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SAMPLING AND ANALYSIS PLAN: SUPPLEMENTAL INVESTIGATION OF THE LYMAN MILL REACH SEDIMENT AND FLOOD PLAIN SOILS

Centredale Manor Restoration Project Superfund Site in North Providence, Rhode Island

Prepared for
Emhart Industries, Inc.
c/o Sullivan & Worcester, LLP
1666 K Street, NW
Washington, DC 20006

Prepared by
Integral Consulting Inc.
45 Exchange Street
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June 21, 2010

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June 21, 2010

Project No. C656-0003

Ms. Anna Krasko
Project Manager
United States Environmental Protection Agency
5 Post Office Square, Suite 100
Mail Code OSRR07-1
Boston, MA 02109-3912

Subject: **Integral's Sampling and Analysis Plan for the Supplemental Investigation of the Lyman Mill Reach Sediment and Flood Plain Soils**

Dear Ms. Krasko:

Enclosed please find Integral Consulting Inc.'s Sampling and Analysis Plan (SAP) for the Supplemental Investigation of the Lyman Mill Reach Sediment and Flood Plain Soils. The components of the SAP include: 1) the Quality Assurance Project Plan; 2) the Field Sampling Plan; and 3) the Health and Safety Plan. The SAP has been prepared on behalf of Emhart Industries, Inc., and, though not fully executed, pursuant to the requirements of Administrative Settlement Agreement and Order on Consent ("Settlement Agreement"), EPA Docket No. 01-2010-0045.

In an effort to maintain momentum related the subject supplemental investigation, we are submitting the enclosed documents prior to the USEPA's final sign off on the Settlement Agreement. As such, the enclosed documents should be considered "draft final" until the USEPA has fully executed the Settlement Agreement.

Should you have any questions, please call me at (207) 874-9000 ext. 206.

Sincerely,

Patrick O. Gwinn
Senior Managing Scientist

cc: Louis Maccarone – RIDEM
Deirdre Dahlen – Battelle
Jeffrey Karp – Sullivan & Worcester, LLP
David Scotti – Loureiro Engineering Associates, Inc.

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Title and Approval Sheet

Quality Assurance Project Plan Approvals

EPA Remedial Project Manager:	Anna Krasko	_____	Date: _____
EPA Quality Assurance (QA) Reviewer:		_____	Date: _____
Respondents' Project Coordinator and Integral Project Manager:	Patrick Gwinn	_____	Date: _____
Emhart Industries Project Manger:	Linda Biagioni	_____	Date: _____
Integral Laboratory QA Coordinator:	Craig Hutchings	_____	Date: _____
Columbia Analytical Services Project Manager:	Jeff Christian	_____	Date: _____
Columbia Analytical Services QA Manager:	Julie Gish	_____	Date: _____
Vista Analytical Laboratory Project Manager:	Martha M. Maier	_____	Date: _____
Vista Analytical Laboratory QA Manager:	Rose M. Harrelson	_____	Date: _____

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ACRONYMS AND ABBREVIATIONS

ASTM	American Society for Testing and Materials
CLP	Contract Laboratory Program
CMRP Site	Centredale Manor Restoration Project Superfund Site
COC	chain-of-custody
EDD	electronic data deliverable
EDL	estimated detection limit
Emhart	Emhart Industries, Inc.
FSP	Field Sampling Plan
GC/ECD	gas chromatography/ electron capture detector
GPS	global positioning system
HAZWOPER	Hazardous Waste Operations and Emergency Response
HSP	Health and Safety Plan
HRGC/HRMS	high-resolution gas chromatography with high-resolution mass spectrometry
Integral	Integral Consulting Inc.
LIMS	laboratory information management system
MDL	method detection limit
MRL	method reporting limit
PARCCS	precision, accuracy, representativeness, completeness, comparability, and sensitivity
PCB	polychlorinated biphenyl
PE	performance evaluation
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
RPD	relative percent difference
SAP	Sampling and Analysis Plan
Settlement Agreement	Administrative Settlement Agreement and Order on Consent
SOP	standard operating procedure
SVOC	semivolatile organic compound
TOC	total organic carbon
USEPA	U.S. Environmental Protection Agency

1 PROJECT MANAGEMENT

1.1 DISTRIBUTION LIST

Title	Name
EPA Remedial Project Manager	Anna Krasko
EPA QA Reviewer	
Respondents' Project Coordinator and Integral Project Manager	Patrick Gwinn
Field Lead	Matt Behum
Laboratory QA Coordinator	Craig Hutchings
Database Manager	Tom Schulz
Chemical Testing Laboratory Project Manager (Vista Analytical Laboratory)	Martha M. Maier
Chemical Testing Laboratory QA Manager (Vista Analytical Laboratory)	Rose M. Harrelson
Chemical Testing Laboratory Project Manager (Columbia Analytical Services)	Jeff Christian
Chemical Testing Laboratory QA Manager (Columbia Analytical Services)	Julie Gish

1.2 INTRODUCTION AND TASK ORGANIZATION

This Sampling and Analysis Plan (SAP) has been prepared on behalf of Emhart Industries, Inc. (Emhart), pursuant to the requirements of Administrative Settlement Agreement and Order on Consent ("Settlement Agreement"), EPA Docket No. 01-2010-0045 (USEPA 2010a). Pursuant to the Settlement Agreement, Emhart will conduct a supplemental investigation of the Lyman Reach Stream Sediment and Floodplain Soil ("Oxbow") Area to assist the USEPA in: 1) evaluation of the remedial alternatives set forth in the Agency's Interim Final Feasibility Study Report (USEPA 2010b); 2) analysis of potential risks to human health and the environment; and 3) selection of the proposed remedial action for the Centredale Manor Restoration Project (CMRP) Superfund Site.

This document is the SAP for the Supplemental Investigation of the Lyman Mill Reach Sediment and Floodplain Soil (Oxbow) Area, and consists of the Quality Assurance Project Plan (QAPP) and the Field Sampling Plan (FSP), which is included as Appendix A. The QAPP was prepared consistent with USEPA guidance and requirements for QAPPs (USEPA 1998, 2001), as required by the Settlement Agreement.

This section reviews the organizational structure for activities associated with the Supplemental Investigation of the Lyman Reach and Floodplain soils, including project management and

oversight, field work, sample analysis, and data management. The organizational structure for this project is illustrated in Figure 1. Contact information for key personnel is provided in Section 1.3.

1.3 PROJECT ORGANIZATION

Sullivan & Worcester on behalf of Emhart have retained Integral Consulting Inc. (Integral) to perform the work described in this SAP. Figure 1 illustrates the organization of personnel on the project. The primary contacts for USEPA, Emhart, and Sullivan & Worcester are provided below. A description of the project organization and contacts pertaining to this QAPP are provided in the subsequent table.

1.3.1 Key Project Personnel

USEPA and Respondent Project Managers

Title	Name	Contact Information
USEPA Remedial Project Manager	Anna Krasko	U.S. Environmental Protection Agency, Region 1 Office of Site Remediation and Restoration 5 Post Office Square, Suite 100 Mail Code OSRR07-1 Boston, MA 02109-3912 krasko.anna@epa.gov
Emhart Industries, Inc. Vice President	Linda Biagioni	710 Joppa Road Towson, MD 21286 (410) 716-3208 Linda.biagioni@bdk.com
Sullivan & Worcester, LLP Partner	Jeffrey Karp, Esq.	1666 K Street, NW Washington, DC 20006 (202) 370-3921 jkarp@sandw.com

Integral will conduct the soil sampling investigation described in this QAPP. The names and quality assurance (QA) responsibilities of key Integral project personnel who will be involved in sampling and analysis activities are provided below.

Project Personnel Quality Assurance Responsibilities

Title	Responsibility	Name	Contact Information
Project Manager	Coordination of project information and related communications on behalf of Emhart Industries/Sullivan & Worcester with USEPA project manager; responsible for the successful completion of the Supplement Investigation	Patrick Gwinn	Integral Consulting Inc. 45 Exchange Street, Suite 200 Portland, Maine 04101 (207) 874-9000 ext. 206 pgwinn@integral-corp.com
Integral Corporate Health and Safety Manager	Oversight of health and safety program for field tasks associated with investigation	Eron Dodak	Integral Consulting Inc. 319 SW Washington Street Suite 1150 Portland, OR 97204 (503) 284-5545 ext. 14 edodak@integral-corp.com
Integral Field Lead	Field data collection oversight	Matt Behum	Integral Consulting Inc. 200 Harry S. Truman Parkway, Suite 330 Annapolis, MD 21401 (410) 573-1982 ext. 12 pjensen@integral-corp.com
Integral Field Health and Safety Officer	Implementation of the Health and Safety Plan in the field	Matt Behum	Integral Consulting Inc. 200 Harry S. Truman Parkway, Suite 330 Annapolis, MD 21401 (410) 573-1982 ext. 12 mbehum@integral-corp.com
Project Database Administrator Integral	Database development and data management	Tom Schulz	Integral Consulting Inc. 1205 West Bay Drive NW Olympia, WA 98502 (360) 705-3534 ext. 14 chutchings@integral-corp.com
Integral Project Laboratory QA Coordinator	Coordination with analytical laboratories	Craig Hutchings	Integral Consulting Inc. 1205 West Bay Drive NW Olympia, WA 98502 (360) 705-3534 ext. 17 chutchings@integral-corp.com
Vista Analytical Laboratory Project Manager	Dioxin/furan analysis	Martha M. Maier	1104 Windfield Way El Dorado Hills, CA 95762 (916) 673-1520 mmaier@vista-analytical.com
Vista Analytical Laboratory QA Manager	Laboratory quality assurance	Rose M. Harrelson	1104 Windfield Way El Dorado Hills, CA 95762 (916) 673-1520 rharrelson@vista-analytical.com

Title	Responsibility	Name	Contact Information
Columbia Analytical Services Laboratory Project Manager	Non-dioxin chemical analyses	Jeff Christian	1317 S. 13th Avenue Kelso, WA 98626 (360) 501-3316 jchristian@caslab.com
Columbia Analytical Services Laboratory QA Manager	Laboratory quality assurance	Julie Gish	1317 S. 13th Avenue Kelso, WA 98626 (360) 501-3317 jgish@caslab.com

1.3.2 Laboratories

The following responsibilities apply to the analytical laboratory project managers and QA managers to be used for this task.

The laboratory project manager at each laboratory is responsible for the successful and timely completion of sample analyses, and for performing the following tasks:

- Ensure that samples are received and logged in correctly, that the correct methods and modifications are used, and that data are reported within specified turnaround times
- Review analytical data to ensure that procedures were followed as required in this QAPP, the cited methods, and laboratory standard operating procedures (SOPs)
- Keep the task QA coordinator apprised of the schedule and status of sample analyses and data package preparation
- Notify the task QA coordinator if problems occur in sample receiving, analysis, or scheduling, or if control limits cannot be met
- Take appropriate corrective action as necessary
- Report data and supporting QA information as specified in this QAPP.

The laboratory QA manager at each laboratory is responsible for overseeing the QA activities in the laboratory and ensuring the quality of the data for this project. Specific responsibilities include the following:

- Oversee and implement the laboratory's QA program
- Maintain QA records for each laboratory production unit
- Ensure that QA and quality control (QC) procedures are implemented as required for each method and provide oversight of QA/QC practices and procedures
- Review and address or approve nonconformity and corrective action reports

- Coordinate response to any QC issues that affect this project with the Laboratory Project Manager.

1.4 SITE DEFINITION AND PROJECT OVERVIEW

1.4.1 Site Definition

The supplemental investigation focuses on the Lyman Mill Reach Sediment and Flood Plain Soils (including the Oxbow Area); an area designated by USEPA within the CMRP Site. The Lyman Mill Reach Sediment and Flood Plain Soils generally comprise the following areas within the CMRP Site:

1. The stream sediments in the reach of the Woonasquatucket River between the Allendale Dam and the northern extent of Lyman Mill Pond,
2. The flood plain soils in the Oxbow Area,
3. The emergent wetlands east of the Oxbow Area,
4. The emergent wetland at the confluence of Assapumpset Brook and the Woonasquatucket River (western shore of Lyman Mill Pond), and
5. The emergent wetland at the southeastern portion of Lyman Mill Pond.

As part of the Remedial Investigation of the CMPR Site, USEPA collected three sediment samples and four surface wetland soil samples in the Oxbow Area, which were analyzed for dioxins/furans, polychlorinated biphenyls (PCBs)/pesticides, semivolatile compounds, and metals. Dioxins/furans, PCBs, pesticides, semivolatile compounds and metals were all detected in the USEPA samples collected from the Oxbow Area (USACE 2006). Using these data, USEPA conducted baseline human health and ecological risk assessments for the Oxbow Area (USACE 2006). Based on the results of the baseline risk assessments, USEPA has proposed two remedial alternatives (other than the No Further Action alternative) for this area, which include either partial or targeted excavation with enhanced natural recovery. Current USEPA cost estimates for these two options range from $9.7 million to $42 million (USEPA 2010b).

The Oxbow Area is an approximately 40-acre complex of riverine, emergent, scrub-shrub, and forested wetland, and adjacent uplands. Most of the wetlands are within the 100-year floodplain of the Woonasquatucket River (USACE 2008). The analytical data from the seven samples collected by USEPA in the Oxbow Area are insufficient to adequately characterize the spatial or vertical distribution of contaminants detected in soil and/or sediment within the Oxbow Area. Additionally, there are insufficient data to accurately characterize the extent of contamination present in soils in the emergent wetlands noted in items 3 through 5 above.

The purpose of the supplemental investigation presented in this FSP is to assist the USEPA in: 1) evaluation of the remedial alternatives set forth in the Agency's Interim Final Feasibility Study Report (USEPA 2010b); 2) analysis of potential risks to human health and the environment; and 3) selection of the proposed remedial action for the Centredale Manor Restoration Project (CMRP) Superfund Site.

1.5 DATA QUALITY OBJECTIVES AND CRITERIA

1.5.1 Data Quality Objectives

The data quality objectives for this project are to ensure that field activities, including sample and field data collection, and laboratory analyses are conducted in a manner that is consistent with the practices generally considered acceptable for environmental site characterization. Meeting these objectives will ensure that the field and laboratory data resulting from the field sampling efforts are valid and are considered to be legally defensible. Thus, this QAPP specifies the measures and procedures to obtain data that are of sufficient quality and quantity to achieve the data quality objectives.

1.5.2 Measurement Performance Criteria

QA objectives are generally defined in terms of six parameters: precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS). Data quality objective development focuses on identifying the end use of the data to be collected and on determining the degree of certainty with respect to PARCCS necessary to satisfy the intended use of the data. The measurements and their performance criteria for this project are discussed in Section 2.

1.6 SPECIAL TRAINING AND CERTIFICATION

A technical team will be assembled with the requisite experience and technical skills to successfully complete the Lyman Mill Stream Sediment and Floodplain Soils supplemental investigation. All technical team personnel involved in sample collection will have extensive environmental sampling experience.

