachlorobiphenyl
- 2,3',4,4',5-Pentachlorobiphenyl
- 2,3',4,4',5,5'-Hexachlorobiphenyl

2,3,3',4,4',5'-Hexachlorobiphenyl
2,3,3',4,4',5,5'-Heptachlorobiphenyl
2,3,3',4,4',5-Hexachlorobiphenyl
2,3,3',4,4'-Pentachlorobiphenyl
2,4'-Dichlorobiphenyl
2,4,4'-Trichlorobiphenyl
3,3',4,4'-Tetrachlorobiphenyl
3,3',4,4',5-Pentachlorobiphenyl
3,3',4,4',5,5'-Hexachlorobiphenyl
3,4,4',5-Tetrachlorobiphenyl
Aroclor 1016
Aroclor 1260
Aroclor 1221
Aroclor 1232
Aroclor 1242
Aroclor 1248
Aroclor 1254
Aroclor 5432
Aroclor 5460
Aroclor 6040
Aroclor 6062
Aroclor 6070
Tetrachloro-m-xylene(Sur)
Decachlorobiphenyl (Sur)
Hexabromobiphenyl (Sur)

2.0 SUMMARY OF METHOD

- 2.1 Method 8082A provides gas chromatographic conditions for the detection of ppb levels of certain polychlorinated biphenyls, polychlorinated terphenyls and congeners.
- 2.2 Prior to the use of this method, appropriate sample extraction techniques must be used.

3.0 INTERFERENCES

- 3.1 Interferences by phthalate esters can pose a major problem in PCB determinations when using the electron capture detector. These compounds generally appear in the chromatogram as large-eluting peaks, especially in the 20% and 50% fraction from cleanups. Interferences from phthalates compounds can best be minimized by avoiding contact with any plastic materials.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph

Hewlett Packard GC Systems 5890 equipped with autosampler is used. EnviroQuant software systems for data recording and processing is interfaced with the GC system.

4.2 Columns

4.2.1 Dual column connected with a "Y" connector to a single injection port is used. In this mode, material injected is split between the columns and detected by 2 separate ECD detectors.

4.2.2 Column 1: 30 meter, 5% phenyl methyl polysiloxane (RTX-CLP) fused silica column is used.

4.2.3 Column 2: 30 meter, 14% cyanopropyl methyl polysiloxane (RTX-CLP2) fused silica column is used.

4.2.4 5-meter guard column (inert) is used at the "Y" connector.

4.3 GC Condition

4.3.1 The following GC parameters are implemented when PCBs are analyzed:

- Injection port temperature 200°C
- Detectors temperature 300°C
- A double ramp temperature program is used

1st Ramp:

Initial temperature	150°C
Initial time	1.0 min
Rate	15°C/min
Final temperature	200°C
Final time	4.0 min

2nd Ramp:

Initial temperature	200°C
Rate	10°C/min
Final temperature	300°C
Final time	5.0 min

- Equilibrium time 0.75 min
- Septum purge on at 0.75 min at about 2ml/min
- Attenuation 0
- Signal ranges 0
- Splitless purge 50ml/min
- Makeup gas 80ml/min

4.3.2 The following GC parameters are implemented when PCTs are analyzed:

Injector Temp	200°C
Detector Temp	300°C
Initial Temp	150°C
Initial Time	1.0 min.
Ramp Rate	15°/min.
Final Temp	300°C
Final Time	15 min.

The Electronic Pressure Controller should be programmed as follows:

Initial Pressure Time	20min.
Initial Pressure	8 PSI
Rate	99 PSI
Final Pressure	25 PSI
Final Time	13min.

4.4 Gases

4.4.1 Helium ultrapure grade is used as a gas carrier at about 5ml per min at 50° measured at the column end.

4.4.2 Make up gas is Argon/Methane (5% methane) and the flow rate is about 80 ± 5ml per min for each detector.

5.0 CALIBRATION AND STANDARDIZATION

Calibration standards are prepared at five concentration levels through dilution of the stock standards with hexane (refer to standard logbook). Concentrations of the five level calibration and single point Aroclor are listed on Table 2.

5.1 Aroclors

To demonstrate the linearity of the detector, for PCBs a five-point concentration of a mixture of Aroclor 1016 and Aroclor 1260 should be analyzed. Mid-level point of other five Aroclor (listed on Table 2) are required to aid in pattern recognition and single point calibration.

For PCTs, a five point concentration of standards are analyzed (listed on Table 2) and calibration curves for each target analyte is established.

5.2 PCB Congeners

If samples are to be determined for individual PCB congeners, prepare a minimum of five concentrations of PCB congeners.

TABLE 2

Compound	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	Level 6 (ug/L)
2-Chlorobiphenyl	2	5	25	50	75	100
2,3-Dichlorobiphenyl	2	5	25	50	75	100
2,2',5-Trichlorobiphenyl	2	5	25	50	75	100
2,4',5-Trichlorobiphenyl	2	5	25	50	75	100
2,2',3,5'-Tetrachlorobiphenyl	2	5	25	50	75	100
2,2',5,5'-Tetrachlorobiphenyl	2	5	25	50	75	100
2,3',4,4'-Tetrachlorobiphenyl	2	5	25	50	75	100
2,2',3,4,5'-Pentachlorobiphenyl	2	5	25	50	75	100
2,2',4,5,5'-Pentachlorobiphenyl	2	5	25	50	75	100
2,3,3',4',6-Pentachlorobiphenyl	2	5	25	50	75	100
2,2',3,4,4',5'-Hexachlorobiphenyl	2	5	25	50	75	100
2,2',3,4,5,5'-Hexachlorobiphenyl	2	5	25	50	75	100
2,2',3,5,5',6-Hexachlorobiphenyl	2	5	25	50	75	100
2,2',4,4',5,5'-Hexachlorobiphenyl	2	5	25	50	75	100
2,2',3,3',4,4',5-Heptachlorobiphenyl	2	5	25	50	75	100
2,2',3,4,4',5,5'-Heptachlorobiphenyl	2	5	25	50	75	100
2,2',3,4,4',5,6-Heptachlorobiphenyl	2	5	25	50	75	100
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	2	5	25	50	75	100
2,3,4,4',5-Pentachlorobiphenyl	2	5	25	50	75	100
2,2',3,3',4,4',5,6-Octachlorobiphenyl	2	5	25	50	75	100
2,2',3,3',4,4'-Hexachlorobiphenyl	2	5	25	50	75	100
2,2',3,5'-Tetrachlorobiphenyl	2	5	25	50	75	100
2,3',4,4',5,5'-Hexachlorobiphenyl	2	5	25	50	75	100
2,3',4,4',5-Pentachlorobiphenyl	2	5	25	50	75	100
2,3,3',4,4',5'-Hexachlorobiphenyl	2	5	25	50	75	100
2,3,3',4,4',5,5'-Heptachlorobiphenyl	2	5	25	50	75	100
2,3,3',4,4',5-Hexachlorobiphenyl	2	5	25	50	75	100
2,3,3',4,4'-Pentachlorobiphenyl	2	5	25	50	75	100
2,4'-Dichlorobiphenyl	2	5	25	50	75	100
2,4,4'-Trichlorobiphenyl	2	5	25	50	75	100
3,3',4,4'-Tetrachlorobiphenyl	2	5	25	50	75	100
3,4,4',5-Tetrachlorobiphenyl	2	5	25	50	75	100
3,3',4,4',5-Pentachlorobiphenyl	2	5	25	50	75	100
3,3',4,4',5,5'-Hexachlorobiphenyl	2	5	25	50	75	100
AR1016		100	250	500	750	1000
AR1221				500		
AR1232				500		
AR1242				500		
AR1248				500		
AR1254				500		
AR1260		200	500	1000	1500	2000
AR5432		250	500	1000	2000	4000
AR5460		250	500	1000	2000	4000
AR6040				1000		
AR6062				1000		
AR6070				1000		
Tetrachloro-m-xylene		5	12.5	25.0	37.5	50.0
Decachlorobiphenyl		5	12.5	25.0	37.5	50.0
Hexabromobiphenyl		5	12.5	25.0	37.5	50.0

5.3 Surrogates. Tetrachloro-m-xylene, Decachlorobiphenyl and Hexabromobiphenyl are used in PCB/PCT analyses. Calibration levels are indicated on Table 2.

5.4 Calibration

If samples are to be analyzed for PCBs.

5.4.1 Five-level calibration of a mixture of Aroclor 1016 and Aroclor 1260 are initially analyzed. Percent RSD (Relative Standard Deviation) must be $\leq 20\%$ for the Aroclors except surrogates. Analysis of single point mid level concentration of other five Aroclor listed in Table 1 are required. If PCB's are detected in the sample, patterns are compared for fingerprint match with initial standards and corresponding PCB's and retention time. Calculation is based on average area quantification of five representative peaks. When interferences are present, those peaks with less interferences may be chosen for quantification. Fewer peaks (minimum of 3 peaks) may be used for quantitation if there are considerable interferences.

5.4.2 Daily continuing calibration: Analyst should alternate the use of high and low concentrations of Aroclor 1016 and Aroclor 1260 at the beginning of each shift. Percent difference should be kept at $\pm 20\%$ for the Aroclors except for surrogates. If this criteria is exceeded for any Aroclor, calculate the average percent difference for all analytes in the calibration.

5.4.3 Mid sequence standard: mid-level mixture of Aroclor 1016/1260 should be injected every ten injections of samples and/or QC and at the end of every 12 hours, whichever is more frequent. All samples that were injected after the standard exceed the criteria of linearity must be re-injected if analyses indicate the presence of specific target analyte. However, if continuing calibration is $\geq 20\%$ and no target analyte was detected, re-injection of the samples is not necessary. If continuing calibration $\leq 20\%$ and no target analyte was detected, re-injection is necessary. Experience of the chromatographer is an influential factor in the determination of sample reanalysis.

If samples are to be analyzed for PCTs.

5.4.4 Initially 5 levels of standards containing AR5460 are analyzed and calibration curves will be established. Mid-level continuing calibrations are analyzed during the sequence run when PCTs are required. If PCTs are detected in the sample, patterns and retention times are compared with the calibration standard. Calculation is based on average area or peak height of 5 representative peaks. When interferences are present, those peaks with less interference may be chosen for quantification. Also fewer peaks (minimum of 3) may be used for quantification if there are considerable interferences. Surrogate

Decachlorobiphenyl (DCB) and Tetrachloro-m-xylene (TCMX) are used for quality control monitoring. Acid clean up can be performed if matrix effects are observed. However, only one surrogate needs to be calculated for recovery.

If samples are to be analyzed for congeners.

5.4.5 Initially five point calibration of PCB congeners are analyzed. The mean of the RSD value for all congeners must be equal or less than 20%. The initial calibration must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. A calibration standard must also be injected at the interval of 10 samples or/and QC samples, and at the end of the analysis sequence.

$$\% \text{ Difference} = \frac{CF_1 - CF}{CF_1} * 100$$

Where:

CF₁ = mean calibration factor from the initial calibration

CF = calibration factor from calibration verification standard

The calibration factor for each analyte calculated must not exceed a difference of more than 20% when compared to the mean calibration factor from the initial calibration. When calibration verification standard failed to meet the QC criteria, all samples that were injected after the last standard that met the QC criteria must be re-injected. However, if the sensitivity of the instrument had increase and target compounds are not present in the sample, there is no need for re-analysis

5.4.6 If samples are to be analyzed for low level congeners:

- The lowest calibration level shall be 2.0 ug/L.
- During sample preparation use 30 grams of sample with a final volume 2.0 ml
- The reporting limit shall be 0.1ug/kg.