Sampling personnel who enter the exclusion zone and contaminant reduction zone (see Attachment A1, Sections 5.1.1 and 5.1.2 for definition and discussion of these zones) may be required to have completed the 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) standard training course and 8-hour refresher courses (see the Health and Safety Plan [HSP] for further explanation). The training provides employees with knowledge and skills that enable them to perform their jobs safely and with minimum risk to their personal health. Documentation of course completion will be maintained in personnel files.

Selected laboratories will hold certification through the National Environmental Laboratory Accreditation Program for the methods that the particular laboratory will perform, where applicable. Training and certification requirements for laboratory personnel will be provided in the laboratory QA plans (Appendix B).

1.7 DOCUMENTS AND RECORDS

Records will be maintained documenting all activities and data related to sample collection and to laboratory analyses. Results of data verification and validation activities will also be documented. Procedures for documentation of these activities are described in this section.

This QAPP, the FSP (Appendix A), and the HSP (Attachment A1 of Appendix A) will be provided to every task participant listed in Section 1.1. Any revisions or amendments to any of the documents that make up the FSP will also be provided to these individuals.

1.7.1 Field Records

Components of field documentation are discussed in Section 3 of the FSP. Centredale Manor technical team field leads will ensure that the field team receives the final, approved version of this QAPP, including the FSP and HSP, prior to the initiation of field activities. Field records that will be maintained include the following:

- Field logbooks
- Photo documentation
- Field data and sample collection information forms
- Field change request forms and corrective action forms (as needed)
- Sample tracking/chain-of-custody (COC) forms.

The following reports will be completed, as necessary, to document an audit or a deviation from the FSP or the QAPP:

- Corrective action forms will be used, as necessary, to document any problems encountered during field activities and corrective actions taken
- Field change request forms will be used, as necessary, to document the need for a procedural change or a station location change
- System and performance audit reports will be used, as necessary, to document review or audit of field sampling activities.

Observations recorded in the field logbooks will be used to provide context and aid in presentation and interpretation of analytical results. Field documentation related to sample

collection will be maintained in the project file. Additional details regarding the content and use of these documents are described in Section 3.2 of the FSP.

1.7.2 Laboratory Data Reports

All activities and results related to sample analysis will be documented at each laboratory. Internal laboratory documentation procedures are described in the laboratory QA manuals (Appendix B).

Each laboratory will provide a data package for each sample delivery group or analysis batch that is comparable in content to a full Contract Laboratory Program (CLP) package. The format of the data may differ from CLP requirements. Each data package will contain all information required for a complete QA review, including the following:

- A cover letter discussing analytical procedures and any difficulties that were encountered
- A case narrative referencing or describing the procedures used and discussing any analytical problems and deviations from SOPs and this QAPP
- COCs and cooler receipt forms
- A summary of analyte concentrations (to two significant figures, unless otherwise justified), method reporting limits (MRLs), and method detection limits (MDLs) or estimated detection limits (EDLs)
- Laboratory data qualifier codes appended to analyte concentrations, as appropriate, and a summary of code definitions
- Sample preparation, digestion, extraction, dilution, and cleanup logs
- Instrument tuning data
- Initial and continuing calibration data, including instrument printouts and quantification summaries, for all analytes
- Results for method and calibration blanks
- Results for all QA/QC checks, including but not limited to labeled compounds, surrogate spikes, internal standards, serial dilutions, laboratory control samples, matrix spike samples, matrix spike duplicate samples, and laboratory duplicate or triplicate samples provided on summary forms
- Instrument data quantification reports for all analyses and samples
- Copies of all laboratory worksheets and standards preparation logs.

Data will be delivered by the laboratories in both hard-copy and electronic format to the task QA coordinator, who will be responsible for oversight of data verification and validation and

for archiving the final data and data quality reports in the project file. Electronic data deliverables (EDDs) will be compatible with the project database.

Laboratory data will be maintained by each laboratory for a period of at least five years. These data will include the original instrument data files, reduced and verified data stored in the laboratory information management system (LIMS), and final hard copies and EDDs. The laboratories will obtain approval from the task QA coordinator prior to discarding these data.

1.7.3 Data Quality Documentation

Data verification (i.e., confirming the accuracy and completeness of field and laboratory data) will be completed by the Centredale Manor technical team for data generated in the field, and by each laboratory for the data that it generates. Data validation reports for chemical analyses will be prepared as described in Section 4 and provided to the task QA coordinator.

Ten percent of all electronic database entries provided by each laboratory will be verified against the validated hard-copy data in the data package. Any data tables prepared from the database for data users will include all qualifiers that were applied by the laboratories and during data validation, unless otherwise requested.

1.7.4 Reports and Deliverables

The laboratories will keep the laboratory QA coordinator apprised of their progress on a weekly basis. The laboratories will provide the following information:

- Inventory and status of samples held at the laboratory in spreadsheet format by sample delivery group
- Summaries of out-of-control laboratory QC data and any corrective actions implemented
- Descriptions and justification for any significant changes in methodology or QA/QC procedures.

Once the task is complete, a draft field sampling and data report will be prepared and submitted to USEPA. The draft field sampling and data report will discuss any deviations from this QAPP or the FSP and will contain sample location maps and validated analytical chemistry results. Consistent with the 2010 Settlement Agreement, the draft field sampling and data report will be submitted to USEPA within 45 days after the completion of all laboratory and data validation work.

2 DATA GENERATION AND ACQUISITION

2.1 SAMPLING DESIGN

A total of 42 locations have been identified within the site for characterization of the general area and human health exposure. Surface samples (0 to 12 in.; 0 to 30 cm) will be collected at all locations and vertical profile samples at two depth intervals (12 to 24 in. [30 to 61 cm] and 24 to 36 in. [61 to 91 cm]) will be collected at seven of these locations. All soil samples will be analyzed for grain size, pH, total organic carbon (TOC), percent solids, metals, organochlorine pesticides, Aroclors, semivolatile organic compounds (SVOCs), and dioxins and furans.

2.2 SAMPLING METHODS

Sampling methods that will be used to collect the suite of samples summarized in Section 2.1 are presented in the following sections. Sampling methods are described in detail in the FSP.

2.2.1 Surface Soil Samples for Chemical Analyses

Surface soil samples (0 to 12in.; 0 to 30 cm) for characterization of the general area and human health exposure may be collected with a variety of sampling equipment depending upon the conditions encountered in the field: stainless-steel spades or shovels, or a stainless-steel hand corer in accordance with standard methods used by USEPA (1997).

One surface soil sample will be collected at each sampling location. The soil collected at each station will be placed into a decontaminated stainless-steel bowl and homogenized using a stainless-steel spoon until the soil attains a visually uniform color and texture. Soil subsamples will then be removed for the requisite laboratory analyses.

2.2.2 Vertical Profile Soil Samples for Chemical Analyses

Vertical profile soil samples for characterization of general area or human health exposure will be collected using a portable electric impact corer). The subsurface samples will be collected from a depth of 12 to 24 in. (30 to 61 cm) and 24 to 36 in. (61 to 61 cm) and collocated with the surface soil samples discussed above in Section 2.2.1.

2.3 SAMPLE HANDLING AND CUSTODY

Principal documents used to identify samples and to document sample possession will be field logbooks and COC records. Custody will be documented for all samples at all stages of the

analytical or transfer process. COC procedures for sample handling prior to delivery to each laboratory are outlined in Section 3.3 of the FSP.

Upon receipt of samples at each laboratory, the physical integrity of the containers and seals will be checked, and the samples will be inventoried by comparing sample labels to those on the COC forms. Each laboratory will include the COC and shipping container receipt forms in the data package. Any breaks in the COC or nonconformances will be noted and reported in writing to the project laboratory coordinator within 24 hours of receipt of the samples. Each laboratory QA plan (Appendix B) includes procedures used for accepting custody of samples and documenting samples at the laboratory. The laboratory project manager will ensure that a sample-tracking record is maintained that follows each sample through all stages of sample processing at the laboratory.

Samples will be stored in accordance with Table 2-1. Samples for chemical analyses will be stored under refrigeration (4 ± 2°C). Any sample remaining in the sample jars after aliquots are removed for analysis by the laboratory will be archived by the laboratory performing the analysis. Each laboratory will maintain COC documentation and documentation of proper storage conditions for the entire time that the samples are in its possession.

The laboratories will not dispose of the samples for this task until authorized to do so by the task QA coordinator. After authorization is obtained, each laboratory will dispose of samples, as appropriate, based on matrix, analytical results, and information received from the client.

2.4 LABORATORY AND ANALYTICAL METHODS

Soil samples collected for this study will be analyzed for a variety of chemical and physical parameters as outlined in Table 2-2 and described below. These methods are consistent with requirements provided in SW-846 (USEPA 2008), ASTM (2009), and other established and widely accepted protocols. Analyte lists and expected MRLs and MDLs are provided in Tables 2-3 and 2-4.

Grain size distribution will be determined according to American Society for Testing and Materials (ASTM) Method D-422 (ASTM 2009), with modifications described in USEPA (1986). Organic material in the samples will not be oxidized prior to analysis. The silt and clay fractions will be determined by pipette.

The pH of the samples will be determined according to USEPA Method 9045C (USEPA 2008). Samples will be mixed with reagent water and the pH of the resulting slurry will be measured.

TOC will be analyzed by USEPA Method 9060 (USEPA 2008), modified for soil. Samples will be pretreated with hydrochloric acid to remove inorganic carbon, dried at 70°C, and analyzed by combustion in an induction furnace. Quadruplicate TOC analyses, as specified in the method,

will not be required for this project. Instead, laboratory duplicate analyses will be performed at a frequency of 5 percent, or one duplicate analysis per 20 samples.

Percent solids will be determined according to USEPA Method 160.3. Samples will be homogenized and dried at 103 to 105°C. These results will be used to calculate analyte concentrations on a dry-weight basis and will also be reported in the database.

Samples analyzed for metals will be digested with strong acid per USEPA Method 3050 (USEPA 2008) and analyzed by inductively coupled plasma-atomic emission spectrometry per USEPA Method 6010B (USEPA 2008).

Organochlorine pesticides will be extracted by soxhlet per USEPA Method 3540C (USEPA 2008) and be analyzed by gas chromatography with an electron capture detector (GC/ECD) in accordance with USEPA Method 8081A (USEPA 2008). Florisil® column cleanup (USEPA Method 3620), sulfur removal (USEPA Method 3660B) (USEPA 2008), and gel permeation chromatography cleanup (USEPA Method 3640A) (USEPA 2008) will be performed on the extract, if necessary, to achieve the MRL/MDLs specified in Table 2-3.

Aroclors will be extracted by soxhlet per USEPA Method 3450C (USEPA 2008) and be analyzed by GC/ECD in accordance with USEPA Method 8082A. Acid cleanup (USEPA Method 3665) (USEPA 2008) and sulfur removal (USEPA Method 3660B) (USEPA 2008) will be performed on the extract, if necessary, to achieve the MRL/MDL specified in Table 2-3.

SVOCs will be extracted by soxhlet and analyzed by gas chromatography/mass spectrometry in accordance with USEPA Method 8270C (USEPA 2008). Tentatively identified compounds will not be reported. Gel permeation chromatography cleanup (USEPA Method 3640A) (USEPA 2008) will be performed on the extract, if necessary, to achieve the MRL/MDLs specified in Table 2-3.

Dioxins and furans in soil samples will be extracted and analyzed in accordance with USEPA Method 1613B (USEPA 1994). All extracts will undergo acid/base silica gel cleanup and alumina cleanup. Extracts will also undergo Florisil cleanup if necessary to achieve the MRL/MDLs specified in Table 2-3. Samples will be analyzed by high-resolution gas chromatography with high-resolution mass spectrometry (HRGC/HRMS). Detection limits are calculated on an individual compound and sample basis and depend on the signal-to-background ratio for the specific labeled isomer.

2.5 QUALITY CONTROL

QC samples will be prepared in the field and at each laboratory to monitor the bias and precision of the sample collection and analysis procedures.

2.5.1 Field Quality Control

Field QC samples for this study will include field split samples (homogenization duplicates), equipment rinse blanks (field blanks), a rinsate blank, and a performance evaluation (PE) sample.

Field split samples will be collected at a minimum frequency of one for every 20 field samples processed. Field split samples are generated by the field team by splitting the soil sample into two aliquots for laboratory analysis. Data for field split samples are used to evaluate sample handling and analysis procedures and used in the evaluation of precision of the results.

Equipment rinse blanks (field blanks) are collected by pouring water over each type of nondedicated equipment in direct contact with the soils being collected Two equipment rinse blanks will be collected for this task. Equipment rinse blanks will be analyzed for metals and all organic parameters to monitor equipment decontamination procedures and to check for other sources of contamination.

A rinsate blank will be prepared in the field to evaluate potential background concentrations present in the water used for the equipment rinse blank. One rinsate blank will be collected at a minimum frequency of one for each batch of rinsate used for collecting the equipment rinse blanks.

PE samples are samples containing a known concentration of the analytes of interest in a contaminate-free media, such as sand. The USEPA will provide PE samples that will be submitted to the laboratories and used to evaluate the laboratory's ability to produce accurate results.

Procedures for preparing field split samples and equipment rinse blanks are presented in Section 2.3 of the FSP. Validation criteria and procedures for field QC samples are described in Sections 4.1 and 4.2 of this QAPP.

2.5.2 Laboratory Quality Control

Extensive and detailed requirements for laboratory QC procedures are provided in the methods that will be used for this investigation (Table 2-2). QC requirements include control limits and requirements for corrective action in many cases. QC procedures will be completed by each laboratory, as required by each protocol and as indicated in this QAPP.

The overall quality objective for this task is to develop and implement procedures that will ensure the collection of representative data of known and acceptable quality. The QA procedures and measurements that will be used for this project are based on USEPA guidance (USEPA 1990, 1994, 2008) and on established laboratory methods from other sources (ASTM 2009), as applicable.

The frequency of analysis for laboratory control samples, matrix spike samples, matrix spike duplicates or laboratory duplicates, and method blanks will be one for every 20 samples or one per extraction batch, whichever is more frequent. Surrogate spikes, labeled compounds, and internal standards will be added to every field sample and QC sample, as required. Calibration procedures will be completed at the frequency specified in each method description. Performance-based control limits have been established by each laboratory. These and all other control limits specified in the method descriptions will be used by the laboratories to establish the acceptability of the data or the need for reanalysis of the samples.

Where discrepancies exist between this QAPP and laboratory SOPs and QA manuals, this QAPP will take precedence.

PARCC parameters (i.e., precision, accuracy or bias, representativeness, completeness, comparability) are commonly used to assess the quality of environmental data. Bias represents the degree to which a measured concentration conforms to the reference value. The results for matrix spikes, laboratory control samples, field blanks, and method blanks will be reviewed to evaluate bias of the data. The following calculation is used to determine percent recovery for a matrix spike sample:

$$\%R = [(M-U) / C] \times 100$$

Where:

%R	=	percent recovery
M	=	measured concentration in the spiked sample
U	=	measured concentration in the unspiked sample
C	=	concentration of the added spike

The following calculation is used to determine percent recovery for a laboratory control sample or reference material:

$$\%R = (M / C) \times 100$$

Where:

%R	=	percent recovery
M	=	measured concentration in the spiked sample
U	=	measured concentration in the unspiked sample
C	=	concentration of the added spike

Results for field and method blanks can reflect systematic bias that results from contamination of samples during collection or analysis. Any analyte detected in field or method blanks will be evaluated as potential indicators of bias.