5.4.7 When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations, raw data output showing the results of manual integration and notation of date and initials of person performing the task.

6.0 SAMPLE COLLECTION, HANDLING, PRESERVATION AND HOLDING TIMES

6.1 Sample extracts are preserved by keeping them cool to 4°C.

6.2 The holding times for extraction of the samples are 7 days of sampling for water samples and 14 days of sampling for soil samples. If sample has been extracted outside the holding time, note the aberration on the sample non-conformity report, and notify the manager for further instruction.

- 6.3 Sample must be analyzed within 40 days of the extraction. If sample has been analyzed outside the holding time, note the aberration on the sample non-conformity report, and notify the manager.

7.0 METHOD DETECTION LIMITS

- 7.1 Method detection limits for this method are listed in the GPL Laboratory Method Detection Limit and Reporting Limit table.

8.0 METHOD PERFORMANCE

- 8.1 The MDL concentrations listed in the GPL MDL table are generally obtained using organic-free reagent water. Results were also obtained by extracting seven spiked replicates the same way as the samples and analyzing them. MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. Precision and accuracy studies are performed once a year at a minimum. Single operator precision, overall precision and method accuracy were found to be directly related to the concentration of the parameter.

9.0 PROCEDURE

9.1 Retention time windows

- 9.1.1 Make 3 injections of midpoint standard mixtures throughout the course of a 72-hour period. Calculate standard deviation of the three absolute retention times for each single component standard. For multi-response products, choose one major peak from the cluster and calculate the standard deviation of the three retention times for that peak.

- 9.1.1.1 Plus or minus three times the standard deviation of the absolute retention times for each standard will be used to define retention time window. For multippeak products, primarily combinations of chromatography pattern and retention times are used.

- 9.1.1.2 In those cases where the standard deviation for a particular standard is zero, substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.

- 9.1.1.3 When a new GC column is installed retention time window must be established.

9.2 Sample Preparation

For sample preparation of soil and water, refer to GPL SOPs N.6 and N.7.

- 9.3 When all calibration requirements on both columns are met, sample analysis may begin. After each batch of 10 runs linearity should be checked before any more

samples are analyzed. All samples that were injected after the standard exceeding the criteria must be re-injected, if the initial analysis indicates the presence of a specific Aroclor that exceeded the criteria.

- 9.4 Dilution must be made if the response exceeds the linear range of the compounds.
- 9.5 Peak identification is primarily based on detection on both columns within the established retention time. When results are confirmed using two dissimilar columns, the agreement between the quantitative results should be evaluated after identification has been confirmed. Calculate the relative percent difference (RPD) between the two results using the formula below.

$$RPD = \frac{|R1 - R2|}{(R1+R2)/2} \times 100$$

where R1 and R2 are the results on the two columns and the vertical bars in the equation above indicate the absolute value of the difference. Therefore, the RPD is always a positive value.

- 9.5.1 If one result is significantly higher (e.g., > 40%), check the chromatogram for anomalies. If there is no evidence of chromatographic problem, report the higher result with a J flag (for DOD project) and noted in the case narrative. The experience of the analyst may prove invaluable in determining whether the results are confirmed or not based on retention times and agreement between quantitation results of both columns.
 - 9.6 GCMS Confirmation may be implemented if the concentration permits.
 - 9.7 Wipe samples are treated like a solid sample and results are reported as ug per wipe. All QC parameters used for soil samples are applicable to wipes except for MS/MSD analysis. An actual MS/MSD analysis is impractical since only one wipe is sampled at a location and it cannot be split.
 - 9.8 If peak detection and pattern identification are prevented by interferences, the extract should undergo acid cleanup using Method 3665A or sulfur cleanup using Method 3660B. The experience of the analyst may prove invaluable in determining which cleanup method is appropriate for particular samples.
- 10.0 DATA ANALYSIS AND CALCULATIONS
- 10.1 All quantifications are based on external standard calculations.
 - 10.1.1 Calculation for calibration factor

$$\text{Calibration factor} = \frac{\text{Total Average Area of Peak}^*}{\text{Mass injected (nonograms)}}$$

- * for multipeak analytes use total areas of all designated peaks for quantitation

10.1.2 Percent Difference

$$\text{Percent difference} = \frac{(R_1 - R_2)}{R_1} \times 100$$

R₁ = Calibration factor from first analysis

R₂ = Calibration factor from succeeding analyses

10.1.3 The concentration of each Aroclor in the sample may be determined by calculating the amount of standard injected from the peak response, using the calibration factor determined from the initial five point calibration for Aroclor 1016 and for 1260. For other Aroclor that may be present in the sample, CF from the single point calibration standard of the specific Aroclor will be used for calculation.

$$\text{Aqueous Concentration (ug/L)} = [(A_x)(A)(V_t)(D)] / [(A_s)(V_i)(V_s)]$$

$$\text{Solid Concentration (ug/kg)} = [(A_x)(D)(A)(V_i)] / [(A_s)(W_s)(V_i)]$$

where:

- A_x = Response for the analyte in the sample, (area or peak height)
- A = Amount of standard injected in ng
- A_s = Response for the external standard
- V_i = Volume of extract injected
- D = Dilution factor, if any
- V_t = Volume of total extract
- V_s = Volume of sample extracted
- s = Weight of sample extracted

For non-aqueous samples, the unit is µg/kg and dry weight of sample is used for W_s.

11.0 QUALITY CONTROL

11.1 Required Instrument QC

11.1.1 Percent RSD should be ≤20% when 5-point calibration factors are compared.

11.1.2 Percent difference of daily response of a given analyte should be within ±20% when compared to initial responses.

11.1.3 All succeeding standards in an analysis sequence must fall within daily retention time window established by the first standard of the sequence.

11.1.4 Control limits for MS/MSD, LCS, surrogates are established semi-annually for internal use.

11.1.5 For DOD projects, LCS, MS/MSD limits are evaluated against the DOD QSM control limits policy.

11.2 Matrix Spikes

11.2.1 MS and MSD must be analyzed with each batch of up to 20 samples of the same matrix processed together. If less than twenty samples are analyzed per month, MS/MSD must be analyzed on per month basis. Percent recoveries and Relative Percent Difference (RPD) should be calculated as follows:

$$\text{Matrix Spike \%} = \frac{\text{SSR} - \text{SR}}{\text{SA}} \times 100$$

where:

SSR = Spike Sample Results
SR = Sample Results
SA = Spike Added from Spiking Mix

$$\text{RPD} = \frac{D1 - D2}{(D1 + D2)/2} \times 100$$

D1 = First Sample Value
D2 = Second Sample Value

If samples are analyzed for PCBs only, then matrix spikes must be analyzed for AR1016 and AR1260.

If samples are analyzed for PCTs only, then matrix spikes must be analyzed for AR5460.

The MS/MSD is evaluated by comparing the precision of target analytes to the recovery windows established. MS/MSD data evaluation is more complex than method blank or LCS data since MS/MSD measure matrix effect in addition to sample preparation and analysis error. MS/MSD that fail to meet the acceptance criteria would indicate that a potential matrix effect is present. The laboratory must assess the batch to determine whether the spike results are attributable to matrix affect, or the result of another problem in the analytical process. If all the QC batch elements, which are not affected by the sample matrix, are in control (e.g., method blank, LCS), and if there is no evidence that the spiking was not properly performed, the poor spike recovery may be attributed to matrix effect. If the LCS compounds that are not affected by the sample matrix are out of control, and if the same compounds in the MS/MSD are outside control limit, then matrix spiked sample(s) must be re-processed through the entire analytical procedure.

11.3 Lab Control Sample (LCS)

11.3.1 A control check sample is extracted and analyzed at frequency similar to MS/MSD with each extracted batch. Lab Control Spikes (LCS) are analyzed per batch or every 20 samples, whichever comes first. LCS compound must be within the established control limit. Statistical control limit are based on at least 30 data points. Data points used in the data set must not be selectively included or excluded. If recovery of LCS compound falls outside the established limit, corrective action must be taken. After corrective action, if LCS analyte recovery is still outside QC acceptance limits, the entire associated samples batch must be re-extracted if holding times have not elapsed. When PCB analysis is needed the control check sample should be spiked with AR1016 and 1260 at the same levels as matrix spikes. When PCT analysis is needed the control check sample should be spiked with AR5460 at the same levels as matrix spikes.

11.4 Blanks

11.4.1 Blank is extracted and analyzed with each analytical batch. Blanks must be contaminant free. Concentration of any confirmed peak should be less than one half of the reporting limit.

11.5 Surrogates

11.5.1 Percent recoveries for the surrogates are determined by plotting percent recoveries of surrogates measured in 20 consecutive blanks. Control limits for each surrogate compound is measured using the following formula:

$$\begin{aligned}\text{Upper Control Limit (UCL)} &= p + 3s \\ \text{Lower Control Limit (LCL)} &= p - 3s\end{aligned}$$

where p is the mean recovery and s is the standard deviation.

11.5.2 Two surrogate (TCMX and DCB) are added to each sample, however, only one need to be calculated for recovery. Calculate surrogate standard recovery on all samples, blanks and spikes. Determine if the recovery is within limits. If recovery is not within limit, re-extract and reanalyze the sample.

11.5.3 For DOD projects, surrogate limits are evaluated against the DOD QSM control limits policy.

12.0 SAFETY

- 12.1 Safety glasses for eye protection, laboratory coats for body protection, latex gloves for hand protection.
- 12.2 Due to the toxicity or carcinogenicity of each reagent, each chemical compound should be treated as a potential health hazard.
- 12.3 Material Safety Data Sheets (MSDS) can be found on the procurement bookshelves located in the company library.
- 12.4 Preparation of the standard should be handled under a hood.

13.0 POLLUTION PREVENTION

- 13.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

14.0 WASTE MANAGEMENT

- 14.1 Several wastes that GPL generates can be handled in a fairly routine manner. The process of describing the method for waste disposal of chemicals including standards and reagent solutions, and process waste, and samples is described in Standard Operating Procedures D.1 and D.2.

15.0 DEFINITIONS

- 15.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

16.0 REFERENCES

- Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, 3rd Edition.
- DOD Quality System Manual , Final Version 3, January 2006

SGS Environmental Services
Standard Operating Procedure

Standard Operating Procedures
Sample Processing

Issue date: 03/31/08
Revision: 28

SGS Environmental Services
5500 Business Drive
Wilmington, North Carolina 28405

Approved by:

Name, Last Updated By

Date

Name, Laboratory Manager

Date

Name, QA/QC Manager

Date

SGS Environmental Services

Standard Operating Procedure

Purpose

To describe the procedures followed for the conditioning, extraction, splitting/archiving, spiking, concentration and cleanup of samples.