Precision reflects the reproducibility between individual measurements of the same property. Precision will be evaluated using the results of matrix spike duplicates, laboratory duplicates, and field splits. Precision is expressed in terms of the relative standard deviation for three or more measurements and the relative percent difference (RPD) for two measurements. The following equation is used to calculate the RPD between measurements:

$$RPD = |[(C1-C2) / ((C1 + C2) / 2)]| \times 100$$

Where:

RPD	=	relative percent difference
C1	=	first measurement
C2	=	second measurement

The relative standard deviation is the ratio of the standard deviation of three or more measurements to the average of the measurements, expressed as a percentage.

Completeness will be calculated as the ratio of usable data (i.e., unqualified data and U- or J-qualified data) to generated data, expressed as a percentage. Completeness will be calculated for each suite of analytes for each sample type and sampling event.

Additional laboratory QC results will be evaluated to provide supplementary information regarding overall quality of the data, performance of instruments and measurement systems, and sample-specific matrix effects.

QC samples and procedures are specified in each method protocol that will be used for this project. Methods are summarized in Table 2-2. All QC requirements will be completed by each laboratory as described in the protocols, including the following (as applicable to each analysis):

- Instrument tuning
- Initial calibration
- Initial calibration verification
- Continuing calibration verification
- Calibration or instrument blanks
- Method blanks
- Laboratory control samples
- Internal standards
- Surrogate spikes/labeled compounds

- Matrix spikes
- Matrix spike duplicates or laboratory duplicates.

To alert the data user to possible bias or imprecision, data qualifiers will be applied to reported analyte concentrations when associated QC samples or procedures do not meet control limits. Laboratory control limits for the methods that will be used for this Site investigation are provided in Tables 2-5 through 2-8. Control limits are periodically updated by the laboratory, and the control limits that are in effect at the laboratory at the time of analysis will be used for sample analysis and data validation. Data validation criteria and procedures are described in Section 4.

MRLs reflect the sensitivity of the analysis. Target MRLs for this study are summarized in Tables 2-3 and 2-4.

MDLs will be determined by each laboratory for each analyte, as required by USEPA (2008). MDLs are statistically derived and reflect the concentration at which an analyte can be detected in a clean matrix (e.g., sand or distilled water) with 99 percent confidence that a false positive result has not been reported. MRLs are established by the laboratories at levels above the MDLs for the project analytes. The MRL values are based on the laboratories' experience analyzing environmental samples and reflect the typical sensitivity obtained by the analytical system in environmental samples. For this task, the concentration of the lowest standard in the initial calibration curve for each analysis is at the level of the MRL. This allows reliable quantification of concentrations to the MRL in the absence of matrix interferences.

Dioxin and furan analyte concentrations for this task will be reported to the sample-specific EDLs as described in *USEPA Contract Laboratory Program Functional Guidelines for Chlorinated Dioxin/Furan Data Review* (USEPA 2005). Other analyte concentrations will be reported to the MDL. Analytes detected at concentrations between the MRL and the EDL or MDL will be reported with a J qualifier to indicate that the value is an estimate (i.e., the analyte concentration is below the calibration range). Nondetects will be reported at the EDL for dioxins and furan congeners, and to the MRL for all other analyses. The MRLs, EDLs, and MDLs will be adjusted by each laboratory, as necessary, to reflect sample dilution, percent moisture, and/or matrix interference.

2.5.3 Representativeness and Comparability of All Data

Representativeness and comparability are qualitative QA/QC parameters. Representativeness is the degree to which data represent a characteristic of an environmental condition. In the field, representativeness will be addressed primarily in the sampling design by the selection of sampling sites and sample collection procedures. In the laboratories, representativeness will be ensured by the proper handling and storage of samples and initiation of analysis within holding times.

Comparability is the qualitative similarity of one dataset to another (i.e., the extent to which different datasets can be combined for use). Comparability will be addressed through the use of field and laboratory methods that are consistent with methods and procedures recommended by USEPA and are commonly used for soil studies.

2.6 INSTRUMENT AND EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

Analytical instrument testing, inspection, maintenance, setup, and calibration will be conducted by each laboratory in accordance with the requirements identified in the laboratory's SOPs and manufacturer instructions. In addition, each of the specified analytical methods provides protocols for proper instrument setup and tuning, and critical operating parameters. Instrument maintenance and repair will be documented in the maintenance log or record book.

2.7 INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY

Laboratory instruments will be properly calibrated, and the calibration will be verified with appropriate check standards and calibration blanks for each parameter before beginning each analysis. Instrument calibration procedures and schedules will conform to analytical protocol requirements and descriptions provided in the laboratory's QA plans.

All calibration standards will be obtained from either the USEPA repository or a commercial vendor, and the laboratory will maintain traceability back to the National Institute of Standards and Technology. Stock standards will be used to make intermediate standards and calibration standards. Special attention will be given to expiration dating, proper labeling, proper refrigeration, and prevention of contamination. Documentation relating to the receipt, mixing, and use of standards will be recorded in a laboratory logbook. All calibration and spiking standards will be checked against standards from another source.

Field instruments will not be used in connection with this sampling.

2.8 INSPECTION AND ACCEPTANCE OF SUPPLIES AND CONSUMABLES

The quality of supplies and consumables used during sample collection and laboratory analysis can affect the quality of the project data. All equipment that comes into contact with the samples and extracts must be sufficiently clean to prevent detectable contamination, and the analyte concentrations must be accurate in all standards used for calibration and QC purposes.

The quality of laboratory water used for decontamination will be documented at the laboratory that provides that water. Precleaned sample jars (with documentation) will be provided by the

laboratories. All containers will be visually inspected prior to use, and any suspect containers will be discarded.

Reagents of appropriate purity and suitably cleaned laboratory equipment will also be used for all stages of laboratory analyses. Details for acceptance requirements for supplies and consumables at the laboratories are provided in the laboratory SOPs and QA manuals (to be submitted under separate cover). All supplies will be obtained from reputable suppliers with appropriate documentation or certification. Supplies will be inspected to confirm that they meet use requirements, and certification records will be retained by Integral (i.e., for supplies used in the field) or the laboratories.

2.9 NONDIRECT MEASUREMENTS

Nondirect measurements will not be used in connection with this task.

2.10 DATA MANAGEMENT

Data for this task will be generated in the field and at the laboratories. Procedures to be used to transfer data from the point of generation to the task database are described in this section.

2.10.1 Field Data

Daily field records (a combination of field logbooks, field forms, global positioning system [GPS] records, and COC forms) will make up the main documentation for field activities. Detailed guidelines for entry of information during field sampling are provided in the FSP, which is included as Appendix A to this QAPP. Upon completion of sampling, hard-copy notes and forms will be scanned to create an electronic record for use in creating the draft field sampling and data report. Information on sampling locations, dates, depths, equipment, and other conditions, and sample identifiers, will be entered into the task database. One hundred percent of hand-entered data will be verified based on hard-copy records. Electronic QA checks to identify anomalous values will also be conducted following entry.

COC forms will be generated in the field at the time that samples are packaged for delivery to analytical laboratories.

2.10.2 Laboratory Data

All data collected from each laboratory instrument, either manually or electronically, are reviewed and confirmed by analysts before reporting. The analytical laboratories will submit data in both electronic and hard-copy format. The project data manager will provide the desired format for EDDs to the laboratories and discuss these specifications with laboratory QA

managers prior to data delivery and tailor them as necessary to specific laboratory capabilities. QA checks of format and consistency will be applied to EDDs received from the laboratory. After any issues have been resolved, the data will be loaded into the task database. Data summaries will be produced from the database for use by data validators. Validators will return edited versions of these summaries, and the edits will then be incorporated into the database.

3 ASSESSMENT AND OVERSIGHT

This task will rely on the knowledge and expertise of the Centredale Manor technical team. The field team and laboratories will stay in close verbal contact with the task manager and task QA coordinator during all phases of this task. This level of communication will serve to keep the management team informed about activities and events, and will allow for informal but continuous task oversight.

3.1 ASSESSMENT AND RESPONSE ACTIONS

Assessment activities will include readiness reviews by the field coordinator prior to sampling, by the database administrator prior to release of the final data to the data users, and internal review while work is in progress. An informal technical systems audit may be conducted if problems are encountered during any phase of this project.

The first readiness review will be conducted by the field lead prior to field sampling to verify that all field equipment is ready for transfer to the Site. The field lead will also verify that the field team and any subcontractors have been scheduled and briefed and that the contracts for the subcontractors have been signed by both parties. Any deficiencies noted during this readiness review will be corrected prior to initiation of sampling activities.

The second readiness review will be completed by the database administrator before final data are released for use to verify that all results have been received from each laboratory, data validation and data quality assessment have been completed for all of the data, and data qualifiers have been entered into the database and verified. Any deficiencies noted during this review will be corrected by the database administrator, the task QA coordinator, or their designee. Data will not be released for final use until all data have been verified and validated. No report will be prepared in conjunction with the readiness reviews. However, the technical team coordinator and data users will be notified when the data are ready for use.

Technical review of intermediate and final work products generated for this task will be completed throughout the course of all sampling, laboratory, data validation, data management, and data interpretation activities to ensure that every phase of work is accurate and complete and follows the QA procedures outlined in this QAPP. Any problems that are encountered will be resolved between the reviewer and the person completing the work. Any problems that cannot be easily resolved or that affect the final quality of the work product will be brought to the attention of the Centredale Manor technical team coordinator and project coordinator.

Each laboratory will be required to have implemented a review system that serves as a formal surveillance mechanism for all laboratory activities. Details are provided in the laboratory QA plans (Appendix B).

Technical system audits may be conducted if serious problems are encountered during sampling or analysis operations. If completed, these audits will be conducted by the task QA coordinator or designee, or by the laboratory, as appropriate. These audits may consist of onsite reviews of any phase of field or laboratory activities or data management. Results of any audits will be provided in the draft field sampling and data report.

Any task team member who discovers or suspects a nonconformance is responsible for reporting the nonconformance to the task manager, the task QA coordinator, or the laboratory project or QA manager, as applicable. The task QA coordinator will ensure that no additional work dependent on the nonconforming activity is performed until a confirmed nonconformance is corrected.

3.2 REPORTS TO TASK MANAGER

The laboratories will keep the task laboratory coordinator informed of their progress on a weekly basis. The laboratories will provide the following information:

- Inventory and status of samples held at the laboratory in spreadsheet format by sample delivery group
- Summaries of any laboratory QC data outside of control limits and any corrective actions implemented
- Descriptions and justification for any significant changes in methodology or QA/QC procedures.

The task laboratory coordinator will provide this information to the task QA coordinator, who will provide this information to the task manager.

Each laboratory will be required to have implemented routine systems of reporting nonconformance issues and their resolution. These procedures are described in the laboratory QA manuals (Appendix B). Laboratory nonconformance issues will also be described in the draft field sampling and data report if they affect the quality of the data.

Data packages and EDDs will be prepared by each laboratory upon completion of analyses for each sample delivery group. The case narrative will include a description of any problems encountered, control limit exceedances (if applicable), and a description and rationale for any deviations from protocol. Copies of corrective action reports generated at the laboratory will also be included with the data package.

Data validation reports will be prepared following receipt of the complete laboratory data packages for each sample delivery group. These reports will be provided to the task QA coordinator when validation is completed for each parameter. A summary of any significant data quality issues will be provided to USEPA with the draft field sampling and data report.

4 DATA VALIDATION AND USABILITY

Data generated in the field and at the laboratories will be verified and validated according to criteria and procedures described in this section. Data quality and usability will be evaluated, and a discussion will be included in the draft field sampling and data report.

4.1 CRITERIA FOR DATA REVIEW, VERIFICATION, AND VALIDATION

Field and laboratory data for this task will undergo a formal verification and validation process. All entries into the database will be verified. All errors found during the verification of field data, laboratory data, and the database will be corrected prior to release of the final data.

Data verification and validation for all analytes will be completed in accordance with *Guidance on Environmental Data Verification and Validation* (USEPA 2002). Data verification and validation for metals and organic analytes other than dioxins will be completed according to methods described in *Region I EPA-New England Data Validation Functional Guidelines for Evaluating Environmental Analyses* (USEPA 1996). Data verification and validation for dioxins will be completed according to methods described in *USEPA Contract Laboratory Program Functional Guidelines for Chlorinated Dioxin/Furan Data Review* (USEPA 2005). No guidelines are available for validation of data for grain size, pH, TOC, or percent moisture. These data will be validated using procedures described in USEPA's Region 1 functional guidelines for inorganic data review (USEPA 1996), as applicable, and their respective methods.

Performance-based control limits established by the laboratories and control limits provided in the method protocols will be used to evaluate data quality and determine the need for data qualification. Performance-based control limits are established periodically by each laboratory. Current laboratory control limits for surrogate compounds, laboratory control samples and duplicates, and matrix spikes and duplicates are provided in Tables 2-5 through 2-8. Control limits are updated periodically by the laboratories, and the control limits that are in affect at the laboratory at the time of analysis will be used for data validation.

Results for field split samples will be evaluated against a control limit of 50 percent RPD for values greater than five times the MRL. For values less than five times the MRL the absolute difference should be less than two times the MRL. Data will not be qualified as estimated if these control limits are exceeded, but RPD results and absolute differences will be tabulated, and any exceedances will be discussed in the draft field sampling and data report. Equipment rinse blanks will be evaluated and data qualifiers will be applied in the same manner as method blanks, as described in USEPA's Region 1 functional guidelines for data review (USEPA 1996).

Data will be rejected if control limits for acceptance of data are not met, as described in USEPA (1996, 2005).

4.2 VERIFICATION AND VALIDATION METHODS

Field data will be verified during preparation of samples and COC forms. Field data and COC forms will be reviewed daily by the field lead. After field data are entered into the project database, 100 percent verification of the entries will be completed by a second party to ensure the accuracy and completeness of the database. Any discrepancies will be resolved before the final database is released for use.

Procedures for verification and validation of laboratory data and field quality control samples will be completed as described in the following EPA guidance documents for data validation:

- *Guidance on Environmental Data Verification and Validation* (USEPA 2002)
- *Region I EPA-New England Data Validation Functional Guidelines for Evaluating Environmental Analyses* (USEPA 1996)
- *USEPA Contract Laboratory Program Functional Guidelines for Chlorinated Dioxin/Furan Data Review* (USEPA 2005).

Control limits that will be used to qualify data are described in Section 4.1, above.

Data verification and validation will be completed as described in Section 4.1 by a data validation contractor. The first data package generated for each analysis method will be fully validated, equivalent to a Stage 4 validation as described in USEPA (2009). If no major problems are encountered during validation of this package, full validation will be completed at a rate of approximately 10 percent of the samples analyzed. Validation for the remaining data will be based on a review of the sample and QC data, equivalent to a Stage 2B validation. If problems are encountered, the laboratory will be contacted for resolution. Additional Stage 4 validation will be completed if required to fully assess the quality of the data to verify that the laboratory errors have been addressed.