Summary

The primary objective of sample processing is to quantitatively remove the analytes of interest from the matrix into an appropriate solvent, and then to remove the solvent, leaving a dried extract that can then be reconstituted prior to analysis. Samples are first conditioned using homogenization for biological samples and grinding for solids with a particle size greater than 1 mm. If appropriate, a percent solids or percent lipids determination will be made during sample conditioning. The samples are then aliquotted and fortified with extraction standard. The samples are extracted using one of a variety of procedures depending on the matrix. These include Soxhlet-Dean Stark (SDS), continuous liquid-liquid extraction (CLLE), and specialized extraction procedures for wipes, milk, oils, and synthetic precipitation. If necessary, extracts may be split after extraction, with a portion of the extract archived for future use. The resulting extracts are concentrated using mini Snyder columns (K-D) or heated nitrogen blow down (Turbovap). Concentrated extracts are subjected to cleanup procedures in order to remove potential interferences. The cleanup procedures involve column chromatography using different packing materials as appropriate. The cleanup procedures result in small volumes of extracts which are then reduced to dryness.

3.1 Sample Conditioning

3.1.1 Homogenization

3.1.1.1 Equipment/Supplies

- Heavy-duty meat grinder, freezer, tissue macerator, butcher knife, commercial food containers, suitable personal protection equipment (PPE), waste receptacle, cut-resistant gloves.

3.1.1.2 Procedure

- Prior to processing tissue samples, it must be determined as to the exact tissue that is to be analyzed. Analytical requests include whole fish, whole fish less skin, fillets, specific organs or various portions of the above. Once identified, the appropriate tissue must be frozen.
- When practical, samples are ground and homogenized while frozen.
- If specific portions must be dissected from the whole specimen the remaining tissue can be refrozen. When the entire sample, usually fish, requires analysis, reduce the sample down to manageable means using a butcher knife.

Note: Great care must be exercised when reducing a frozen fish with a knife. Cut-resistant gloves must be worn to protect the hands as well as to hold the fish in place.

- When analyzing specific portions, such as a fillet, dissect the required portion under stringent safety guidelines to protect the analyst from injury as well as to ensure the integrity of the sample.
- Once reduced in size, process the tissue through a meat grinder and collect into a suitable container.
- To ensure complete homogeneity process the ground sample two more times.

Note: Further reduction can be achieved by processing a portion of the specimen with a commercially available tissue macerator. However, continuous reprocessing of the sample through a meat grinder will often achieve the desired results.

- Record all observations.

3.1.2 Grinding

3.1.2.1 Equipment/Supplies

- Mortar and pestle, heavy-duty meat grinder, freezer, mesh sieves of various pore sizes, suitable PPE, waste receptacle.

3.1.2.2 Procedure

- After donning the appropriate PPE, open the sample in the hood.
- Remove any large objects such as rocks.
- If necessary pass the sample through a series of sieves to help facilitate the partitioning of the various particle sizes.
- Particles requiring grinding can in most instances be reduced by means of a mortar and pestle.
- In some cases reducing the temperature of the sample will aide in the grinding process.
- Some matrices such as certain papers, grains and amorphous solids need to be reduced using a Wiley Mill or other suitable mechanism.
- After sufficient reduction in the gross particle size, recombine and mix the various fractions of the sample.
- Record all observations.

3.1.3 Determination of Particle Size

3.1.3.1 Equipment/Supplies

- Clean stainless steel forceps, fume hood, aluminum foil, suitable PPE, waste receptacle.

3.1.3.2 Procedure

- Spread a piece of clean aluminum foil in an appropriately ventilated fume hood.
- After donning the appropriate PPE, open the sample in the hood.

SGS Environmental Services Standard Operating Procedure

- With the aid of forceps spread a representative portion of sample on the aluminum foil.
- Estimate the size of the particles in the sample.
- If the estimate determines that the average particle size is greater than 1 mm, then the sample must be ground before extraction.
- Record all observations.

3.1.4 Percent Solids (by Modified ASTM D2216)

3.1.4.1 Equipment/Supplies

- Drying oven, dessicator, balance, aluminum weigh boats, and glass fiber filters.

3.1.4.1.1. Solids

- Label each weigh boat required with the unique sample ID number.
- Thoroughly homogenize the sample, and grind the sample if necessary.
- Weigh out 5-20 g of sample into the weigh boat.
- Record the tare and sample weights taken and make note of any unique characteristics.
- Note the time and date, then dry the sample in a $110^{\circ}\text{C} \pm 5^{\circ}\text{C}$ oven for 12 hours.
- After at least one hour, the sample may be weighted back in order to get an estimated result, as calculated below, that can be used to approximate the minimum extraction amount. If this is performed, note the time and date and record the appropriate data. Then place the sample back in the oven.
- Remove the sample and allow to cool in a dessicator.
- Note the time and date then reweigh the sample and record the dried weight.
- Calculate the % solids as follows:

$$\% \text{ solids} = \frac{\text{weight of sample after drying}}{\text{weight of sample before drying}} \times 100\%$$

- Continue to re-weigh the sample, waiting at least one hour between weightings, until its mass changes by less than 1%. Use the final dried weight in the calculation of percent solids provided for sample reporting.

3.1.4.1.2. Aqueous

- Desiccate and weigh a glass fiber filter to three significant figures.
- Filter 10.0 ± 0.02 mL of well-mixed sample through the filter.
- Dry the filter a minimum of 12 H at $110^{\circ}\text{C} + 5^{\circ}\text{C}$, and cool in a dessicator.
- Calculate percent solids by dividing the weight of solids trapped by 10, then times 100 for percent.

3.1.5 Percent Lipids

3.1.5.1 Equipment/Supplies

- balance

3.1.5.2 Procedure

- Extract the sample via the appropriate method (see Sec. 3.2.2).
- Concentrate the sample extract to “dryness” inside a tared container.
- Calculate the lipids content to the nearest three significant figures:

$$\text{Percent Lipids} = \frac{\text{Weight of Residue (in g)}}{\text{Weight of Wet Sample (in g)}} \times 100$$

3.1.6 High Moisture Procedures

- Soxhlet extraction: High moisture samples may be extracted using the Soxhlet/Dean-Stark (SDS) method (sec. 3.2.2). The Dean-Stark adapter continuously removes extracted moisture that may be drained off during the extraction.
- Filter/dual extraction: Samples that are very high in moisture content may be vacuum filtered. The filtrate may then be extracted by CLLE (sec. 3.2.5), and the filter cake may be extracted by SDS. The two extracts can then be recombined and treated as one sample. The following equation is used to calculate percent solids after filtering and drying the filter.

SGS Environmental Services Standard Operating Procedure

$$\% \text{ solids} = \frac{\text{weight of sample after drying} - \text{weight filter}}{10\text{g}} \times 100\%$$

- **Freeze Drying:** Samples with less than 30% solids should be considered for freeze drying (FD). This instrument consists of a Labconco manifold (#7522700), vacuum pump (#117) and an Edwards pirani gauge (#501). 150ml – 2000mL glassware is used depending on the amount of sample to be dried. A 10g equivalent is weighted into a FD vessel of sufficient size to allow a thin shell of sample to be frozen to the walls of the vessel. Cap the vessel and store it in a freezer at a 45° angle. Prepare the FD manifold by adding dry ice to the trap using the gloves provided and filling it with ethanol. This chamber is maintained at –78°C. Attach the vacuum hose and turn the pump on. To form the shell remove the chilled samples from the freezer and roll them in the dry ice/ ethanol bath. This shell freezing increases the surface area of sample exposed to vacuum, thus creating more consistent and faster drying. After the shell has formed attach the vessel to the manifold and slowly open its valve until the vessel is under vacuum. Once all samples have been added the vacuum should be maintained at 10⁻² torr. Repeat the shell drying for each sample. Refill the trap as needed, about every 5 hours. After 10 hours, remove each sample and check to see if they have dried completely. If so, one can store them for extraction. If not, reattach them to the manifold for another 2 hours, repeat as necessary until dry.

3.1.7 Sub Sampling

3.1.7.1 Equipment/Supplies

- Beaker, shaker, spatula.

3.1.7.2 Procedure

3.1.7.2.1. Water

- Shake sample to homogenize. Particulates must be incorporated and evenly distributed.
- A powered shaker may be used.

3.1.7.2.2. Soil

- Stirring the entire sample in its container is sufficient for wet samples.
- For dry samples, degenerate fractional shoveling is used to correctly take a representative subsample.
- Remove the sample containers cap and place it upside-down near the container and obtain a clean small beaker for the reject pile.
- Care should be taken when non-indigenous soils are handled. One must place foil down over the workspace to catch any spills. This foil and any items used in contact with the soil must be packed and properly labeled with purple stickers to insure proper disposal.
- Discard any foreign objects such as sticks, leaves, and rocks.
- The “top” layer of soil in the container is placed in the cap to prevent grab sampling errors. Mix the remaining sample in its jar.
- Proceed to place every third scoop in the subsample pile (usually a tared beaker or thimble). The other scoops go into the reject pile (the beaker obtained earlier).
- Once the desired mass of subsample has been acquired from the jar, return the reject pile to the jar. Using the cap, replace the “top” layer of soil and re-seal the jar prior to storage.
- Reference: Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples, EPA/600/R-03/027, by Gerlach and Nocerino November 2003

3.2 Sample Extraction

3.2.1 Waste Dilution

3.2.1.1 Equipment/Supplies

- 60 mL vial, balance

3.2.1.2 Solvent

- Hexanes

3.2.1.3 Procedure

- Weigh out 1 gram of oil into a 60 mL vial.
- Spike the sample with the appropriate amount of extraction standard as listed in the spike profile document.
- Add 5 mL of hexanes to the vial, making sure the sample is dissolved in the solvent.
- Proceed directly to PCU cleanup.
- If the matrix is known to be exceedingly difficult, an extra dilution may be performed. After weighing out the sample, add 20 mL of hexanes before spiking. Split the diluted sample into equal

SGS Environmental Services

Standard Operating Procedure

portions, spike one portion with standards, and archive the remaining portions. Then proceed to PCU cleanup.

3.2.2 Soxhlet/Dean-Stark (SDS)

3.2.2.1 Equipment/Supplies

- Complete SDS assembly, heating mantle, chiller, stainless steel spatula, thimble.

3.2.2.2 Extraction Solvents

- Dioxin solids: Toluene
- PCB solids: Methylene chloride
- All tissues: Methylene chloride

3.2.2.3 Procedure

3.2.2.3.1. Pre-extraction

For methods requiring pre-extraction, the following steps are followed:

- Add 2-4 Teflon™ chips inside the 500-mL round-bottom flask.
- Add 300 mL of extraction solvent to the flask.
- Place the thimble holder onto the flask.
- Add a thimble to the holder.
- Assemble the condenser.
- Turn on the water recirculator, which is set to 5°C.
- Turn on the heating mantle to position “64” for toluene, “50” for DCM.
- Allow the reflux to go on for at least three hours.
- Turn off the heating element, and allow the unit to cool down.
- Transfer the solvent to solvent waste.

3.2.2.3.2. Extraction

- Add 300 mL of extraction solvent to the round-bottom flask.
- Add 2-4 fresh Teflon™ chips.
- For dioxin methods, add 0.5 mL purified tridecane to the flask.
- Affix the thimble holder and insert the thimble containing the sample aliquot mixed with sodium sulfate.
- Spike the sample with the extraction standards as listed on the spike profile and allow to absorb. **Record the ES lot#, its concentration, and the volume (µL) spiked.**
- Spike the appropriate samples with matrix spike as listed on the spike profile and allow to absorb. **Record the MX lot#, its concentration, and the volume (µL) spiked.**
- Assemble the DS and condenser components (DS used for toluene extractions only).
- Turn on the water recirculator, then the heater. The heater should be set to “50” for methylene chloride, “64” for toluene.
- Insulate the round-bottom flask with foil.
- Allow the extraction to continue for 16 to 24 H.
- During the extraction, verify that the solvent cycles normally.
- Drain water from the Dean-Stark as appropriate to prevent water from returning to the boiling flask.

3.2.2.3.3. Concentration of the extract

- At the end of the reflux cycle time, proceed with the sample concentration as described below.
- **Do not turn off the heater yet.**
- Drain the toluene present in the DS side arm (if used) directly to solvent waste.
- Open the stopcock on the thimble holder and drain the extraction solvent from the soxhlet until approximately 20 mL remains inside the RB flask.
- Turn off the heater when the solvent reaches the 20-mL mark. **Do not go to “near dryness”.**
- Remove the heating mantle and allow the flask to cool down.
- Transfer the sample to a 60 mL vial for Turbovap concentration (3.6.3).

3.2.3 Extraction of XAD Air Samples by SDS

3.2.3.1 Equipment/Supplies

- Complete SDS assembly, heating mantle, chiller, XAD, thimble, boiling chips.

3.2.3.2 Solvent

- Toluene, tridecane

3.2.3.3 Procedure

- Add 2-4 fresh boiling chips to each round bottom flask.

SGS Environmental Services

Standard Operating Procedure

- Add 350 mL of toluene and 0.5 mL of purified tridecane.
- Adapt the thimble holder and insert thimble containing XAD.
- Spike the sample with extraction standard as listed on the spike profile and allow it to absorb. **Record the ES lot#, its concentration, and the volume (µL) spiked.**
- Spike the appropriate samples with matrix spike as listed on the spike profile and allow it to absorb. **Record the MX lot#, its concentration, and the volume (µL) spiked.**
- Assemble the DS and condenser components.
- Cover the opened extremity of the condenser with foil.
- Turn the chiller on, which is set to 5°C.
- Turn on the heating mantle, which is set at 65.
- Allow the extraction to continue for 16 to 24 hours.
- During the extraction, verify that the solvent cycles normally.
- Drain the water as appropriate to prevent water from returning to the boiling flask.
- At the end of the reflux cycle time, proceed with the sample concentration as described below.
- **Do not turn off the heater yet.**
- Drain the toluene present in the DS side arm directly to solvent waste.
- Drain the toluene present in the thimble holder to solvent waste.
- Continue to drain the toluene until approximately 20 mL of toluene remains inside the RB flask.
- Turn off the heater when the toluene reaches the 20 mL mark. **Do not go to “near dryness”.**
- Remove the heating mantle and allow the flask to cool down.
- Transfer sample to 60 mL vial. Split sample and archive as necessary.
- Concentrate sample in turbovap (3.6.3).

3.2.4 Separatory Funnel Extraction

3.2.4.1 Equipment/Supplies

- 2-liter separatory funnel, glass funnels, 250-mL beakers, 90-mm filter set-up with a 1-liter vacuum container, Na₂SO₄, glass wool, 2 µm x 90-mm GMF 150 filters, wide range pH paper, 40 mL vials.

3.2.4.2 Solvent

- Methylene chloride, H₂SO₄.

3.2.4.3 Procedure

- Prepare drying funnels by packing the neck of a glass funnel with glass wool.
- Add approximately 10-15 g of Na₂SO₄ on top of the glass wool and position over an appropriately labeled collection vial.
- Add 0.1 mL of tridecane if necessary.
- Place the sample bottle on the balance and zero the scale.
- Add the contents of the sample bottle to the separatory funnel, reweigh the bottle and record weight of the displaced sample volume.
- Test the pH of the sample and record the pH on the extraction sheet.
- Add 5 mL H₂SO₄ to the sample to reduce pH to <2.
- Add extraction standard to the sample, shake vigorously and allow it to dissolve.
- Add 60 mL of CH₂Cl₂ to the separatory funnel.
- Shake for at least 2 min, periodically purging the separatory funnel of any excess pressure.
- Allow the sample-solvent solution to settle out and form a distinct interface.
- Drain the solvent through the sodium sulfate funnel into the collection vial.
- Repeat the extraction twice more, collecting all the solvent into the same vial.
- Concentrate aliquots of the sample in the Turbovap (3.6.3).
- Prepare a 25 mL sodium sulfate column as above and dry the extract.
- If necessary, decant the water present before adding the sample to the drying column.

3.2.5 Continuous Liquid-Liquid Extraction

3.2.5.1 Equipment/Supplies

- 1000 mL extraction body, drying adapter, concentrator, CLLE rack with condenser and water lines, water heater, water pump, chiller, Na₂SO₄, boiling chips, balance, deionized water, tridecane

3.2.5.2 Solvents

- Dichloromethane, H₂SO₄

3.2.5.3 Procedure

- Remove samples from designated storage area and allow to come to room temperature.
- Make sure the individual water supply valves on the CLLE rack are closed.
- Turn on the main pump valve to “extraction.” This valve is located inside the cabinet.
- Open the water return valve, located on the extraction rack near the tank.
- Add several boiling chips to each concentrator.
- Attach the extraction body, drying adapter and concentrator to the CLLE rack.
- Attach the water supply and return lines to the concentrator, and open the valves.

SGS Environmental Services

Standard Operating Procedure

- Turn on the pump power switch on the side of the cabinet to “extraction.”
- Turn on the water heater and allow the temperature to come up to 160°C.
- Make sure the chiller is running at the proper temperature (10°C).
- Add 250 mL of dichloromethane to each extraction body.
- Check and record the pH of the sample.
- Place sample jar on the balance and zero the scale.
- Add the contents of the sample bottle to the extraction body.
- Reweigh the sample jar on the zeroed scale and record the weight of the displaced sample volume.
- Rinse the jar with 50 mL of methylene chloride and add to the extraction body.
- Add 2 mL of 5:1 H₂SO₄ to each sample to lower the pH (<2).
- Spike the sample with extraction standards as listed on the spike profile.
- Attach the condenser to the extraction body and open the stopcock.
- Allow the samples to extract for 4-6 hours.
- When the extraction is complete, close the stopcock and allow the extract to concentrate down to approximately 1 mL.
- Turn off the water heater.
- Turn off the power to the water pump.
- Close the main return valve.
- Switch the main pump valve to “drain.”
- Turn on the power to the “drain” pump.
- Crack open the individual water return valves to allow air in, and water to drain out.
- Run the “drain” pump until the water has completely drained.
- Turn off the power to the “drain” pump.
- Close the individual water supply and return valves.
- Turn off the chiller.
- Transfer the extract to a 60 mL vial containing the cleanup standards and 100 uL tridecane.
- Properly dispose of the remaining sample water and dichloromethane.
- Concentrate the extract to near dryness in the Turbovap (3.6.3).

3.2.6 Milk Extraction

3.2.6.1 Equipment/Supplies

- Centrifuge, 40-mL vials, 4-oz jar, glass funnel.

3.2.6.2 Solvents/Reagents

- Hexane, Ether, Ethanol, Potassium Oxalate, sodium sulfate, Conc. H₂SO₄.

3.2.6.3 Procedure

- Add 90 mL of milk to a 4-oz jar.
- Add ES.
- Shake well and let stand for 10 min.
- Transfer the milk inside six 40-mL vials (15 g per vial).
- Add to each vial 2 mL of potassium oxalate solution (1800 mg in 12 mL water; or 20 mg per gram of milk).
- Shake.
- Add 15 mL ethanol to each vial.
- Shake.
- Add 5 mL hexane.
- Centrifuge for 15 min. @ 2000 rpm.
- Draw the hexane directly into another 4-oz jar through a sodium sulfate funnel, combining all six vials.
- Repeat the hexane/centrifuge steps two more times.
- Finally, add 5 mL diethyl ether.
- Shake.
- Centrifuge.
- Draw the organic layer and combine with the hexane layers.
- Concentrate the hexane/ether inside the Turbovap.
- Dissolve the residue in hexane (25 mL).
- Treat the hexane two times with conc. H₂SO₄ using a 40-mL vial and 10 mL of acid.
- Centrifuge as needed.
- Pull off the hexane layer and Turbovap to near dryness.

3.2.7 Fish Oil

3.2.7.1 Equipment/Supplies

- 2-liter flask, sodium sulfate columns, vessels to receive the extract (approx. 300mL), 10mL disposable pipettes, Na₂SO₄, glass wool, acidic silica gel.

3.2.7.2 Solvent

SGS Environmental Services

Standard Operating Procedure

- Hexane.

3.2.7.3 Procedure

- Position columns over an appropriately labeled collection vessel containing 500 μ L of tridecane.
- Pipet 10g of sample into a flask. Record the actual amount. Make any notes pertaining to the sample matrix at this time (color, viscosity, etc.).
- Tilt the flask to coat the bottom with sample so that the nonane based additions of extraction standard and/or matrix spike, as listed in the spike profile, do not land on glass. (Blanks and OPRs should receive 50ml of hexane and 100 μ L of corn oil). Tilt the flask again as each spike is absorbed by the oil. Record all spike information.
- Add hexane to bring volume to 200ml. Swirl the flask to incorporate the oil.
- Begin to add the silica gel a little at a time, swirling each time to prevent clumps. As much as 100g of silica gel may be needed to treat 10g of oil. A thick, dark paste will form. The static extraction time should be at least 2 minutes, swirl the flask vigorously every 30 seconds. About a minute after the last swirl the hexane will supernate and can be decanted. Pour the hexane thru the sodium sulfate columns. This decantation should go as dry as possible.
- Add 50mL of hexane, swirl and decant, again, as dry as possible. Two additional extractions, each with 10 to 20mL of hexane should be performed.

3.2.7.4 Comments

The vessel used to collect the sample under each sodium sulfate column is usually a rotovap round bottom flask. These are large enough to hold the volume of hexane used. Rotovapping the extracts down to the tridecane is recommended over evaporation. Since so much acidic silica gel is used, it is a good idea to make a fresh lot for each batch so as not to deplete our regular supply.