The accuracy and completeness of the database will be verified at each laboratory when the EDDs are prepared and again as part of data validation. Ten percent of entries to the database from laboratory EDDs will be checked against hard-copy data packages. In addition to verification of field and laboratory data and information, data qualifier entries into the database will be verified. Any discrepancies will be resolved before the final database is released for use.

Target MRLs are provided in Tables 2-3 and 2-4. Reporting limits for nondetects will be compared to the MRL goals to evaluate method sensitivity for each sample. Any exceedance of actual MRLs over the target MRLs will be discussed in the draft field sampling and data report.

4.3 RECONCILIATION WITH USER REQUIREMENTS

The goal of data validation is to determine the quality of each data result and to identify those that do not meet the task measurement quality objectives. Nonconforming data may be qualified as estimated (i.e., a J qualifier will be applied to the result) or rejected as unusable (i.e., an R qualifier will be applied to the result) during data validation if criteria for data quality are not met. Rejected data will not be used for any purpose. An explanation of the rejected data will be included in the draft field sampling and data report.

Data qualified as estimated will be used for all intended purposes and will be appropriately qualified in the final project database. However, these data are less precise or less accurate than unqualified data. Data users, in cooperation with the Centredale Manor technical team coordinator and the task QA coordinator, are responsible for assessing the effect of the inaccuracy or imprecision of the qualified data on statistical procedures and other data uses. The data quality discussion in the draft field sampling and data report will include all available information regarding the direction or magnitude of bias or the degree of imprecision for qualified data to facilitate the assessment of data usability.

5 REFERENCES

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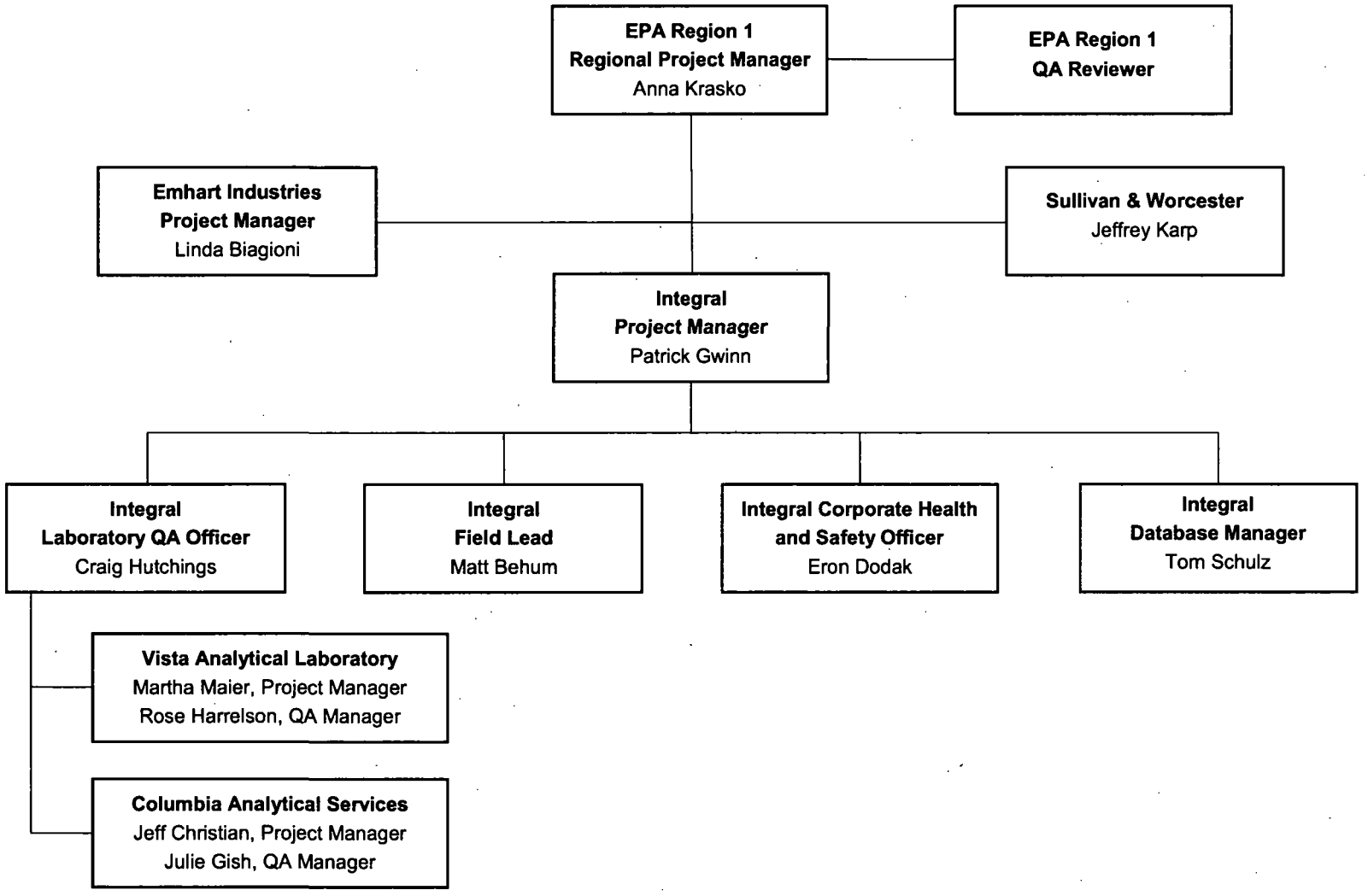
USEPA. 2010b. Interim Final Feasibility Study Centredale Manor Restoration Project Superfund Site, North Providence, Rhode Island.

FIGURES



integral consulting inc.

Figure 1
Project Organization Chart
Sampling and Analysis Plan
Centredale Manor Restoration Project Superfund Site

TABLES

Table 2-1. Sample Containers, Preservation, and Holding Time Requirements

Container[a]						
Type	Size	Laboratory	Analysis	Preservation	Holding Time	Sample Size
Sediment						
WMG	16 oz	CAS	Grain Size	4±2°C	6 months	100 g
WMG	8 oz	CAS	Metals	4±2°C	6 months	10 g
			Percent solids	4±2°C	6 months	10 g
			pH	4±2°C	14 days [b]	20 g
			TOC	4±2°C	28 days	1 g
WMG	16 oz	CAS	Pesticides	4±2°C	14 days / 40 days [c]	25 g
			PCBs	4±2°C	14 days / 40 days [c]	25 g
			SVOCs	4±2°C	14 days / 40 days [c]	25 g
WMG	8 oz	Vista	Dioxins	4±2°C	1 year	10 g
Equipment Rinse Blanks						
HDPE	500 mL	CAS	Metals	5 ml of 1:1 HNO_3; 4±2°C	6 months	100 mL
AG	1 L	CAS	Pesticides	4±2°C	7 days / 40 days [c]	1 L
AG	1 L	CAS	PCBs	4±2°C	7 days / 40 days [c]	1 L
AG	1 L	CAS	SVOCs	4±2°C	7 days / 40 days [c]	1 L
AG	1 L	Vista	Dioxins	4±2°C	1 year	1 L

Notes:

AG = amber glass
CAS = Columbia Analytical Services
HDPE = high density polyethylene
PCB = polylchlorinated biphenyl
QA = quality assurance
SVOC = semivolatile organic compound
TOC = total organic carbon
Vista = Vista Analytical Laboratory
WMG = wide mouth glass

[a] Collection of twice the indicated number of containers will be necessary for 5% of samples to allow for laboratory QA procedures.
[b] There is no established holding time for pH measurement by method 9045C; the holding time shown is based on best professional judgment.
[c] Holding time to extraction / holding time to analysis

Table 2-2. Laboratory Methods for Sediment and Equipment Blank Samples

		Sample Preparation		Quantitative Analysis	
Analytes	Laboratory	Protocol	Procedure	Protocol	Procedure
Sediments					
Conventional Parameters					
Grain size	CAS	NA	--	ASTM D-422M	Sieves and pipette method
pH	CAS	USEPA 9045C	Slurry	USEPA 9045C	Electrode
Total organic carbon	CAS	USEPA 9060A	Acid pretreatment	USEPA 9060A	Combustion; infrared detection
Percent solids	CAS	USEPA 160.3 modified	Oven drying	USEPA 160.3 modified	Balance
Metals					
Antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, lead, manganese, molybdenum, nickel, selenium, silver, thallium, vanadium, zinc	CAS	USEPA 3050	Strong acid digestion	USEPA 6010B	ICP-AES
Organics					
Organochlorine pesticides	CAS	USEPA 3540C	Soxhlet extraction	USEPA 8081A	GC/ECD
		USEPA 3640A	Gel permeation chromatography [a]		
		USEPA 3620C	Florisil® chromatography [a]		
		USEPA 3660C	Sulfur cleanup [a]		
Polychlorinated biphenyls (Aroclors)	CAS	USEPA 3540C	Soxhlet extraction	USEPA 8082A	GC/ECD
		USEPA 3665A	Acid cleanup		
		USEPA 3660C	Sulfur cleanup [a]		
Semivolatile organic compounds	CAS	USEPA 3540 / 3541	Soxhlet / Automated Soxhlet extraction	USEPA 8270C	GC/MS
		USEPA 3640A	Gel permeation chromatography [a]		
Polychlorinated dioxins and furans	Vista	USEPA 1613B	Soxhlet extraction	USEPA 1613B	HRGC/HRMS
			Acid Base Silica Gel column		
			Alumina cleanup		
			Florisil® chromatography [a]		

Table 2-2. Laboratory Methods for Sediment and Equipment Blank Samples

Analytes	Laboratory	Sample Preparation		Quantitative Analysis	
		Protocol	Procedure	Protocol	Procedure
Equipment Rinse Blanks					
Metals					
Antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, lead, manganese, molybdenum, nickel, selenium, silver, thallium, vanadium, zinc	CAS	USEPA 3050	Strong acid digestion	USEPA 6010B	ICP-AES
Organics					
Pesticides	CAS	USEPA SW846-3510C	Separatory funnel	USEPA 8081A	Dual column GC/ECD
Polychlorinated biphenyls (Aroclors)	CAS	USEPA SW846-3510C	Separatory funnel	USEPA 8082A	Dual column GC/ECD
Semivolatile organic compounds	CAS	USEPA SW846-3510C	Separatory funnel	USEPA 8270C	GC/MS
Polychlorinated dioxins and furans	Vista	USEPA 1613B	Soxhlet extraction	USEPA 1613B	HRGC/HRMS

Notes:

-- = not available
AES = atomic emission spectroscopy
ASTM = American Society for Testing and Materials
CAS = Columbia Analytical Services
ECD = electron capture detector
GC = gas chromatography
HRGC = high-resolution gas chromatography
HRMS = high-resolution mass spectrometry
ICP = inductively coupled plasma/mass spectrometry
MRL = method reporting limit
MS = mass spectrometry
NA = not applicable
USEPA = U.S. Environmental Protection Agency
Vista = Vista Analytical Laboratory

[a] Cleanup procedures will be performed as necessary to achieve the method reporting limits and method detection limits listed in Table MRLs.

Table 2-3. Analytes, Method Reporting Limits, and Method Detection Limits for Sediment Samples

Analyte	CAS Number	MDL [a]	MRL [a,b,c]
Columbia Analystical Services			
Conventionals			
Grain size (percent)	--	NA	0.1
pH (pH units)	--	NA	0.1
Total organic carbon (percent)	--	0.04	0.05
Percent solids (percent of whole weight)	--	NA	0.1
Metals (mg/kg)			
Antimony	7440-36-0	0.5	2
Arsenic	7440-38-2	0.9	2
Barium	7440-39-3	0.06	0.5
Beryllium	7440-41-7	0.03	0.1
Cadmium	7440-43-9	0.03	0.1
Chromium	7440-47-3	0.2	0.5
Cobalt	7440-48-4	0.2	0.5
Copper	7440-50-8	0.3	0.6
Lead	7439-92-1	0.4	2
Manganese	7439-96-5	0.02	0.2
Molybdenum	7439-98-7	0.08	0.4
Nickel	7440-02-0	0.07	0.4
Selenium	7782-49-2	0.7	4
Silver	7440-22-4	0.2	0.5
Thallium	7440-28-0	0.4	2
Vanadium	7440-62-2	0.3	1
Zinc	7440-66-6	0.3	1
Organochlorine Pesticides (µg/kg-dry weight)			
4,4'-DDD	72-54-8	0.11	1
4,4'-DDE	72-55-9	0.11	1
4,4'-DDT	50-29-3	0.17	1
Aldrin	309-00-2	0.16	1
alpha-BHC	319-84-6	0.11	1
beta-BHC	319-85-7	0.18	1
delta-BHC	319-86-8	0.074	1
gamma-BHC (Lindane)	58-89-9	0.08	1
Dieldrin	60-57-1	0.14	1
Endosulfan I	959-98-8	0.063	1
Endosulfan II	33213-65-9	0.14	1
Endosulfan sulfate	1031-07-8	0.11	1
Endrin	72-20-8	0.094	1
Endrin aldehyde	7421-93-4	0.12	1
Endrin ketone	53494-70-5	0.093	1
alpha-Chlordane (cis-)	5103-71-9	0.1	1
gamma-Chlordane (trans-)	5103-74-2	0.09	1
Technical Chlordane	12789-03-6	1.9	10
Heptachlor	76-44-8	0.12	1
Heptachlor epoxide	1024-57-3	0.084	1
Methoxychlor	72-43-5	0.19	1
Toxaphene	8001-35-2	4.8	50
PCB Aroclors (µg/kg-dry weight)			
Aroclor 1016	12674-11-2	2.1	10
Aroclor 1221	11104-28-2	2.1	20
Aroclor 1232	11141-16-5	2.1	10
Aroclor 1242	53469-21-9	2.1	10
Aroclor 1248	12672-29-6	2.1	10
Aroclor 1254	11097-69-1	2.1	10
Aroclor 1260	11096-82-5	2.1	10

Table 2-3. Analytes, Method Reporting Limits, and Method Detection Limits for Sediment Samples

Analyte	CAS Number	MDL [a]	MRL [a,b,c]
Aroclor 1262	37324-23-5	2.1	10
Aroclor 1268	11100-14-4	2.1	10
Semivolatile Organic Compounds (µg/kg-dry weight)			
1,1'-Biphenyl	92-52-4	0.009	0.33
1,2,4-Trichlorobenzene	120-82-1	0.011	0.33
1,2-Dichlorobenzene	95-50-1	0.0179	0.33
1,3-Dichlorobenzene	541-73-1	0.0183	0.33
1,4-Dichlorobenzene	106-46-7	0.0175	0.33
2,2'-oxybis(1-Chloropropane)	108-60-1	0.0141	0.33
2,4,5-Trichlorophenol	95-95-4	0.0171	0.33
2,4,6-Trichlorophenol	88-06-2	0.0143	0.33
2,4-Dichlorophenol	120-83-2	0.0164	0.33
2,4-Dimethylphenol	105-67-9	0.0151	0.33
2,4-Dinitrophenol	51-28-5	0.112	2
2,4-Dinitrotoluene	121-14-2	0.0149	0.33
2,6-Dinitrotoluene	606-20-2	0.0156	0.33
2-Chloronaphthalene	91-58-7	0.01	0.33
2-Chlorophenol	95-57-8	0.0099	0.33
2-Methylnaphthalene	91-57-6	0.011	0.33
2-Methylphenol (o-cresol)	95-48-7	0.0167	0.33
2-Nitroaniline	88-74-4	0.0169	0.33
2-Nitrophenol	88-75-5	0.0139	0.33
3 & 4-Methylphenol	--	0.0168	0.33
3,3'-Dichlorobenzidine	91-94-1	0.027	0.33
3-Nitroaniline	99-09-2	0.0175	0.33
4,6-Dinitro-2-methylphenol	534-52-1	0.143	2
4-Bromophenyl-phenylether	101-55-3	0.0122	0.33
4-Chloro-3-methylphenol	59-50-7	0.0166	0.33
4-Chloroaniline	106-47-8	0.0144	0.33
4-Chlorophenyl-phenyl ether	7005-72-3	0.016	0.33
4-Nitroaniline	100-01-6	0.179	2
4-Nitrophenol	100-02-7	0.146	2
Acenaphthene	83-32-9	0.0134	0.33
Acenaphthylene	208-96-8	0.016	0.33
Anthracene	120-12-7	0.0139	0.33
Benzaldehyde	100-52-7	0.021	0.33
Benzo(a)anthracene	56-55-3	0.0123	0.33
Benzo(a)pyrene	50-32-8	0.0198	0.33
Benzo(b)fluoranthene	205-99-2	0.0172	0.33
Benzo(ghi)perylene	191-24-2	0.0202	0.33
Benzo(k)fluoranthene	207-08-9	0.0194	0.33
Benzoic acid	65-85-0	0.139	2
Benzyl alcohol	100-51-6	0.0168	0.33
Bis(2-Chloroethoxy)methane	111-91-1	0.011	0.33
Bis(2-chloroethyl)ether	111-44-4	0.0117	0.33
Bis(2-Ethylhexyl)phthalate	117-81-7	0.0186	0.33
Butyl benzyl phthalate	85-68-7	0.0163	0.33
Carbazole	86-74-8	0.0112	0.33
Chrysene	218-01-9	0.0118	0.33
Dibenzo(a,h)anthracene	53-70-3	0.0275	0.33
Dibenzofuran	132-64-9	0.0118	0.33
Diethyl phthalate	84-66-2	0.0141	0.33
Dimethyl phthalate	131-11-3	0.0164	0.33
Di-n-butyl phthalate	84-74-2	0.0121	0.33
Di-n-octylphthalate	117-84-0	0.024	0.33
Fluoranthene	206-44-0	0.0115	0.33

Table 2-3. Analytes, Method Reporting Limits, and Method Detection Limits for Sediment Samples

Analyte	CAS Number	MDL [a]	MRL [a,b,c]
Fluorene	86-73-7	0.013	0.33
Hexachlorobenzene	118-74-1	0.0147	0.33
Hexachlorobutadiene	87-68-3	0.0141	0.33
Hexachlorocyclopentadiene	77-47-4	0.0125	0.33
Hexachloroethane	67-72-1	0.0216	0.33
Indeno[1,2,3-cd]pyrene	193-39-5	0.0389	0.33
Isophorone	78-59-1	0.014	0.33
Naphthalene	91-20-3	0.0144	0.33
Nitrobenzene	98-95-3	0.0261	0.33
N-Nitrosodi-n-propylamine	621-64-7	0.0191	0.33
N-Nitrosodiphenylamine	86-30-6	0.018	0.33
Pentachlorophenol	87-86-5	0.125	2
Phenanthrene	85-01-8	0.01	0.33
Phenol	108-95-2	0.0195	0.33
Pyrene	129-00-0	0.014	0.33
Vista Analytical Laboratory			
Polychlorinated Dioxins and Furans (ng/kg-dry weight)			
2,3,7,8-TCDD	1746-01-6	0.23	0.5
1,2,3,7,8-PeCDD	40321-76-4	0.06	2.5
1,2,3,4,7,8-HxCDD	39227-28-6	0.08	2.5
1,2,3,6,7,8-HxCDD	57653-85-7	0.07	2.5
1,2,3,7,8,9-HxCDD	19408-74-3	0.05	2.5
1,2,3,4,6,7,8-HpCDD	35822-46-9	0.10	2.5
ODDD	3268-87-9	0.16	5
2,3,7,8-TCDF	51207-31-9	0.05	0.5
1,2,3,7,8-PeCDF	57117-41-6	0.08	2.5
2,3,4,7,8-PeCDF	57117-31-4	0.08	2.5
1,2,3,4,7,8-HxCDF	70648-26-9	0.31	2.5
1,2,3,6,7,8-HxCDF	57117-44-9	0.05	2.5
2,3,4,6,7,8-HxCDF	60851-34-5	0.07	2.5
1,2,3,7,8,9-HxCDF	72918-21-9	0.03	2.5
1,2,3,4,6,7,8-HPCDF	67562-39-4	0.65	2.5
1,2,3,4,7,8,9-HPCDF	55673-89-7	0.09	2.5
OCDF	39001-02-0	0.87	5

Notes:

-- = not available
MDL = method detection limit
MRL = method reporting limit
NA = not applicable
PCB = polychlorinated biphenyl

[a] Method detection limits are updated periodically by the laboratories. MDLs that are in effect at the laboratory at the time of analysis will be used. These may differ slightly from the MDL shown in this table.

[b] The MRL is provided on a dry-weight basis and assumes 50% moisture in the samples. The MRL for project samples will vary with moisture content in the samples.

[c] The MRL represents the level of lowest calibration standard (i.e., the practical quantitation limit).

Table 2-4. Analytes, Method Reporting Limits, and Method Detection Limits for Equipment Rinse Blanks

Analyte	CAS Number	MDL [a]	MRL [a,b,c]
Columbia Analystical Services			
Metals (µg/L)			
Antimony	7440-36-0	3	10
Arsenic	7440-38-2	4	10
Barium	7440-39-3	0.4	2
Beryllium	7440-41-7	0.09	0.2
Cadmium	7440-43-9	0.3	0.5
Chromium	7440-47-3	0.4	2
Cobalt	7440-48-4	0.4	1
Copper	7440-50-8	0.8	2
Lead	7439-92-1	4	10
Manganese	7439-96-5	0.2	0.6
Molybdenum	7439-98-7	0.6	2
Nickel	7440-02-0	0.7	2
Selenium	7782-49-2	5	20
Silver	7440-22-4	0.7	2
Thallium	7440-28-0	2	10
Vanadium	7440-62-2	1	2
Zinc	7440-66-6	0.7	2
Organochlorine Pesticides (µg/L)			
4,4'-DDD	72-54-8	0.00150	0.01
4,4'-DDE	72-55-9	0.00036	0.01
4,4'-DDT	50-29-3	0.00058	0.01
Aldrin	309-00-2	0.00040	0.01
alpha-BHC	319-84-6	0.00033	0.01
beta-BHC	319-85-7	0.00083	0.01
delta-BHC	319-86-8	0.00057	0.01
gamma-BHC (Lindane)	58-89-9	0.00044	0.01
Dieldrin	60-57-1	0.00035	0.01
Endosulfan I	959-98-8	0.00044	0.01
Endosulfan II	33213-65-9	0.00040	0.01
Endosulfan sulfate	1031-07-8	0.00047	0.01
Endrin	72-20-8	0.00068	0.01
Endrin aldehyde	7421-93-4	0.00046	0.01
Endrin ketone	53494-70-5	0.00066	0.01
alpha-Chlordane (cis-)	5103-71-9	0.0040	0.01
gamma-Chlordane (trans-)	5103-74-2	0.00032	0.01
Technical Chlordane	12789-03-6	0.015	0.2
Heptachlor	76-44-8	0.00036	0.01
Heptachlor epoxide	1024-57-3	0.00032	0.01
Methoxychlor	72-43-5	0.00093	0.01
Toxaphene	8001-35-2	0.028	0.5
PCB Aroclors (µg/L)			
Aroclor 1016	12674-11-2	0.0094	0.2
Aroclor 1221	11104-28-2	0.0094	0.4
Aroclor 1232	11141-16-5	0.0094	0.2
Aroclor 1242	53469-21-9	0.0094	0.2
Aroclor 1248	12672-29-6	0.0094	0.2
Aroclor 1254	11097-69-1	0.0094	0.2
Aroclor 1260	11096-82-5	0.0094	0.2
Aroclor 1262	37324-23-5	0.0094	0.2
Aroclor 1268	11100-14-4	0.0094	0.2

Table 2-4. Analytes, Method Reporting Limits, and Method Detection Limits for Equipment Rinse Blanks

Analyte	CAS Number	MDL [a]	MRL [a,b,c]
Semivolatile Organic Compounds (µg/L)			
1,1'-Biphenyl	92-52-4	0.66	10
1,2,4-Trichlorobenzene	120-82-1	0.355	10
1,2-Dichlorobenzene	95-50-1	0.431	10
1,3-Dichlorobenzene	541-73-1	0.352	10
1,4-Dichlorobenzene	106-46-7	0.317	10
2,2'-oxybis(1-Chloropropane)	108-60-1	0.311	10
2,4,5-Trichlorophenol	95-95-4	0.381	10
2,4,6-Trichlorophenol	88-06-2	0.203	10
2,4-Dichlorophenol	120-83-2	0.297	10
2,4-Dimethylphenol	105-67-9	0.264	10
2,4-Dinitrophenol	51-28-5	2.22	25
2,4-Dinitrotoluene	121-14-2	0.274	10
2,6-Dinitrotoluene	606-20-2	0.349	10
2-Chloronaphthalene	91-58-7	0.290	10
2-Chlorophenol	95-57-8	0.311	10
2-Methylnaphthalene	91-57-6	0.239	10
2-Methylphenol (o-cresol)	95-48-7	0.328	10
2-Nitroaniline	88-74-4	0.336	25
2-Nitrophenol	88-75-5	0.373	10
3 & 4-Methylphenol	--	0.478	10
3,3'-Dichlorobenzidine	91-94-1	0.270	25
3-Nitroaniline	99-09-2	3.25	25
4,6-Dinitro-2-methylphenol	534-52-1	2.12	25
4-Bromophenyl-phenylether	101-55-3	0.274	10
4-Chloro-3-methylphenol	59-50-7	0.490	10
4-Chloroaniline	106-47-8	0.375	10
4-Chlorophenyl-phenyl ether	7005-72-3	0.278	10
4-Nitroaniline	100-01-6	4.03	25
4-Nitrophenol	100-02-7	1.92	25
Acenaphthene	83-32-9	0.281	10
Acenaphthylene	208-96-8	0.236	10
Anthracene	120-12-7	0.612	10
Benzaldehyde	100-52-7	0.50	10
Benzo(a)anthracene	56-55-3	0.591	10
Benzo(a)pyrene	50-32-8	0.651	10
Benzo(b)fluoranthene	205-99-2	0.584	10
Benzo(ghi)perylene	191-24-2	0.812	10
Benzo(k)fluoranthene	207-08-9	0.827	10
Benzoic acid	65-85-0	5.819	25
Benzyl alcohol	100-51-6	0.377	10
Bis(2-Chloroethoxy)methane	111-91-1	0.276	10
Bis(2-chloroethyl)ether	111-44-4	0.333	10
Bis(2-Ethylhexyl)phthalate	117-81-7	1.89	10
Butyl benzyl phthalate	85-68-7	0.470	10
Carbazole	86-74-8	0.237	10
Chrysene	218-01-9	0.787	10
Dibenzo(a,h)anthracene	53-70-3	0.752	10
Dibenzofuran	132-64-9	0.325	10
Diethyl phthalate	84-66-2	0.289	10
Dimethyl phthalate	131-11-3	0.254	10
Di-n-butyl phthalate	84-74-2	0.364	10
Di-n-octylphthalate	117-84-0	0.626	10
Fluoranthene	206-44-0	0.652	10
Fluorene	86-73-7	0.323	10
Hexachlorobenzene	118-74-1	0.628	10

Table 2-4. Analytes, Method Reporting Limits, and Method Detection Limits for Equipment Rinse Blanks

Analyte	CAS Number	MDL [a]	MRL [a,b,c]
Hexachlorobutadiene	87-68-3	0.291	10
Hexachlorocyclopentadiene	77-47-4	1.21	10
Hexachloroethane	67-72-1	0.289	10
Indeno[1,2,3-cd]pyrene	193-39-5	0.684	10
Isophorone	78-59-1	0.246	10
Naphthalene	91-20-3	0.365	10
Nitrobenzene	98-95-3	0.567	10
N-Nitrosodi-n-propylamine	621-64-7	0.496	10
N-Nitrosodiphenylamine	86-30-6	0.48	10
Pentachlorophenol	87-86-5	2.44	25
Phenanthrene	85-01-8	0.482	10
Phenol	108-95-2	0.324	10
Pyrene	129-00-0	0.731	10
Vista			
Polychlorinated Dioxins and Furans (pg/L)			
2,3,7,8-TCDD	1746-01-6	0.35	5
1,2,3,7,8-PeCDD	40321-76-4	0.99	25
1,2,3,4,7,8-HxCDD	39227-28-6	1.14	25
1,2,3,6,7,8-HxCDD	57653-85-7	0.95	25
1,2,3,7,8,9-HxCDD	19408-74-3	1.10	25
1,2,3,4,6,7,8-HpCDD	35822-46-9	1.24	25
ODDD	3268-87-9	3.20	50
2,3,7,8-TCDF	51207-31-9	0.40	5
1,2,3,7,8-PeCDF	57117-41-6	0.67	25
2,3,4,7,8-PeCDF	57117-31-4	0.66	25
1,2,3,4,7,8-HxCDF	70648-26-9	0.62	25
1,2,3,6,7,8-HxCDF	57117-44-9	0.60	25
2,3,4,6,7,8-HxCDF	60851-34-5	0.64	25
1,2,3,7,8,9-HxCDF	72918-21-9	1.47	25
1,2,3,4,6,7,8-HPCDF	67562-39-4	1.34	25
1,2,3,4,7,8,9-HPCDF	55673-89-7	1.54	25
OCDF	39001-02-0	2.48	50

Notes:

MDL = method detection limit
MRL = method reporting limit
PCB = polychlorinated biphenyl

[a] Method detection limits are updated periodically by the laboratories. MDLs that are in effect at the laboratory at the time of analysis will be used. These may differ slightly from the MDL shown in this table.

[b] The MRL is provided on a dry-weight basis and assumes 50% moisture in the samples. The MRL for project samples will vary with moisture content in the samples.

[c] The MRL represents the level of lowest calibration standard (i.e., the practical quantitation limit).