3.2.8 Soy/Corn Oil

3.2.8.1 Equipment/Supplies

- Recyclable glass columns, paper clips, glass wool, celite, Carbpak B, 250 mL beakers, 60 mL vials

3.2.8.2 Solvent

- Hexane
- Toluene

3.2.8.3 Procedure

- Weigh out the required amount of sample into a 250 mL beaker. Use an equal amount of hexane for the LMB, OPR and OPRD.
- Spike the samples with the extraction standards as listed on the spike profile. **Record the ES lot#, its concentration, and the volume (μ L) spiked.**
- If the sample is significantly more viscous than hexane, dilute the sample with hexane (usually 1:1).
- Pack a glass column with glass wool, 3-4 cm celite, 1 gram Carbpak B, and a glass wool plug. Secure the glass wool at each end with a paper clip to prevent slipping.
- Secure the column in a clamp with the celite end up and place a "waste" beaker underneath it.
- Wet the column with hexane until it starts to drip into the waste beaker.
- Transfer the sample onto the column.
- When the entire sample has been transferred, rinse the sample beaker with 20 mL hexane and transfer to the column.
- Continue to rinse the column with hexane until all the oil has been rinsed off the column (approximately 20 mL).
- Remove the waste beaker and flip the column over so that the carbon end is up.
- Place a 60 mL vial under the column and elute with 50 mL toluene.
- Repeat with 4 more 50 mL aliquots of toluene.
- Concentrate the toluene and combine into one vial. Continue concentrating to dryness and exchange with hexane, then continue to cleanup steps.

Note: This extraction procedure was designed to accommodate large amounts of oil (50-100 g). For smaller amounts of oil, the sample may be added directly to a PCU cleanup column.

3.2.9 Wipe Extraction

3.2.9.1 Equipment/Supplies

- 4 oz. amber glass jars, filter paper, table shaker and Pasteur pipette.

3.2.9.2 Solvent

- Toluene
- Tridecane

3.2.9.3 Procedure

- Add one piece of filter paper to each jar to be used for lab QC.
- Samples received in containers suitable for use as extraction jars may be left in those containers. Samples received in foil must be transferred to jars with one solvent rinse of the foil.

SGS Environmental Services

Standard Operating Procedure

- Spike the wipe with the extraction standards as listed on the spike profile and allow to absorb. **Record the ES lot#, its concentration, and the volume (µL) spiked.**
- Spike the appropriate samples with matrix spike as listed on the spike profile and allow to absorb. **Record the MX lot#, its concentration, and the volume (µL) spiked.**
- Add toluene to cover the wipe, seal, and shake for 1 hour.
- Label 60mL vial with sample ID number and add 500 µL tridecane. **Record the tridecane lot# and volume added.**
- Remove the jars from the shaker and individually transfer the solvent to the matching vial.
- Refill each jar with toluene, seal, and shake for 30 minutes.
- Observe the solvent level in each vial and concentrate as to facilitate adding the second extraction's solvent.
- Label another set of vials to hold the archived portion of each sample.
- Remove the jars from the shaker and individually transfer the solvent to the vial containing the solvent from the first extraction.
- Split the extracts equally into the archive vials. Store the archive fraction.
- Spike the vial containing the half of the extract to be processed for analysis with the cleanup standards as listed on the spike profile. **Record the CS lot#, its concentration, and the volume (µL) spiked.**
- Concentrate the extract in preparation for cleanup.

3.2.10 Synthetic Precipitation Leaching Procedure

3.2.10.1 Equipment/Supplies

- 2.2L extraction vessels, rotary agitator, filtration device, pH meter, balance, graduated cylinder, disposable lid-liners for extraction vessels, pre-cleaned 0.6-0.8µm Pyrex filters.

3.2.10.2 Reagents

- 60/40 sulfuric/nitric acid mixture, 1M sodium carbonate solution, DI water, acetone.

3.2.10.3 Procedure

- For Eastern Fluid use 20µL of the acid mix and 200µL of the Na₂CO₃ solution per liter of DI water. Stir the fluid well and make any fine pH adjustments down using 5µL of the acid mix or up using 20µL of the Na₂CO₃ solution. The pH of this fluid should be between 4.15 and 4.25.
- Only particles less than 9.5 mm in diameter should be used for the extraction. If the sample contains particles larger than this, they may either be discarded from the aliquot to be extracted or crushed to meet the requirements.
- A dry weight should be reported for each sample that is leached, unless the sample appears to have less than 10% moisture. The amount of sample used for the extraction can then be adjusted based on the amount of moisture contained in the sample.
- If a sample has a dry weight of less than 90%, the following formula must be used to calculate the minimum amount of sample to be extracted: $(50g * 100\% \text{ solids}) = \text{min. amount}$.
- Once the appropriate weight has been calculated, use the following formula to calculate the appropriate amount of extraction fluid to add to the sample: $(20 * \text{dry weight } \% * \text{actual weight used}) / 100 = \text{mL of extraction fluid}$
- If a sample has a dry weight equal to or greater than 90%, 50g of sample and 1000mL of extraction fluid may be assumed for the extraction.
- After adding both sample and extraction fluid to the appropriate vessel, lid-liners are placed over the opening, and the cap is screwed on over the liners. Be sure the caps are secured tightly to prevent leakage.
- Samples are tumbled using a rotary agitator for 18 ± 2 hours.
- Record the temperature of the ambient air around the rotary agitator (ref. form DC13).
- Record the times at which the agitator was both started and stopped.
- Separate the material in the extractor vessel into its liquid and solid phases by filtering through a Pyrex filter. The filter may be changed to facilitate filtration.
- All asterisked (*) data on form DC13 shall also be stored electronically. Review electronic data in the 'prep' table for accuracy and completeness.

3.2.11 Tocopherol Oil

3.2.11.1 Equipment/Supplies

- 2-liter flask, sodium sulfate, vessels to receive the extract (approx. 300mL), 10mL disposable pipettes, Na₂SO₄, glass wool, florisil.

3.2.11.2 Solvent

- Hexane.
- DCM

3.2.11.3 Procedure

- Weigh out the required amount of sample (nominally 20g for Dioxin analysis) into a 250 mL flask and add 50 mL of Hexane. Use an equal amount of hexane for the LMB, OPR and OPRD.

SGS Environmental Services

Standard Operating Procedure

- Spike the samples with the extraction standards as listed on the spike profile. **Record the ES lot#, its concentration, and the volume (µL) spiked.**
- The use of mild heat and sonication may be required to speed the dissolving of the oil in the solvent.
- Pack a giant glass column with glass wool, 2 cm salt, 3" of florisol, and 1 cm of salt.
- Secure the column in a clamp.
- Position a waste collection beaker.
- Wet the column with 30 mL hexane until it starts to drip into the waste beaker.
- Transfer the sample onto the column.
- When the entire sample has been transferred, rinse the sample beaker with 2 mL hexane and transfer to the column.
- Continue to rinse the column with hexane until all the oil has been rinsed off the column (approximately 60 mL).
- Remove the waste beaker and position a sample collection flask.
- Elute the sample with 240 mL dichloromethane.
- Concentrate the DCM and combine into one 60 mL vial.
- Add 50 µL of keeper to each vial. Continue concentrating to dryness and exchange with hexane, then continue to cleanup steps.

Note: This extraction procedure was designed to accommodate large amounts of oil (5-20 g). For smaller amounts of oil, the sample may be added directly to a PCU cleanup column.

3.3 Splitting/Archiving Extracts

3.3.1 Supplies

- Graduated tube, centrifuge tube, Pasteur pipette, glass wool.

3.3.2 Procedure

- Label the graduated and centrifuge tubes as required.
- Using a Pasteur pipette and a glass wool plug, filter the tridecane residue obtained from after concentration directly to the graduated or calibrated tube (e.g., 3.5- and 7-mL marks).
- Add approximately 1 mL of hexane to the sample container, swirl the solvent and transfer to the Pasteur pipette.
- Add another 1 mL hexane inside the RB flask, swirl, and wash the RB flask walls using the pipette.
- Transfer the rinse to the Pasteur pipette.
- Collect all rinses directly in the graduated tube.
- Make sure the volume reaches the 7-mL mark.
- Transfer 3.5 mL to the centrifuge tube or any container suitable for the fractionation.
- Seal the graduated tube using a TeflonTM-lined cap and store the sample inside the refrigerator.

Notes:

- To prevent sample spills, always secure the sample tube and the Pasteur pipette to a rod using easily adjustable clamps.
- In the presence of particulates, it is necessary to rinse the particulates with toluene, and repeat the concentration and solvent exchange steps. **Do not Split/Archive an inhomogeneous sample.**

3.4 Spiking

3.4.1 Equipment/Supplies

- 10 µl – 1000 µl automated pipette.

3.4.2 Procedure

- In each case, add the appropriate amount of standards for the method in use. A 'witness' must be present.
- Note: the composition and amount of standards used may be subject to change based on method requirements and individual client needs. Refer to the spike profile document and project folder for any changes.
- If the sample to be spiked is depleted or if only a limited volume remains, the witness must verify the ID of the sample prior to spiking. The extractionist is responsible for identifying such samples to the witness.
- If the samples to be spiked are air samples (i.e. M23 or TO9A) then the witness must complete a separate form detailing further duties.

3.5 Pipette Calibration

3.5.1 Equipment/Supplies

- 10 µl – 1000 µl automated pipette.
- Analytical Balance
- D.I. water

3.5.2 Procedure

- Pipettes are to be calibrated quarterly.
- The pipettes are calibrated at the high and low volume settings that are provided for each pipette model in the manual.
- D.I. water is transferred into the analytical balance and the results recorded in the Wet Lab Maintenance Logbook five times for each volume setting.

SGS Environmental Services

Standard Operating Procedure

- The average of the five replicates must fall within the acceptable range provided in the pipette manual for each model and volume.
- Pipettes that pass both volume settings will be labeled with the data of calibration and the initials of the calibrator.
- Pipettes that fail will be removed from service until an acceptable calibration is performed.
- If the calibration passes one calibration range but not the other, that pipette may be used for the passing volume only.
- New or repaired pipettes must pass fully before entering service.

3.6 Concentration

3.6.1 Turbovap

3.6.1.1 Equipment

- Zymark Turbovap, vial racks.

3.6.1.2 Procedure

- Set the water bath to the appropriate temperature for the solvent used and allow the bath to come to temperature (approximately 45°C for hexane and methylene chloride, 65°C for toluene).
- Clean the nitrogen needles with CH₂Cl₂ before use.
- Place the 40- or 60-mL sample vials into the TurboVap. (Note: Be sure the correct vial rack is used.)
- Set the nitrogen at 1-2 psi, and set the time to allow the samples to evaporate down 5-10 mL. This will help prevent cross contamination. Once the samples have evaporated enough that solvent will not splash out of the vials, set the nitrogen at 5 psi and set the time to allow the sample to evaporate to near dryness. The TurboVap will beep to signal that time is up. Additional time may be added if samples are not completely evaporated.
- Once the samples have evaporated, remove the vials and rinse the nitrogen needles with toluene.

3.7 Sample Cleanup

3.7.1 Conventional

3.7.1.1 Equipment/Supplies

- Pipettes (25 mL and 10 mL), Turbovap, acid- and base-coated silica, alumina, Florisil, carbon/celite.

3.7.1.2 Solvent

- Hexane, methylene chloride (MC), toluene.