Table 2-5. Laboratory Control Limits for Surrogate Recoveries in Sediment Samples

Analyte	Control Limits for Percent Recovery
Columbia Analytical Services	
Organochlorine Pesticides	
Tetrachloro-m-xylene	21–112
Decachlorobiphenyl	15–130
PCB Aroclors	
Tetrachloro-m-xylene	10–135
Decachlorobiphenyl	35–133
Semivolatile Organic Compounds	
Nitrobenzene-d5	29–100
Phenol-d6	23–90
Terphenyl-d14	37–133
2-Fluorophenol	20–83
2-Fluorobiphenyl	32–104
2,4,6-Tribromophenol	20–123
Vista	
Polychlorinated Dioxins and Furans	
13C-2,3,7,8-TCDD	25–164
13C-1,2,3,7,8-PeCDD	25–181
13C-1,2,3,4,7,8-HxCDD	32–141
13C-1,2,3,6,7,8-HxCDD	28–130
13C-1,2,3,4,6,7,8-HpCDD	23–140
13C-OCDD	17–157
13C-2,3,7,8-TCDF	24–169
13C-1,2,3,7,8-PeCDF	24–185
13C-2,3,4,7,8-PeCDF	21–178
13C-1,2,3,4,7,8-HxCDF	26–152
13C-1,2,3,6,7,8-HxCDF	26–123
13C-2,3,4,6,7,8-HxCDF	28–136
13C-1,2,3,7,8,9-HxCDF	29–147
13C-1,2,3,4,6,7,8-HpCDF	28–143
13C-1,2,3,4,7,8,9-HpCDF	26–138
13C-OCDF	17–157
37Cl4-2,3,4,7,8-TCDD	35–197

Notes:

PCB = polychlorinated biphenyl

Control limits are updated periodically by the laboratories. Control limits that are in effect at the laboratory at the time of analysis will be used for sample analysis and data validation. These may differ slightly from the control limits shown in this table.

Table 2-6. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples for Sediment Samples

	Accuracy		Precision	
Analyte	Matrix Spike Recovery (Percent)	LCS Recovery (Percent)	Type of Duplicate	Control Limit RPD
Columbia Analytical Services				
Conventionals				
Grain size	NA	NA	Triplicate	10 [a]
pH	NA	NA	Duplicate	20
Total organic carbon	77–155	82–119	MSD	20
Percent solids	NA	NA	Duplicate	20
Metals				
Antimony	10–118	45–157	Duplicate	30
Arsenic	54–136	89–124	Duplicate	30
Barium	60–139	89–124	Duplicate	30
Beryllium	78–115	89–123	Duplicate	30
Cadmium	78–118	92–126	Duplicate	30
Chromium	32–170	90–130	Duplicate	30
Cobalt	67–129	91–125	Duplicate	30
Copper	24–173	87–124	Duplicate	30
Lead	46–151	69–145	Duplicate	30
Manganese	28–181	88–129	Duplicate	30
Molybdenum	81–112	90–132	Duplicate	30
Nickel	77–121	91–129	Duplicate	30
Selenium	68–147	72–120	Duplicate	30
Silver	52–148	88–123	Duplicate	30
Thallium	32–161	64–189	Duplicate	30
Vanadium	64–132	88–129	Duplicate	30
Zinc	13–172	83–132	Duplicate	30
Organochlorine Pesticides				
4,4'-DDD	19–143	46–146	MSD	40
4,4'-DDE	22–142	46–141	MSD	40
4,4'-DDT	19–154	46–151	MSD	40
Aldrin	22–135	37–134	MSD	40
alpha-BHC	23–133	36–139	MSD	40
beta-BHC	22–142	38–142	MSD	40
delta-BHC	25–148	48–145	MSD	40
gamma-BHC (Lindane)	26–135	40–142	MSD	40
Dieldrin	26–133	46–136	MSD	40
Endosulfan I	15–119	35–121	MSD	40
Endosulfan II	13–129	39–128	MSD	40
Endosulfan sulfate	20–134	43–138	MSD	40
Endrin	22–145	40–152	MSD	40
Endrin aldehyde	10–129	32–132	MSD	40
Endrin ketone	19–139	47–135	MSD	40
alpha-Chlordane (cis-)	24–132	41–134	MSD	40
gamma-Chlordane (trans-)	24–133	41–135	MSD	40

Table 2-6. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples for Sediment Samples

	Accuracy		Precision	
Analyte	Matrix Spike Recovery (Percent)	LCS Recovery (Percent)	Type of Duplicate	Control Limit RPD
Heptachlor	21–136	39–135	MSD	40
Heptachlor epoxide	25–129	45–118	MSD	40
Methoxychlor	24–151	42–147	MSD	40
PCB Aroclors				
PCB-1016	27–174	48–121	MSD	40
PCB-1260	20–185	53–129	MSD	40
Semivolatile Organic Compounds				
1,1'-Biphenyl	26–110	43–116	MSD	40
1,2,4-Trichlorobenzene	28–105	42–91	MSD	40
1,2-Dichlorobenzene	29–93	41–86	MSD	40
1,3-Dichlorobenzene	28–91	41–84	MSD	40
1,4-Dichlorobenzene	28–95	41–85	MSD	40
2,2'-oxybis(1-Chloropropane)	25–106	39–94	MSD	40
2,4,5-Trichlorophenol	27–115	46–103	MSD	40
2,4,6-Trichlorophenol	24–119	46–101	MSD	40
2,4-Dichlorophenol	16–119	43–94	MSD	40
2,4-Dimethylphenol	10–117	16 –89	MSD	40
2,4-Dinitrophenol	10–145	28–116	MSD	40
2,4-Dinitrotoluene	36–127	48–119	MSD	40
2,6-Dinitrotoluene	36–125	49–114	MSD	40
2-Chloronaphthalene	26–114	44–95	MSD	40
2-Chlorophenol	27–96	42–86	MSD	40
2-Methylnaphthalene	30–105	44–95	MSD	40
2-Methylphenol (o-cresol)	14–103	35–92	MSD	40
2-Nitroaniline	34–121	48–108	MSD	40
2-Nitrophenol	20–119	44–95	MSD	40
3 & 4-Methylphenol	10–111	38–94	MSD	40
3,3'-Dichlorobenzidine	10–120	38–116	MSD	40
3-Nitroaniline	15–123	46–111	MSD	40
4,6-Dinitro-2-methylphenol	10–138	41–119	MSD	40
4-Bromophenyl-phenylether	32–127	49–110	MSD	40
4-Chloro-3-methylphenol	13–122	44–101	MSD	40
4-Chloroaniline	11–98	37–96	MSD	40
4-Chlorophenyl-phenyl ether	35–115	46–105	MSD	40
4-Nitroaniline	10–137	39–120	MSD	40
4-Nitrophenol	12–141	41–121	MSD	40
Acenaphthene	30–113	49–97	MSD	40
Acenaphthylene	18–128	46–99	MSD	40
Anthracene	21–134	51–108	MSD	40
Benzaldehyde	10–106	10–106	MSD	40
Benzo(a)anthracene	33–121	54–109	MSD	40
Benzo(a)pyrene	31–122	51–114	MSD	40

Table 2-6. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples for Sediment Samples

	Accuracy		Precision	
Analyte	Matrix Spike Recovery (Percent)	LCS Recovery (Percent)	Type of Duplicate	Control Limit RPD
Benzo(b)fluoranthene	33–124	53–110	MSD	40
Benzo(ghi)perylene	26–126	50–110	MSD	40
Benzo(k)fluoranthene	36–117	52–112	MSD	40
Benzoic acid	0–176	10–104	MSD	40
Benzyl alcohol	25–106	35–96	MSD	40
Bis(2-Chloroethoxy)methane	33–106	45–93	MSD	40
Bis(2-chloroethyl)ether	29–103	42–88	MSD	40
Bis(2-Ethylhexyl)phthalate	35–126	51–115	MSD	40
Butyl benzyl phthalate	34–125	51–114	MSD	40
Carbazole	49–118	51–113	MSD	40
Chrysene	33–121	53–109	MSD	40
Dibenzo(a,h)anthracene	40–118	53–114	MSD	40
Dibenzofuran	35–112	47–101	MSD	40
Diethyl phthalate	37–128	45–121	MSD	40
Dimethyl phthalate	36–123	48–113	MSD	40
Di-n-butyl phthalate	34–139	50–124	MSD	40
Di-n-octylphthalate	36–136	51–121	MSD	40
Fluoranthene	25–135	48–116	MSD	40
Fluorene	31–116	46–103	MSD	40
Hexachlorobenzene	37–119	50–109	MSD	40
Hexachlorobutadiene	27–107	42–91	MSD	40
Hexachlorocyclopentadiene	10–79	10–68	MSD	40
Hexachloroethane	31–91	40–87	MSD	40
Indeno[1,2,3-cd]pyrene	29–132	53–112	MSD	40
Isophorone	33–103	42–93	MSD	40
Naphthalene	31–103	44–92	MSD	40
Nitrobenzene	34–99	44–92	MSD	40
N-Nitrosodi-n-propylamine	32–107	41–96	MSD	40
N-Nitrosodiphenylamine	14–143	44–113	MSD	40
Pentachlorophenol	10–135	39–112	MSD	40
Phenanthrene	35–119	52–108	MSD	40
Phenol	18–106	40–87	MSD	40
Pyrene	24–126	45–116	MSD	40
Vista				
Polychlorinated Dioxins and Furans				
2,3,7,8-TCDD	NA	67–158	NA	NA
2,3,7,8-TCDF	NA	75–158	NA	NA
1,2,3,7,8-PeCDD	NA	70–142	NA	NA
1,2,3,7,8-PeCDF	NA	80–134	NA	NA
2,3,4,7,8-PeCDF	NA	68–160	NA	NA
1,2,3,4,7,8-HxCDD	NA	70–164	NA	NA
1,2,3,6,7,8-HxCDD	NA	76–134	NA	NA
1,2,3,7,8,9-HxCDD	NA	64–162	NA	NA

Table 2-6. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples for Sediment Samples

	Accuracy		Precision	
Analyte	Matrix Spike Recovery (Percent)	LCS Recovery (Percent)	Type of Duplicate	Control Limit RPD
1,2,3,4,7,8-HxCDF	NA	72–134	NA	NA
1,2,3,6,7,8-HxCDF	NA	84–130	NA	NA
1,2,3,7,8,9-HxCDF	NA	78–130	NA	NA
2,3,4,6,7,8-HxCDF	NA	70–156	NA	NA
1,2,3,4,6,7,8-HpCDD	NA	70–140	NA	NA
1,2,3,4,6,7,8-HpCDF	NA	82–122	NA	NA
1,2,3,4,7,8,9-HpCDF	NA	78–138	NA	NA
OCDD	NA	78–144	NA	NA
OCDF	NA	63–170	NA	NA

Notes:

LCS = laboratory control sample
MSD = matrix spike duplicate
NA = not applicable
PCB = polychlorinated biphenyl
RPD = relative percent difference

Control limits are updated periodically by the laboratories. Control limits that are in effect at the laboratory at the time of analysis will be used for sample analysis and data validation. These may differ slightly from the control limits shown in this table.

[a] RPD control limit is not applicable for grain size. Laboratory control limit is ±10% in the weight of the fraction.

Table 2-7. Laboratory Control Limits for Surrogate Recoveries in Equipment Rinse Blanks

Analyte	Control Limits for Percent Recovery
Columbia Analytical Services	
Organochlorine Pesticides	
Tetrachloro-m-xylene	20–102
Decachlorobiphenyl	35–128
PCB Aroclors	
Tetrachloro-m-xylene	21–114
Decachlorobiphenyl	36–113
Semivolatile Organic Compounds	
Nitrobenzene-d5	46–115
Phenol-d6	38–107
Terphenyl-d14	22–146
2-Fluorophenol	39–103
2-Fluorobiphenyl	48–114
2,4,6-Tribromophenol	46–127
Vista	
Polychlorinated Dioxins and Furans	
13C-2,3,7,8-TCDD	25–164
13C-1,2,3,7,8-PeCDD	25–181
13C-1,2,3,4,7,8-HxCDD	32–141
13C-1,2,3,6,7,8-HxCDD	28–130
13C-1,2,3,4,6,7,8-HpCDD	23–140
13C-OCDD	17–157
13C-2,3,7,8-TCDF	24–169
13C-1,2,3,7,8-PeCDF	24–185
13C-2,3,4,7,8-PeCDF	21–178
13C-1,2,3,4,7,8-HxCDF	26–152
13C-1,2,3,6,7,8-HxCDF	26–123
13C-2,3,4,6,7,8-HxCDF	28–136
13C-1,2,3,7,8,9-HxCDF	29–147
13C-1,2,3,4,6,7,8-HpCDF	28–143
13C-1,2,3.4,7,8,9-HpCDF	26–138
13C-OCDF	17–157
37Cl4-2,3,4,7,8-TCDD	35–197

Notes:

PCB = polychlorinated biphenyl

Control limits are updated periodically by the laboratories. Control limits that are in effect at the laboratory at the time of analysis will be used for sample analysis and data validation. These may differ slightly from the control limits shown in this table.