3.7.1.3 Procedure

- Assemble the acid/base silica column in a 25 mL pipette as follows from bottom: glass wool, 1 g SiO₂, 4 g NaOH coated silica, 1 g SiO₂, 8 g H₂SO₄ coated silica, 2 g SiO₂, 4 g Na₂SO₄.
- Wash column with 50 mL hexane.
- Load sample and two 1-mL rinses of hexane.
- Elute with 100 mL hexane.
- The eluate is concentrated to near dryness and is now ready for the alumina column.
- The alumina column is prepared in a 10 mL pipette using 6 g acidic alumina and 4 g sodium sulfate.
- Load sample and two hexane rinses onto alumina column.
- Wash with 2%.
- Elute with MC to collect dioxins/furans.
- Turbovap to near dryness.
- Assemble the carbon column in a 10 mL pipette using 6 g carbon/celite with glass wool plugs.
- Load the sample onto the column with hexane, and allow the hexane to run completely through the column.
- Invert the column and elute with toluene.
- Turbovap the sample in toluene to dryness.

3.7.2 PCU-F

3.7.2.1 Equipment/Supplies

- Recyclable glass columns, Turbovap, acid- and base-coated silica, sodium sulfate, Florisil (store supplies inside dessicator).

3.7.2.2 Solvent

- Hexane, DCM.

3.7.2.3 Procedure

- Assemble the acid/base silica column.
- Assemble the florisil column.
Note: For tissue samples (e.g., fish) use the anthropogenic isolation column.
- Assemble waste collection setup.

SGS Environmental Services

Standard Operating Procedure

- Wet columns with hexane.
 - Fill 2% line.
 - Flush center fitting.
 - Load sample plus two vial rinses.
 - Elute acid base column with hexane.
 - Elute florisil with 2%.
 - Position collection vial.
 - Elute sample with 100% CH₂Cl₂.
- Note: To perform method, start PCU software, load method APC-2000.txt, and hit start. When method is complete, concentrate samples using Turbovap.

3.7.3 PCU-A

3.7.3.1 Equipment/Supplies

- Recyclable glass columns, TurboVap, acid- and base-coated silica, sodium sulfate, alumina (store supplies inside dessicator).

3.7.3.2 Solvent

- Hexane, Methylene Chloride.

3.7.3.3 Procedure

- Assemble the acid/base silica column.
 - Assemble the alumina column.
- Note: For tissue samples (e.g., fish) use the anthropogenic isolation column.
- Assemble waste collection setup.
 - Wet columns with hexane.
 - Fill 2% line.
 - Flush center fitting.
 - Load sample plus two vial rinses.
 - Elute acid base column with hexane.
 - Elute alumina with 2%.
 - Position collection vial.
 - Elute sample with 100% CH₂Cl₂.

Note: To perform method, start PCU software, load method PCU-abal.txt, and hit start. When method is complete, concentrate samples using Turbovap.

3.7.4 Acid Treatment for Lipids

3.7.4.1 Equipment/Supplies

- Separatory funnel (250 mL), vacuum concentrator (VC), glass funnel, rotovap flask, conc. H₂SO₄, sodium sulfate.

3.7.4.2 Solvent

- Hexane.

3.7.4.3 Procedure

- This section assumes that the extraction was completed with methylene chloride and the solvent **completely** removed under reduced vacuum. The lipids content of the sample is determined before engaging in the step that follows.
- Use 200 mL hexane per 10 g of lipids/fat.
- Add the hexane to the fat and allow the fat to dissolve completely.
- Transfer to a 250-mL separatory funnel and add two 5-mL rinses.
- If the volume of hexane is above 200 mL, repeat what follows on 100-mL (or less) portions.
- Add carefully 50 mL of conc. H₂SO₄ (or 50 mL per 100 mL hexane or 10 g fat).
- Mix gently the two phases; do not shake hard to prevent the formation of emulsions. Tipping the separatory funnel and releasing vapors is done gently several times. Let the system settle before drawing the lower acid layer to wastes. Repeat this step two more times with another 2 x 50-mL of acid. All collected acid layers are discarded slowly in the sink by allowing tap water to run abundantly.
- The hexane layer is passed through a sodium sulfate funnel directly into a rotovap flask.

3.7.5 Florisil Column Cleanup

3.7.5.1 Equipment/Supplies

- Recyclable glass columns, Turbovap, sodium sulfate, Florisil (store supplies inside dessicator).

3.7.5.2 Solvent

- Hexane, Methylene Chloride.

3.7.5.3 Procedure

- Assemble the bottom column as follows from bottom: filter, florisil.

SGS Environmental Services

Standard Operating Procedure

- Assemble the top column as follows from bottom: filter, sodium sulfate.
 - Position waste collection assembly.
 - Wet columns with 2%.
 - Wet columns with hexane.
 - Load sample plus two vial rinses.
 - Elute florisil column with hexane.
 - Elute with 2%.
 - Position collection vial.
 - Elute sample with 100% CH₂Cl₂.
- Note: To perform method, start PCU software, load method fonly3.txt, and hit start. When method is complete, concentrate samples using Turbovap.

3.7.6 Alumina Column Cleanup

3.7.6.1 Equipment/Supplies

- Recyclable glass columns, Turbovap sodium sulfate, alumina (store supplies inside dessicator).

3.7.6.2 Solvent

- Hexane, Methylene Chloride.

3.7.6.3 Procedure

- Assemble bottom column as follows from bottom: filter, alumina.
 - Assemble top column as follows from bottom: filter, sodium sulfate.
 - Position waste collection assembly.
 - Fill 2% line.
 - Wet columns with hexane.
 - Load sample plus two vial rinses.
 - Elute alumina column with hexane.
 - Elute with 2%.
 - Position collection vial.
 - Elute sample with 100% CH₂Cl₂.
- Note: To perform method, start PCU software, load method a-only.txt, and hit start. When the method is complete, concentrate samples using Turbovap.

3.7.7 Carbon Cleanup

3.7.7.1 Equipment/Supplies

- Disposable glass pipettes, glass wool, celite, carbon/celite mixture

3.7.7.2 Solvent

- Methylene Chloride, Toluene, Binary mixture (50% Methylene Chloride:50% Hexane), Tertiary mixture (75% Methylene Chloride:20% Methanol:5% Toluene)

3.7.7.3 Procedure

- Assemble the carbon column by placing a glass wool plug in the disposable pipette, add 5 mm of celite, 5 mm carbon/celite mixture, then another plug of glass wool.
- Secure the column in a clamp and place a "waste" jar underneath the column. The column should be positioned so that the celite end is up.
- Rinse the column with 4 mL of toluene. Allow the solvent to run completely through the column. Continue to rinse the column with 2 mL of tertiary, 4 mL of binary, and 2 mL methylene chloride. Be sure to let the solvent run completely through the column between each rinse, but do not let the column go dry.
- Load the sample onto the column with three 1-2 mL rinses of methylene chloride.
- Rinse the column with 10 mL binary, then 5 mL tertiary.
- Flip the column over so that the celite end is down. Remove the waste jar and position a collection vial underneath the column.
- Elute the sample off the column with 20 mL toluene.

3.7.8 Drinking Water Cleanup

3.7.8.1 Equipment/Supplies

- 10 mL drying column, H₂SO₄ treated silica gel, glass wool, sodium sulfate, 60mL vial.

3.7.8.2 Solvent

- Methylene chloride, tridecane

3.7.8.3 Procedure

- Assemble the silica column as follows from bottom: glass wool, 1gram H₂SO₄ treated silica, sodium sulfate.
- Pre-clean the column with three 10mL rinses of DCM and drain into the appropriate solvent waste container.

SGS Environmental Services

Standard Operating Procedure

- Place a 60mL vial containing 10 μ L of tridecane under column.
- Load sample with methylene chloride.
- Rinse concentrator with three 2mL rinses of methylene chloride.
- Rinse column twice with 10mL of methylene chloride.
- Turbovap the sample to .5mL and transfer to GC vial.

3.7.9 Wipe Cleanup

3.7.9.1 Equipment/Supplies

- Silica gel
- Acid Treated silica gel
- Florisil
- 10mL disposable pipette
- Glass wool

3.7.9.2 Solvents

- Dichloromethane
- Hexane

3.7.9.3 Procedure

- Ensure that a keeper is present in the samples. Blow them down and exchange into hexane.
- Add a glass wool plug to each column to be prepared and attach it to the rack.
- Fit a funnel into the end of each column to facilitate the addition of cleanup media.
- Add a "Pinch" of untreated silica. (Measuring scoops provided.)
- Add a "Dash" of florisil.
- Add a "Pinch" of acid treated silica.
- Add a "Pinch" of sodium sulfate.
- Position waste collection vessel(s).
- Wet the column with hexane, and then add the sample.
- Add 2mL of Hexane and allow the eluent to flow to waste.
- Add 4 x 5mL aliquots of 2%DCM/Hexane.
- Position sample collection vial.
- Add 4 x 5mL of DCM.
- Turbovap the sample to 0.5 mL and transfer to GC vial.

3.7.10 Florisil Cleanup for Method 1668A

3.7.10.1 Summary

PCB sample extract cleanup is accomplished by passing the extract through the column under controlled conditions described below.

3.7.10.2 Equipment/Supplies

- Florisil column (sec.2.12)
- Labeled 60mL reusable vials with snyder column
- Disposable glass pipettes
- Water bath
- Fume hood
- Tridecane
- Teflon boiling chips

3.7.10.3 Solvent

- Hexane
- 6% ether:hexane

3.7.10.4 Procedure

- Position waste collection vial under florisil column.
- Wet column with 20mL hexane and allow solvent to flow to waste.
- Wet column with 10mL hexane and allow solvent to flow to waste.
- Transfer sample from concentrator to column using disposable pipet.
- Rinse the sample vial twice with 1mL portions of hexane and apply to column.
- Position properly labeled vial under column.
- Elute sample from column with 50mL of 6% ether:hexane.
- Position 2nd labeled vial under florisil and elute with another 50mL of 6% ether:hexane.
- Add boiling chips to collection vials.
- Both vials should be concentrated using snyder column and water bath inside a fume hood.
- Vials should be combined and transferred to a labeled GC vial containing 20 μ L of tridecane.

3.7.11 Acid silica cleanup for 1668A (mini silica)

3.7.11.1 Summary

SGS Environmental Services Standard Operating Procedure

PCB sample extract cleanup is accomplished by passing the extract through the column under controlled conditions described below.

3.7.11.2 Equipment/Supplies

- 25mL acid silica cleanup column (sec. 2.13)
- Disposable pipettes
- Water bath
- Fume hood
- Tridecane
- Turbo evaporator
- 60mL reusable vials with snyder column

3.7.11.3 Solvent

- DCM

3.7.11.4 Procedure

- Position waste collection vial under silica column.
- Rinse the column with 10mL of DCM and allow to flow to waste.
- Rinse the column with 5mL of DCM and allow to flow to waste.
- Position properly labeled vial under column.
- Load sample directly from concentrator.
- Rinse concentrator with 2x 1mL rinses of DCM and apply to column.
- Flush column with 3mL of DCM.
- Add teflon boiling chips to collection vial.
- Vial should be concentrated using snyder column and water bath inside a fume hood.
- Sample should be transferred to a labeled GC vial containing 20 μ L of tridecane.

3.7.12 Acid silica with florisil cleanup for dioxins

3.7.12.1 Summary

Dioxin sample extract cleanup is accomplished by passing the extract through the column under controlled conditions described below.

3.7.12.2 Equipment/Supplies

- 25mL drying column
- Florisil
- Acid coated silica gel
- Silica gel
- Sodium sulfate
- Glass wool
- Disposable pipettes
- Turbo evaporator
- 60mL vials.

3.7.12.3 Solvent

- Hexane
- Dichloromethane

3.7.12.4 Procedure

- Assemble the column as follows from bottom: glass wool, 5mm silica, 15mm florisil, 5mm silica, 80mm acid silica, 10mm sodium sulfate.
- Position waste collection vial under silica column.
- Rinse the column with 2x 10mL of dichloromethane and allow to flow to waste.
- Rinse the column with 2x 10mL of hexane and allow to flow to waste.
- Load the sample from the 60mL vial.
- Rinse vial with 2x 1mL rinses of hexane and apply to column.
- Flush column with 2x 20mL of hexane.
- Position properly labeled 60mL vial under column.
- Elute sample with 50mL of dichloromethane.
- Vial should be concentrated using the turbovap.
- Sample should be transferred to a labeled GC.

SGS Environmental Services
Standard Operating Procedure

Standard Operating Procedures
For Analysis & Reporting (8290)

Issue date: 03/28/08

Revision: 17

SGS Environmental Services
5500 Business Drive
Wilmington, North Carolina 28405

Approved by:

Name, Last Updated By

Date

Name, Laboratory Manager

Date

Name, QA/QC Manager

Date

SGS Environmental Services

Standard Operating Procedure

Purpose

To describe the processes used in operating the HRGC/HRMS system, as well as the procedures followed in the generation, interpretation and review of laboratory data for Method 8290.

Summary

This SOP details how to analyze and report samples by EPA Method 8290 (Analyte list found in Table 1). HRGC/HRMS is used to detect and quantify PCDD/Fs. Samples arrive at the MS lab having been extracted and fractionated using procedures in Section 3. Analyses are grouped into 12-hour runs, which include analyses of samples and standards mixtures. Upon completion of the run, the analyst reviews the data associated with both standards and samples in order to confirm the validity of the run and to determine any potential need for re-analysis or re-extraction. The analyst generates quantitation reports and chromatograms using sophisticated software. These reports are used to generate forms that summarize the results of the analysis.

Table 1: Analyte List with Reporting Limits per Matrix

Analyte	Solid (pg/g)	Aqueous (pg/L)
2378-TCDD	1	10
12378-PeCDD	5	50
123478-HxCDD	5	50
123678-HxCDD	5	50
123789-HxCDD	5	50
1234678-HpCDD	5	50
OCDD	10	100
2378-TCDF	1	10
12378-PeCDF	5	50
23478-PeCDF	5	50
123478-HxCDF	5	50
123678-HxCDF	5	50
234678-HxCDF	5	50
123789-HxCDF	5	50
1234678-HpCDF	5	50
1234789-HpCDF	5	50
OCDF	10	100

4.1 Operation of HRGC/HRMS

4.1.1 Equipment

- HP6890 GC, Micromass Autospec Ultima high resolution mass spectrometer, vortex mixer, 10-100 uL pipette

4.1.2 Procedure

- Recall the GC temperature/pressure/flow program.
- Recall the MS experiment (see Table 2).
- Perform any necessary maintenance.
- Tune the MS resolution to 100 ppm at 5% height.
- Acquire location data to calibrate the MS and print a copy of function one MS resolution.
- Inject the window defining/GC resolution/continuing calibration mix (RETCON). Evaluate descriptor-switching times for accuracy. If any window defining peaks have shifted outside the descriptor windows, adjust the switching times before injecting any samples. This injection is also used to verify that there is less than or equal to 25% peak to valley for the close eluter of 2,3,7,8-TCDD. Print a copy of the GC resolution check. If the valleys are within specifications, proceed to calibrate or verify a previous calibration. If not, further investigation and/or maintenance may be required. Re-inject this solution after maintenance to check for improvement.
- Now that the GC/MS resolution and descriptor switching times have been verified, a series of five initial calibration standards may be injected and reviewed for method requirements. If an initial calibration already exists, a RETCON may be analyzed to verify continuing calibration. If the curve or the RETCON passes method requirements, sample analysis may begin.
- Reconstitution of a sample is accomplished by adding nonane containing the injection standards (JS), capping the vial, and mixing well with a vortex mixer. The amount of standard to be added can be found in the spike profile document.
- Samples are injected under conditions identical to those used to establish calibration.
- A "back-end" RETCON must be injected within 12 H from the front end RETCON or the RETCON in the curve. This standard is used to verify sufficient stability of the calibration after sample analysis. It

SGS Environmental Services

Standard Operating Procedure

has requirements set by the method. Depending on the back-end CS3 results, different calibration files maybe required to quantify the samples.

- A “back-end” print out of the MS resolution must also be performed.
- The calibration data from a sequence is filed in a folder cabinet under the day it was analyzed and includes the all GC/MS resolution checks, window verification, valley verification, front/back end RETCONS, runlogs and window defining mix (WDM) retention time sheets.
- Each sample hardcopy should include the quant report, totals pages, deviations, chromatograms, and report forms.
- GC Column: DB-5MS, 60 m, id 0.25 mm, 0.25 μ m.

SGS Environmental Services
Standard Operating Procedure

Table 2: Mass Descriptors used for Selected Ion Recording HRMS

Function (#)	Channel (#)	Mass (amu)	Dwell Time (ms)	I.C. Delay (ms)
1	1	303.9016	100	20
1	2	305.8987	100	10
1	3	315.9419	40	10
1	4	316.9824	20	10
1	5	316.9824	(Lock)	50
1	6	317.9389	40	10
1	7	319.8965	100	10
1	8	321.8936	100	10
1	9	327.8847	40	10
1	10	331.9368	40	10
1	11	333.9339	40	10
1	12	375.8364	30	20
2	1	339.8597	100	20
2	2	341.8568	100	10
2	3	351.9	40	10
2	4	353.897	40	10
2	5	355.8546	100	10
2	6	357.8517	100	10
2	7	366.9792	20	10
2	8	366.9792	(Lock)	50
2	9	367.8949	40	10
2	10	369.8919	40	10
2	11	409.7974	30	20
3	1	373.8207	100	20
3	2	375.8178	100	10
3	3	380.976	20	10
3	4	380.976	(Lock)	50
3	5	383.8639	40	10
3	6	385.861	40	10
3	7	389.8156	100	10
3	8	391.8127	100	10
3	9	401.8559	40	10
3	10	403.853	40	10
3	11	445.7555	30	20
4	1	407.7818	100	20
4	2	409.7788	100	10
4	3	417.8253	40	10
4	4	419.822	40	10
4	5	423.7767	100	10
4	6	425.7737	100	10
4	7	430.9728	20	10
4	8	430.9728	(Lock)	50
4	9	435.8169	40	10
4	10	437.814	40	10
4	11	479.7165	30	20
5	1	441.7427	100	20
5	2	443.7398	100	10
5	3	454.9728	20	10
5	4	454.9728	(Lock)	50
5	5	457.7377	100	10
5	6	459.7348	100	10
5	7	469.778	40	10
5	8	471.775	40	10
5	9	513.6775	30	20

4.2 Data Generation, Interpretation and Review

SGS defines a batch of samples as no more than 20 samples processed with in a 12-hour shift. One LMB and one OPR are processed in every analytical batch, following the same procedures used for the field samples. Generally, soil is replaced by salt (Na₂SO₄), effluent by deionized water and biological tissues by vegetable oil. An invalid LMB or OPR requires a re-extraction of the affected samples.

4.2.1 Interferences

Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms. The use of high purity reagents and solvents helps minimize interference problems. All new materials used in the analysis shall be demonstrated to be free from interferences by running an initial reagent blank. Interferences co-extracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the analytes. The elimination of interferences is essential. Cleanup steps are used to reduce or eliminate these interferences and thereby permit reliable determination of the analyte as close as possible to the specified sensitivity.

4.2.2 Method Detection Limit Study and IPR Schedule

SGS Environmental Services Standard Operating Procedure

On an annual schedule, the laboratory shall perform a Method Detection Limit study (MDLs) utilizing a solid and aqueous matrix. MDLs are to be calculated for each 2,3,7,8-substituted congener. All MDL studies will be conducted following the guidelines set forth in 40 CFR, Part 136, appendix B. An MDL verification sample will be analyzed concurrently at an estimated 2 to 3 times the expected calculated MDL value for use with USACE clients. In addition, at the time of the MDL a continuing calibration will be analyzed at a concentration other than the normally utilized CS3 to further demonstrate instrument performance.

Each analyst is required to successfully complete an Initial Demonstration of Precision and Accuracy (IPR) before working on samples. An IPR is conducted for each matrix and extraction technique utilized. IPRs are repeated with each relevant change to a technique.

4.2.3 Initial Calibrations

On an annual schedule, SGS uses the concentrations in Table 4 to construct the initial calibration. The percent relative standard deviations for the mean response factors from the seventeen unlabeled standards must not exceed +/- 20%. The percent relative standard deviations from the labeled standards (i. e. extraction standards, cleanup standards and sampling standards) must not exceed +/- 30%. The signal to noise ratio for all signals present must be ≥ 10 . The ion abundance ratios must be within specified control limits (see Table 3). SGS uses the concentrations in Table 4 to construct the initial calibration. The concentration of the lowest calibration point is a modification of the referenced method. It is lower than required.

Table 3. Theoretical Ion Abundance Ratios and Their Control Limits

Level of Chlorination	Theoretical Ratio	Control Limits	
		Lower	Upper
4	0.77	0.65	0.89
5	1.55	1.32	1.78
6	1.24	1.05	1.43
6 ^a	0.51	0.43	0.59
7	1.04	0.88	1.20
7 ^b	0.44	0.37	0.51
8	0.89	0.76	1.02

^a Used only for ¹³C-HxCDF

^b Used only for ¹³C-HpCDF

A new initial calibration is required when the continuing calibration criteria below are not met. Routine maintenance may be performed to correct any failures. Any major maintenance to the analytical system such as slit cleaning, analyzer lens cleaning, magnet shifts, and detector disk changes warrant a new ICAL. At a minimum, a new initial calibration must be performed annually.