Table 2-8. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples for Equipment Rinse Blanks

	Accuracy		Precision	
Analyte	Matrix Spike Recovery (Percent)	LCS Recovery (Percent)	Type of Duplicate	Control Limit RPD
Columbia Analytical Services				
Metals				
Antimony	86–116	90–113	Duplicate	20
Arsenic	79–121	90–112	Duplicate	20
Barium	80–124	91–113	Duplicate	20
Beryllium	87–114	91–113	Duplicate	20
Cadmium	71–142	93–113	Duplicate	20
Chromium	89–117	93–114	Duplicate	20
Cobalt	88–117	93–114	Duplicate	20
Copper	86–113	91–111	Duplicate	20
Lead	75–130	92–113	Duplicate	20
Manganese	84–121	92–112	Duplicate	20
Molybdenum	91–116	92–113	Duplicate	20
Nickel	86–120	91–118	Duplicate	20
Selenium	82–119	88–113	Duplicate	20
Silver	79–120	93–110	Duplicate	20
Thallium	75–125	80–120	Duplicate	20
Vanadium	89–115	92–111	Duplicate	20
Zinc	87–113	92–112	Duplicate	20
Organochlorine Pesticides				
4,4'-DDD	29–125	33–132	MSD	30
4,4'-DDE	24–129	41–116	MSD	30
4,4'-DDT	28–139	42–143	MSD	30
Aldrin	18–111	10–102	MSD	30
alpha-BHC	31–123	36–122	MSD	30
beta-BHC	31–118	42–125	MSD	30
delta-BHC	40–129	48–123	MSD	30
gamma-BHC (Lindane)	31–123	44–117	MSD	30
Dieldrin	32–121	50–115	MSD	30
Endosulfan I	17–118	35–115	MSD	30
Endosulfan II	19–122	28–128	MSD	30
Endosulfan sulfate	30–120	38–118	MSD	30
Endrin	34–133	48–126	MSD	30
Endrin aldehyde	10–108	27–104	MSD	30
Endrin ketone	34–113	30–124	MSD	30
alpha-Chlordane (cis-)	34–120	45–115	MSD	30
gamma-Chlordane (trans-)	35–119	47–113	MSD	30
Heptachlor	23–124	40–115	MSD	30
Heptachlor epoxide	28–122	49–109	MSD	30
Methoxychlor	30–137	43–143	MSD	30
PCB Aroclors				
PCB-1016	31–118	41–113	MSD	30
PCB-1260	47–115	47–117	MSD	30

Table 2-8. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples for Equipment Rinse Blanks

	Accuracy		Precision	
Analyte	Matrix Spike Recovery (Percent)	LCS Recovery (Percent)	Type of Duplicate	Control Limit RPD
Semivolatile Organic Compounds				
1,1'-Biphenyl	43–118	43–118	MSD	30
1,2,4-Trichlorobenzene	49–97	51–103	MSD	30
1,2-Dichlorobenzene	47–94	55–97	MSD	30
1,3-Dichlorobenzene	45–92	55–95	MSD	30
1,4-Dichlorobenzene	47–93	54–96	MSD	30
2,2'-oxybis(1-Chloropropane)	43–106	53–105	MSD	30
2,4,5-Trichlorophenol	60–112	62–117	MSD	30
2,4,6-Trichlorophenol	60–113	63–117	MSD	30
2,4-Dichlorophenol	55–105	59–107	MSD	30
2,4-Dimethylphenol	22–116	32–100	MSD	30
2,4-Dinitrophenol	38–122	33–126	MSD	30
2,4-Dinitrotoluene	62–123	61–126	MSD	30
2,6-Dinitrotoluene	69–113	65–122	MSD	30
2-Chloronaphthalene	48–106	54–107	MSD	30
2-Chlorophenol	49–100	59–101	MSD	30
2-Methylnaphthalene	57–100	57–106	MSD	30
2-Methylphenol (o-cresol)	52–100	51–108	MSD	30
2-Nitroaniline	23–138	62–117	MSD	30
2-Nitrophenol	54–104	58–108	MSD	30
3 & 4-Methylphenol	54–108	47–118	MSD	30
3,3'-Dichlorobenzidine	12–120	52–120	MSD	30
3-Nitroaniline	57–119	63–120	MSD	30
4,6-Dinitro-2-methylphenol	57–120	51–127	MSD	30
4-Bromophenyl-phenylether	61–114	64–118	MSD	30
4-Chloro-3-methylphenol	61–111	65–111	MSD	30
4-Chloroaniline	42–117	57–111	MSD	30
4-Chlorophenyl-phenyl ether	56–116	59–113	MSD	30
4-Nitroaniline	37–130	53–127	MSD	30
4-Nitrophenol	57–119	55–126	MSD	30
Acenaphthene	57–107	61–110	MSD	30
Acenaphthylene	59–101	58–109	MSD	30
Anthracene	59–112	62–116	MSD	30
Benzaldehyde	70–130	70–130	MSD	30
Benzo(a)anthracene	63–110	63–113	MSD	30
Benzo(a)pyrene	54–123	57–124	MSD	30
Benzo(b)fluoranthene	59–117	66–117	MSD	30
Benzo(ghi)perylene	61–116	68–116	MSD	30
Benzo(k)fluoranthene	57–118	63–119	MSD	30
Benzoic acid	10–136	10–109	MSD	30
Benzyl alcohol	51–105	53–111	MSD	30
Bis(2-Chloroethoxy)methane	55–101	57–108	MSD	30
Bis(2-chloroethyl)ether	51–97	56–104	MSD	30
Bis(2-Ethylhexyl)phthalate	58–122	64–122	MSD	30
Butyl benzyl phthalate	60–117	64–121	MSD	30
Carbazole	63–122	63–122	MSD	30
Chrysene	61–113	68–114	MSD	30
Dibenzo(a,h)anthracene	60–120	65–121	MSD	30
Dibenzofuran	61–110	62–112	MSD	30
Diethyl phthalate	61–122	62–122	MSD	30
Dimethyl phthalate	69–110	64–119	MSD	30

Table 2-8. Laboratory Control Limits for Matrix Spike and Laboratory Control Samples for Equipment Rinse Blanks

	Accuracy		Precision	
Analyte	Matrix Spike Recovery (Percent)	LCS Recovery (Percent)	Type of Duplicate	Control Limit RPD
Di-n-butyl phthalate	61–127	65–126	MSD	30
Di-n-octylphthalate	57–129	60–131	MSD	30
Fluoranthene	56–123	60–121	MSD	30
Fluorene	58–112	59–112	MSD	30
Hexachlorobenzene	56–115	63–117	MSD	30
Hexachlorobutadiene	44–94	54–99	MSD	30
Hexachlorocyclopentadiene	10–58	10–60	MSD	30
Hexachloroethane	40–101	50–100	MSD	30
Indeno[1,2,3-cd]pyrene	62–113	68–116	MSD	30
Isophorone	60–99	57–106	MSD	30
Naphthalene	51–99	51–106	MSD	30
Nitrobenzene	31–141	56–108	MSD	30
N-Nitrosodi-n-propylamine	53–109	56–112	MSD	30
N-Nitrosodiphenylamine	10–135	58–121	MSD	30
Pentachlorophenol	42–127	60–116	MSD	30
Phenanthrene	61–111	64–116	MSD	30
Phenol	47–101	48–105	MSD	30
Pyrene	52–121	53–124	MSD	30
Vista				
Polychlorinated Dioxins and Furans				
2,3,7,8-TCDD	NA	67–158	NA	NA
2,3,7,8-TCDF	NA	75–158	NA	NA
1,2,3,7,8-PeCDD	NA	70–142	NA	NA
1,2,3,7,8-PeCDF	NA	80–134	NA	NA
2,3,4,7,8-PeCDF	NA	68–160	NA	NA
1,2,3,4,7,8-HxCDD	NA	70–164	NA	NA
1,2,3,6,7,8-HxCDD	NA	76–134	NA	NA
1,2,3,7,8,9-HxCDD	NA	64–162	NA	NA
1,2,3,4,7,8-HxCDF	NA	72–134	NA	NA
1,2,3,6,7,8-HxCDF	NA	84–130	NA	NA
1,2,3,7,8,9-HxCDF	NA	78–130	NA	NA
2,3,4,6,7,8-HxCDF	NA	70–156	NA	NA
1,2,3,4,6,7,8-HpCDD	NA	70–140	NA	NA
1,2,3,4,6,7,8-HpCDF	NA	82–122	NA	NA
1,2,3,4,7,8,9-HpCDF	NA	78–138	NA	NA
OCDD	NA	78–144	NA	NA
OCDF	NA	63–170	NA	NA

Notes:

LCS = laboratory control sample
MSD = matrix spike duplicate
NA = not applicable
PCB = polychlorinated biphenyl
RPD = relative percent difference

Control limits are updated periodically by the laboratories. Control limits that are in effect at the laboratory at the time of analysis will be used for sample analysis and data validation. These may differ slightly from the control limits shown in this table.

APPENDIX A

FIELD SAMPLING PLAN

SAMPLING AND ANALYSIS PLAN: SUPPLEMENTAL INVESTIGATION OF THE LYMAN MILL REACH SEDIMENT AND FLOOD PLAIN SOILS

APPENDIX A
FIELD SAMPLING PLAN

Centredale Manor Restoration Project Superfund Site in North Providence, Rhode Island

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June 21, 2010

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ACRONYMS AND ABBREVIATIONS

CMRP Site	Centredale Manor Restoration Project Superfund Site
COC	chain-of-custody
COPC	constituents of potential concern
DGPS	differential global positioning system
FSP	Field Sampling Plan
GPS	global positioning system
HSP	Health and Safety Plan
MSL	mean sea level
PE	performance evaluation
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
SOP	standard operating procedure
USEPA	United States Environmental Protection Agency

1 INTRODUCTION

The area of the Centredale Manor Restoration Project Superfund Site (CMRP Site) that has been designated by the United States Environmental Protection Agency (USEPA) as the Lyman Mill Reach Stream Sediment and Flood Plain Soil (including the Oxbow Area) is the focus of the supplemental investigation presented in this Field Sampling Plan (FSP). This area of the CMRP Site generally comprises:

1. The sediments in the reach of the Woonasquatucket River between Allendale Dam and the northern extent of Lyman Mill Pond,
2. The flood plain soils in the Oxbow Area,
3. The emergent wetlands east of the Oxbow Area,
4. The emergent wetland at the confluence of Assapumpset Brook and the Woonasquatucket River (western shore of Lyman Mill Pond), and
5. The emergent wetland at the southeastern portion of Lyman Mill Pond.

As part of the Remedial Investigation of the CMRP Site, USEPA collected three sediment samples and four surface wetland soil samples in the Oxbow Area, which were analyzed for dioxins/furans, PCBs/pesticides, semivolatile organic compounds, and metals. Dioxins/furans, PCBs, pesticides, semivolatile organic compounds and metals were all detected in the USEPA samples collected from the Oxbow Area (USACE 2006). Using these data, USEPA conducted baseline human health and ecological risk assessments for the Oxbow Area (USACE 2006).

The Oxbow Area is a complex of riverine, emergent, scrub-shrub, and forested wetland, and adjacent uplands area encompassing approximately 40 acres. Most of the wetlands are within the 100-year floodplain of the Woonasquatucket River (USACE 2008). The data from the seven samples collected by USEPA in the Oxbow Area are insufficient to adequately characterize the spatial or vertical distribution of contaminants detected in soil and/or sediment within the Oxbow Area. Also, there are insufficient data to accurately characterize the extent of contamination present in floodplain soils in the emergent wetlands noted in items 3 through 5 above. .

The purpose of this supplemental investigation is to collect additional surface and subsurface samples to obtain analytical data that can be used to: (i) better define the extent of contamination; and (ii) to reassess the potential baseline risks to human and ecological receptors that may be exposed to the Lyman Mill Reach Stream Sediment and Flood Plain Soil. This FSP identifies the sampling procedures and field documentation reporting and management requirements associated with this supplemental investigation.

2 SAMPLING PROCEDURES

2.1 SCHEDULE

The start date for the surface soil study will be determined following USEPA approval of this FSP, the Site-Specific Health and Safety Plan (HSP), and the associated Quality Assurance Project Plan (QAPP). However, for planning purposes, it is anticipated that the field sampling event will begin in August of 2010.

2.2 FIELD SURVEY AND SAMPLING METHODS

Access to all of the sampling stations should be possible on foot, or with the use of wooden planks to provide additional stability for sampling activities in muddy or recently flooded stations. If necessary, one or more sleds will be used to transport soil collection and compositing equipment, sample coolers, and multiple sampling equipment boxes containing sample jars and other ancillary equipment. Also, a small boat will be mobilized and will be used as needed to access some locations from the water-side if land-side access is precluded.

2.2.1 Field Equipment and Supplies

Field equipment and supplies include sampling equipment, utensils, decontamination supplies, sample containers, coolers, shipping containers, log books and forms, personal protection equipment, and personal gear. Protective wear (e.g., disposable nitrile gloves) is required to minimize the possibility of cross-contamination between sampling locations. Additional information on protective wear required for this project is described in the HSP provided in Attachment A1. Standard operating procedures (SOPs) for collecting and handling field samples can be found in Attachment A2.

Surface soil samples (0 to 12 in.; 0 to 30 cm) will be collected using stainless-steel spades, shovels, or electric impact hand-held corers. A hand-held electric impact corer will be used for the vertical profile subsurface soil collection in the vadose zone (12 to 24 in. [30 to 61 cm] and 24 to 36 in. [61 to 91 cm]).

Sample jars, preservatives, distilled/deionized water, coolers, and packaging material for the samples will be supplied by the analytical laboratory. Details on the numbers and type of sample containers are provided in the QAPP and in Table A-1 of this FSP. The field personnel in charge of sample handling will use a sample matrix (Table A-2) as a quality control (QC) check to ensure that all samples have been collected at a given station. This table includes the total number and type of sample jars required for each analysis at each sampling station.

Commercially available, pre-cleaned jars will be used for the samples, and the testing laboratories will maintain a record of certification from the suppliers. The bottle shipment documentation will include batch numbers. With this documentation, jars can be traced to the supplier, and, if necessary, bottle-wash analysis results can be reviewed. The bottle-wash certificate documentation will be archived in Integral's project file.

Sample containers will be clearly labeled at the time of sampling. Labels will include the task name, sample location and number, sampler's initials, analyses to be performed, and sample date and time. Sample numbering and identification procedures are described in detail in Sections 3.3 and 3.4.

2.2.2 Sample Location Positioning

Latitude and longitude coordinates will be obtained at the locations where soil samples are collected. A differential global positioning system (DGPS) will be used to document the sample collection locations. The standard projection method to be used during field activities is Horizontal Datum: NAD1983 State Plane, Rhode Island, US feet, mean sea level (MSL). The positioning objective is to accurately determine and record the positions of all sampling locations to within ±2 m. Proposed soil sampling locations are shown in Figure 1 and location coordinates are provided in Table A-3 (42 locations).

The DGPS unit consists of a global positioning system (GPS) receiver and a reference differential receiver located at a horizontal control point. At the control point, the GPS-derived position is compared with the known horizontal location, offsets or biases are calculated, and the correction factors are telemetered to the GPS receiver. Positioning accuracies on the order of ±1 to 3 m can be achieved by avoiding the few minutes per day when the satellites are not providing the appropriate quality of signal (SOP AP-06). The GPS unit provides the operator with a listing of the time intervals during the day when accuracies are decreased. Avoidance of these time intervals permits the operator to maintain better positioning accuracy (SOP AP-06).

2.2.3 Surface Soil Sample Collection

The equipment and procedures that will be used to collect surface soil samples during the 2010 soil study are discussed in the following sections. The field locations that will be sampled are listed in Table A-4. The holding time requirements for the soil samples following field collection are specified in Table A-1.

Surface soil samples (0 to 12 inches; 0 to 30 cm) for characterization of the general area or human health exposure may be collected with a variety of sampling equipment depending upon the conditions encountered in the field: stainless-steel spades, spoons or shovels, a stainless-steel hand corer, or an electric impact hand held corer in accordance with standard methods used by USEPA (1997). Methods for surface soil sampling are provided in SOP SL-05.

One surface soil sample will be collected at each of 42 sampling locations; 32 for the general area samples and 10 for the general surface human health exposure samples. All of the samples will be analyzed for primary constituents of potential concern (COPCs), grain size, pH, total organic carbon, and percent solids.

Thirty-five surface soil samples will be collected from the Oxbow Area, four from the emergent wetland east of the Oxbow, two from the emergent wetland at the confluence of Assapumpset Brook and the Woonasquatucket River, and one from the emergent wetland in the southeastern corner of Lyman Mill Pond.

A variety of vegetation is present in the Oxbow Area, from scrub-shrub to broad-leaf deciduous trees. Sampling stations will be relocated slightly if coincident with woody vegetation. Non-woody vegetative material (such as Joe pye weed, purple loosestrife, sedges, grasses; USACE 2008) will be removed from the surface prior to sample collection and from the sample. Removal of material from the sample will be documented in the field log book.