SGS Environmental Services
Standard Operating Procedure

Table 4. Initial Calibration Concentrations
Concentration (pg/ μ L)

Analyte	CS-1	CS-2	CS-3	CS-4	CS-5
<u>Unlabeled</u>					
2378-TCDD	0.25	2	10	40	200
2378-TCDF	0.25	2	10	40	200
12378-PeCDD	1.25	10	50	200	1000
12378-PeCDF	1.25	10	50	200	1000
23478-PeCDF	1.25	10	50	200	1000
123478-HxCDD	1.25	10	50	200	1000
123678-HxCDD	1.25	10	50	200	1000
123789-HxCDD	1.25	10	50	200	1000
123478-HxCDF	1.25	10	50	200	1000
123678-HxCDF	1.25	10	50	200	1000
123789-HxCDF	1.25	10	50	200	1000
234678-HxCDF	1.25	10	50	200	1000
1234678-HpCDD	1.25	10	50	200	1000
1234678-HpCDF	1.25	10	50	200	1000
1234789-HpCDF	1.25	10	50	200	1000
OCDD	2.5	20	100	400	2000
OCDF	2.5	20	100	400	2000
<u>Extraction Standards</u>					
¹³ C-2378-TCDD	100	100	100	100	100
¹³ C-2378-TCDF	100	100	100	100	100
¹³ C-12378-PeCDD	100	100	100	100	100
¹³ C-12378-PeCDF	100	100	100	100	100
¹³ C-123678-HxCDD	100	100	100	100	100
¹³ C-123478-HxCDF	100	100	100	100	100
¹³ C-1234678-HpCDD	100	100	100	100	100
¹³ C-1234678-HpCDF	100	100	100	100	100
¹³ C-OCDD	200	200	200	200	200
<u>Cleanup Standards</u>					
³⁷ Cl-2378-TCDD	0.25	2	10	40	200
¹³ C-23478-PeCDF	100	100	100	100	100
¹³ C-123478-HxCDD	100	100	100	100	100
¹³ C-123478-HxCDF	100	100	100	100	100
¹³ C-1234789-HpCDF	100	100	100	100	100
<u>Injection Standards</u>					
¹³ C-1234-TCDD	100	100	100	100	100
¹³ C-123789-HxCDD	100	100	100	100	100

4.2.4 Continuing Calibrations

Check that all paperwork is present. A CCal package should contain the documentation listed below.

- Pass: Run log. HRMS Resolution Checks. WDM retention time sheet. WDM chromatograms. GC performance for 2,3,7,8-TCDD. CCal quantitation page. CCal chromatograms. Injection preparation log
- Fail: The analyst listed on the run log can provide any missing paperwork.

SGS Environmental Services Standard Operating Procedure

Review the Run log.

- Pass: Check that the 12-hour windows have not been exceeded between the front end and back end CCals.
- Fail: Re-analysis of samples.

Review the HRMS Resolution checks.

- Pass: Verify 100ppm width at 5% height for PFK mass 318 or higher. Compare the resolution check times to those on the run log to be sure they bracket each sequence.
- Fail: Should a back-end resolution check fail, an assessment should be made to determine any data quality impact.

Review the Window Defining Mix and GC Performance Documentation.

- Pass: Check that the sample numbers on the WDM sheets match those on the runlog. Check that the retention times are correct for the WDM chromatograms.
- Check that the valley between 2,3,7,8-TCDD and its close eluter does not exceed 25%.
- Fail: Any missing peaks in the window-defining sample should be re-identified with a survey scan. Determine proper switching times. These must be entered into the HRMS ion function descriptors before analysis may resume. If the GC performance valley is greater than 25% instrument maintenance may be required. When a valley fails all samples must be re-injected.

Review the CCal Quantitation and Chromatograms.

- Pass: All ion ratios must be within the control limits given in Table 3. The measured RRFs obtained during the front and back-end CCals must be within $\pm 20\%$ of the mean values established during the ICAL for unlabeled standards, and within $\pm 30\%$ of the mean values for labeled standards. Quantitate using the ICAL when no compounds are above 20/30% on the back-end retcon. Quantitate using averaged CCALs when there are no more than two unrelated compounds above 20/30% but less than 25/35% on the back-end retcon. If an RRF is biased high, the CCal is acceptable for all samples in which the failed analyte is not detected
- Fail: Routine instrument maintenance such as installing new injection port hardware, inner source cleaning, retuning, column clipping etc. will usually correct a calibration failure. If these measures do not work, then the laboratory will demonstrate performance after corrective action with either two consecutive acceptable CCALs or a new initial calibration.

Review the Injection Prep log sheet.

- Pass: Check that all samples have been spiked with 2 ng injection standard. Verify that final volume is 20 uL. Be sure that any dilutions or other comments are noted
- Fail: Calculations of sample concentrations should reflect any deviations from normal injection prep parameters.

4.2.5 Quality Control Work Groups

The following elements should be present in a complete work group file:

- LMB topsheets
- LMB totals sheets
- LMB chromatograms
- OPR topsheets
- OPR chromatograms
- Extraction log sheet
- Cleanup log sheet
- Cleanup observation forms
- Dry weight sheet (where applicable)
- Any additional information (ex. re-extract request sheet)

The following procedure should be used for reviewing a work group:

- Review the header information on the LMB topsheets. Verify that the method and client sample ID (LMB or OPR) are correct.
- Review the footer information on the LMB and OPR topsheets. Verify that the following information is correct: extraction date, analysis date, method, matrix, sample weight/volume, percent solids/lipids, pH, work group number, sample data file, retcheck data file, beginning cal data file, ending cal data file and ical data file.
- Verify that no target analytes are present in the LMB above the lower method calibration limit (LMCL). Verify that no target analytes are present above one-half the LMCL for USACE projects. And, that no estimated detection limits (EDL) exceed the LMCL. If target analytes or EDL's are above this limit, the associated samples must have concentrations that exceed 10 times the LMB concentration for the specified analyte. Otherwise, samples must be re-extracted.
- Review the totals data for the LMB. Be sure that any ghosting peaks are removed from the totals concentrations and the associated detection limits are elevated to reflect the subtracted peaks.
- Verify that extraction standards are within method specifications (40-135%) for the LMB and OPR, found on the topsheets.
- Verify that unlabeled recoveries in the OPR meet SGS's recovery limits of 70-130%.

SGS Environmental Services Standard Operating Procedure

4.2.6 Data Review

For quick reference, refer to the chart below

4.2.6.1 Initial Review

- Summary form generation and organization of Ical, Ccal, Samples and QC data is conducted by the analyst.
- Analysts review data upon generation and elicit peer review.
- Data is then released to a secondary review level with the analyst's signature.

4.2.6.2 Final Review

- Assemble project samples and QC data
- Review client information and documentation.
- Complete Data Review Checklist (Section 4, Appendix B) and sign reports.
- Samples are released to clients with the data reviewer's signature after the complete package is reviewed by a QA Manager or Lab Director.

SGS Quality Control Requirements for Method 8290			
QC Check	Frequency	Limits	Corrective Action
Method Blank	One per extraction batch.	PCDD/F < SOP RL or <10% of level in sample.	Affected samples must be re-extracted.
Initial Calibration (ICAL)	As needed to maintain acceptable CCAL, after major maintenance, or at a minimum one per year.	20% RPD for native and 30% RPD for labeled species.	An acceptable ICAL must be established before sample reporting may begin.
Mass Resolution	Before and after each 12 hour analytical sequence.	Mass resolution must be at 10,000 as estimated from a printout of a PFK peak.	MS maintenance.
Descriptor Defining Isomers	At the beginning of each 12h analytical sequence.	First and last eluters must be present within the switching times..	Perform a survey scan to identify the correct switching times.
GC Performance Isomers	At the beginning of each 12h analytical sequence.	Must be <25% valley.	GC maintenance.
OPR (LCS)	One per extraction batch.	70-130 native.	Re-extract batch if low and detected. Evaluate data quality for high and non-detected.
OPRD (LCSD)	One per extraction batch.	70-130, ≤ 20%RSD.	If needed and fails, re-extract batch.
Extraction Standards (ES)	Every sample must receive ES before extraction.	40-135%.	Evaluate data quality, and if needed re-extract sample.
Continuing Calibration (CCAL)	Pre and post sample analysis within 12h.	20/30%. Average CCALs if no more than 2 unrelated compounds above 20/30% but less than 25/35% on the back end.	Acceptable bracketing CCALs must be established before sample reporting may begin.
Lab Sample Duplicate (DUP)	Per client request.	<25%Difference.	Flag failures.
Matrix spike/duplicate (MS/MSD)	One pair per method, per matrix, per extraction technique, per 30 days, per 20 samples.	±30% recovery, 20% RPD.	Flag failures, report OPRD.

4.2.7 Calculations

4.2.7.1 Relative Response Factor

- $RRF = \frac{(\text{Sum Ion Abun. of analyte})(\text{ES Amount})}{(\text{Sum Ion Abun. ES})(\text{Analyte Amount})}$

4.2.7.2 Percent Difference, as for CCal evaluation.

- $\%D = \frac{(\text{CCal RRF} - \text{ICal RRF})(100)}{\text{ICal RRF}}$

4.2.7.3 Target compound calculation

SGS Environmental Services Standard Operating Procedure

- $PCDD/PCDF \text{ (ppt)} = \frac{(\text{Sum Ion Abun. of analyte})(\text{ES Amount})}{(\text{Sum Ion Abun. of Int. Std})(\text{RRF from ICal})(\text{Amt. of Sample})}$
- $EMPC \text{ (ppt)} = \frac{(\text{Sum Ion Abun. of analyte})(\text{ES Amount})}{(\text{Sum Ion Abun. of Int. Std})(\text{RRF from ICal})(\text{Amt. of Sample})}$
- $EDL = \frac{3 (\text{Sum Height of Noise})(\text{Std. Amount})}{(\text{Sum Height of Int. Std})(\text{RF from ICal})(\text{Amt. of Sample})}$

The instrumentation software calculates the noise level. However, manual noise determination may be employed at the reviewer's discretion in order to more accurately report peaks of interest.

4.2.7.4 Extraction Standard Recovery Calculation

- $\% \text{ Recovery} = \frac{(\text{Sum Ion Abun. of ES})(\text{JS Amount})}{(\text{Sum Ion Abun. of JS})(\text{ES RRF from ICal})(\text{ES Amount})}$

The clean-up standard recoveries are calculated as above, substituting the ion abundances from the individual clean-up standard for the extraction standard.

4.2.8 Requests for Re-extraction

Post-analysis is an early place to discover the need for re-extraction, so it is important to review all supporting data. This includes spike profiles, extraction logs, clean-up logs, injection prep logs, observation forms, and the sample tracking forms in the folder. The project or work group folder may contain exceptions or changes to our routine spiking procedures.

One should also check the sample for problems relating to analysis. These problems include response factors that may introduce quantitative errors, interference that could be diluted out (yellow extracts), or any interference that causes de-tuning or chromatographic conditions that could lead to quantitative errors.

Many things can cause the need for a re-extraction. Extraction or clean-up recovery problems not detailed in the related paperwork indicate the need to talk to the person(s) involved. This type of communication can solve difficult problems and help to ensure that the re-extraction is planned well.

In planning a re-extraction the involvement of a technical reviewer is invaluable. Reviewers notice trends and problems that may call the need for re-extracting a batch of samples. They can give insights as to whether re-extraction will even help or if a different extraction approach should be considered.

The QA/QC officer should be consulted when re-extraction is considered so that the impact on other samples, or potentially a batch can be accessed.

The analyst will schedule the re-extraction(s) by generating a form indicating the information necessary for the lab to do the re-extraction. Some info includes the sample id, re-extraction due date, and reason for re-extraction (ref. form DC18).

4.3 Safety

Each employee must read, understand and follow the safety guidelines in the Chemical Hygiene Plan. For further safety precautions, please refer to the referenced test method.

4.4 References

Method 8290, Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry (HRGC/HRMS), Revision 0, September, 1994.

Controlled documents referenced for sample preparation include DC 01-12, 16-18, 24, 27-29 and 55.