Surface soil samples will be placed into a decontaminated, stainless-steel bowl and covered with aluminum foil until a sufficient volume of soil (approximately 2 L per station) is collected. In accordance with USEPA (1997), once enough soil volume is collected, the soil will be homogenized in the bowl using a stainless-steel spoon until the soil attains a visually uniform color and texture. Aliquots of the homogenized soil sample will then be placed in labeled, laboratory-cleaned sample containers with Teflon-lined lids (Table A-1). Each sample container will be clearly labeled with the task name, sample number, type of analysis to be performed, date and time, and initials of person(s) preparing the sample. Immediately after sample containers are filled, the samples will be stored on ice (4±2°C).

As stated above, the sample matrix table (Table A-2) contains a summary of the type and number of jars that are required for each analysis at each sampling station. Integral's field lead and field personnel in charge of sample handling will use this table as a QC check to ensure that all samples at a given station are collected and that the appropriate sample container is used for each sample.

2.2.4 Subsurface Core Sample Collection

The equipment and procedures to be used to collect subsurface soil samples during the study are discussed in the following sections. The field locations that will be sampled are listed in Table A-4. The holding time requirements for the soil samples following field collection are specified in Table A-1. The field staff will use a utility cart or sled, or hand carry all the necessary sampling equipment for soil coring at each of the assigned sampling sites, taking care not to step on the sampling area where surface soil samples (0 to 12 in., 0 to 30 cm) will be or already have been collected. Sampling of subsurface soils with a portable electric impact corer will follow procedures in SOP SD-08 and SOP SL-06. A portable generator, used to power the

impact hammer, will be placed as far away as possible and downwind from the sampling site. The generator will not be placed over an area where other surface soil samples are to be collected.

The subsurface samples will be collected from a depth of 12 to 24 in. (30 to 61 cm) and 24 to 36 in. (61 to 91 cm) and will be collocated with the surface soil samples discussed above in Section 2.2.3. The maximum depth of subsurface soil collection will be 36 in (91 cm) unless the water table is encountered at a shallower depth. If the water table is encountered, the sampling depth will be terminated at the water table to ensure that only soil from the vadose zone is sampled. If possible, the 12 to 24 in. and 24 to 36 in. samples will be collected in the same soil core and separated into the appropriate 12-in. intervals prior to compositing. A minimum of two cores will be collected at each vertical profile sampling station to obtain the necessary volume of soil for all required analyses. If sample volume requirements dictate the need for additional soil, then additional co-located cores will be collected. Soil from the two intervals of interest will be composited separately in stainless-steel bowls prior to placement into the appropriate sample containers. Any separate soil horizons that are observed in the core will be noted on the field form (Attachment 3), but will not alter the collection interval.

If less than 80 percent core recovery versus penetration is achieved in a soil coring effort, the recovered core will be retained but considered insufficient, and another attempt will be made to recover a soil core at the same location. If the specified penetration depth is not achieved after two attempts, the station may be relocated slightly. If the slight relocation of the station does not improve the penetration depth, the station may be temporarily abandoned and Integral's project manager will be notified to assess an alternate method of sample collection.

After the soil cores have been collected, both ends of the cores designated for chemical analysis will be securely capped; labeled with the station identifier, core section, and soil orientation; and fastened in an upright position.

Processing of the core will preferentially take place at the sampling station. If unstable soil conditions or inclement weather occur, sample handling will take place at a specified processing location onsite. At the processing area, the core liner will be laid out horizontally on a clean work surface. The content of the core will be extruded with a plunger onto a clean sheet of aluminum foil. If the core contains a liner that cannot be extruded, the core liner will be cut lengthwise and the core split open. Split cores collected without a liner will be placed next to a tape measure and a station identifier, and will be photographed. Cores will be inspected for physical characteristics and described on a soil core log (Attachment A3).

Cores designated for chemical analysis will be sectioned into 12-in. (30-cm) intervals. Soil touching the sides of the core tube will be excluded from each sample to minimize cross contamination between the 12-in. (30-cm) subsections.

Subsurface soil samples will be placed into a decontaminated, stainless-steel bowl and covered with aluminum foil until a sufficient volume of soil (approximately 2 L per station) is collected. In accordance with USEPA (1997), once enough soil volume is collected, the soil will be homogenized in the bowl using a stainless-steel spoon until the soil attains a visually uniform color and texture. Aliquots of the homogenized soil sample will then be placed in labeled, laboratory-cleaned sample containers with Teflon-lined lids (Table A-1). Each sample container will be clearly labeled with the task name, sample number, type of analysis to be performed, date and time, and initials of person(s) preparing the sample. Immediately after sample containers are filled, the samples will be stored on ice (4±2°C).

As stated above, the sample matrix table (Table A-2) shows the total number of sample jars that are needed for each analysis. Integral's field lead and field personnel in charge of sample handling in the field will use this table as a QC check to ensure that all samples at a given station are collected and that the appropriate sample container is used for each sample.

2.2.5 Equipment Decontamination

Before surface soil sampling begins at a location, the sampling implements (stainless-steel spades or shovels, or a stainless-steel hand corer) will be scrubbed with an aqueous solution of standard detergent (e.g., Alconox® or Liquinox®), rinsed with laboratory-grade distilled/deionized water, and air dried. The same decontamination sequence will apply to equipment used for compositing the soil samples (i.e., stainless-steel bowls and spoons). After cleaning, the decontaminated sample homogenizing equipment will be covered with aluminum foil to protect it from possible contamination.

Prior to subsurface sampling, all core liners will be washed in sequence with an aqueous solution of standard detergent (e.g., Alconox or Liquinox), rinsed with laboratory-grade distilled/deionized water, and then air-dried. During storage and transport, decontaminated Lexan core liners will be capped at both ends to prevent contamination.

All non-dedicated sampling equipment that comes into contact with the soil samples (e.g., core catchers, core liners, stainless-steel bowls, and utensils) will be decontaminated prior to use and between samples. Non-dedicated sampling equipment will be decontaminated following procedures in SOP SL-01, except that no solvent rinse will be used. If samples are collected that include obvious oily contamination, the sampling equipment used to collect and process them will be decontaminated using a separate decontamination station dedicated to decontaminate heavily impacted equipment. This equipment will be wiped with a solvent following the initial decontamination, and it will undergo a second decontamination sequence using the standard decontamination procedures used for the non-oil-impacted equipment.

2.3 FIELD QUALITY CONTROL SAMPLES

Field QC samples will be used to assess sample variability and to evaluate potential sources of contamination. The types of QC samples that will be collected are described in this section. Detailed information on quality assurance (QA)/QC procedures, limits, and reporting are described in detail in the QAPP. The estimated numbers of field QC samples to be collected is listed in the sample matrix table (Table A-2). If QC problems are encountered, they will be brought to the attention of the Integral's QA coordinator. Corrective actions, if appropriate, will be implemented to meet the task's data quality criteria as outlined in the QAPP.

Field QC samples will include field duplicate samples, a performance evaluation (PE) sample, equipment rinse blanks, and rinsate blanks. The following QC samples will be collected in the field and analyzed by the analytical laboratory:

- Field duplicate samples will be collected and analyzed to assess the variability associated with sample processing and laboratory variability. Field duplicate samples will be collected at a minimum frequency of one field split sample per 20 soil samples. Samples will be assigned unique numbers and will not be identified as field splits to the laboratory. Field split samples will be collected from both surface and subsurface soil samples for chemical analysis. A minimum of one field split sample will be collected for each kind of sample collected.
- PE samples are samples of known concentration that provide a measure of analytical performance and/or analytical method bias. When provided (by the USEPA), PE samples for soil will be submitted from the field at a frequency of once per sampling event.
- Equipment rinse blanks will be collected to help identify possible contamination from the sampling environment or from the sampling equipment (e.g., stainless-steel spade, coring device, spoons, and bowls). Equipment rinse blanks will be generated at a frequency of once per sampling event. Field equipment rinse blanks will be collected from both surface and subsurface soil samples for chemical analysis. All equipment rinse samples will be noted clearly in the field log (e.g., sample identifier, equipment type, date and time of collection, analysis, and filter lot number). A minimum of one field equipment rinse blank will be collected for each kind of sampling equipment used for chemical analyses. One equipment rinse blank will be prepared for each analysis type requested.
- A rinsate blank will be prepared in the field to evaluate potential background concentrations present in the water used for the equipment rinse blank. One rinsate blank will be collected at a minimum frequency of one for each batch of rinsate used for collecting the equipment rinse blanks.

2.4 SAMPLE PACKAGING AND TRANSPORT

As mentioned above, sample coolers and packing materials will be supplied by the analytical laboratories. Individual sample jars will be labeled and placed into plastic bags and sealed. Samples then will be packed in a cooler lined with a large plastic bag according to SOP AP-01. Glass jars will be packed to prevent breakage and separated in the cooler by bubble wrap or other shock-absorbent material. Ice in sealed plastic bags then will be placed in the cooler to maintain a temperature of approximately 4°C (±2°C). When the cooler is full, the chain-of-custody (COC) form will be placed into a zip-locked bag and taped to the inside lid of the cooler. A temperature blank will be added to each cooler. Each cooler will be sealed with two COC seals, one each on the front and side of the cooler. Labels indicating "This End Up" with an arrow and "Fragile" will be attached to each cooler.

The shipping containers will be clearly labeled (i.e., name of task, time and date container was sealed, person sealing the cooler, and company name and address) for positive identification. These packaging and shipping procedures are in accordance with U.S. Department of Transportation regulations (49 CFR 173.6 and 49 CFR 173.24). Coolers containing samples for chemical analyses will be transported to the laboratory by courier or overnight shipping service.

After the chemistry samples have been received by the laboratory, they will be stored under refrigeration (4±2°C). Archive soil samples collected from each composite sample for possible future analysis will be stored frozen at –20°C.

2.5 STUDY-DERIVED WASTES

Any soil sample remaining after processing will be deposited in the vicinity of the collection area. Any dry waste (e.g., contaminated boots, bibs, Tyvek™ suits, contaminated soils) present at the end of the sampling event will be segregated and containerized (e.g., 50-gallon drums) and disposed of by a subcontractor specialized in hazardous waste removal. To minimize liquid study-derived waste, sampling will be conducted using separate, pre-cleaned sampling tools. However, should liquid wastes be generated, they will be containerized and disposed of by the hazardous waste disposal subcontractor. The subcontractor will be required to have, at a minimum, a drum management service that provides the following:

- Proper waste identification including full analytical capability
- Pick up and disposal of a broad range of hazardous wastes
- Safe and proper transportation
- Environmentally sound treatment and disposal
- Regularly scheduled service visits with manifest and label preparation.

All disposable materials used for sample collection and processing, such as paper towels and gloves, will be placed in heavyweight garbage bags or other appropriate containers. Disposable supplies that do not contain CMRP Site soil will be removed from the CMRP Site by sampling personnel and will be placed in a normal refuse container for disposal at a solid waste landfill.

3 FIELD DOCUMENTATION

3.1 RECORD KEEPING

The integrity of each sample from the time of collection to the point of data reporting must be maintained. Proper record-keeping and COC procedures will enable samples to be traced from collection to final disposal. Representative photographs will be taken of each area where samples are collected (e.g., along the southern end of the CMRP Site near the Allendale Dam, sampling near apartment complexes, sampling near the emergent wetland at Assapumpset Brook and Lyman Mill Pond). A photograph will be taken of each subsurface soil interval collected at each area of the CMRP Site. Site photos from various angles and close-up views of the overall conditions will also be collected.

3.2 FIELD LOG BOOK

All field activities and observations will be noted in a log book. The field log book will be a bound document and may contain individual field and sample log forms (depending on the sampling activity). Information will include personnel, date, time, station designation, sampler, types of samples collected, and general observations. Any changes that occur during sampling (e.g., personnel, responsibilities, or deviations from the FSP) and the reasons for these changes will be documented. The log book will identify onsite visitors (if any) and the number of photographs taken at each sampling location. Each field lead is responsible for ensuring that their respective field log book and all field data forms are correct. Requirements for log book entries will include the following:

- Log books will be bound, with consecutively numbered pages
- Removal of any pages, even if illegible, will be prohibited
- Entries will be made legibly with black (or dark) waterproof ink
- Unbiased, accurate language will be used
- Entries will be made while activities are in progress or as soon thereafter as possible (the date and time that the notation is made should be recorded, as well as the time of the observation itself)
- Each consecutive day's first entry will be made on a new, blank page
- The date and time, based on a 24-hour clock (e.g., 0900 a.m. for 9:00 a.m. and 2100 for 9:00 p.m.), will appear on each page.

In addition to the preceding requirements, the person recording the information must initial and date each page of the field log book. If more than one individual makes entries on the same

page, each recorder must initial and date each entry. The bottom of the page must be signed and dated by the individual who makes the last entry.

Log book corrections will be made by drawing a single line through the original entry, allowing the original entry to be read. The corrected entry will be written alongside the original. Corrections will be initialed and dated and may require a footnote for explanation.

The type of information that may be included in the field log book and/or field data forms includes the following:

- Task name, task location, and task number
- Task start date and end date
- Weather conditions
- Name of person making entries and names of additional field staff
- Onsite visitors, if any
- Sampling vessel, if any
- Station name and location
- Date and collection time of each sample
- The sample number for each sample to be submitted for laboratory analysis
- The sampling location name, date, equipment type, and sampling location coordinates derived from GPS
- Specific information on each type of sampling activity
- Observations made during sample collection, including weather conditions, complications, and other details associated with the sampling effort
- Sample description (source and appearance, such as color, presence of anthropogenic material, and presence and type of biological structures, other debris, and odor)
- Soil penetration depth (nearest 0.5 cm) based on soil depth at the center of the excavation
- Any visible debris near any of the sampling locations
- Any surface vegetation that is removed from the sampling location prior to sampling
- The locations of any surface water runoff or seeps that are located near any of the sampling stations
- The number of photographs taken at the sampling location
- A record of CMRP Site health and safety meetings, updates, and related monitoring
- Any deviation from the FSP and reasons for deviation (may require a Field Change Form).

In addition, a sampling location map will be updated during sampling and will be maintained throughout the sampling event. All log books must be completed at the time that any observations are made. Copies of all log books and forms will be retained by the technical team.

3.3 CHAIN-OF-CUSTODY PROCEDURES

Samples are in custody if they are in the custodian's view, stored in a secure place with restricted access, or placed in a container secured with custody seals (see SOP AP-03). A COC record will be signed by each person who has custody of the samples and will accompany the samples at all times. Copies of the COC will be included in laboratory and QA/QC reports. Attachment A3 contains an example of the COC form that will be used during the 2010 soil study.

At a minimum, the form will include the following information:

- Site name
- Field lead's name and team members responsible for collection of the listed samples
- Collection date and time for each sample
- Sample type (i.e., sample for immediate analysis or archive)
- Number of sample containers shipped
- Requested analyses
- Sample preservation information (if any)
- Name of the carrier relinquishing the samples to the transporter, noting date and time of transfer and the designated sample custodian at the receiving facility.

Integral's field lead will be the designated field sample custodian for sampling events and will be responsible for all sample tracking and COC procedures for the samples that the team collects in the field. The field sample custodian will be responsible for final sample inventory and will maintain sample custody documentation. The field sample custodian will complete COC forms prior to removing samples from the field. Upon transferring samples to the shipping courier (as appropriate), the field sample custodian will sign, date, and note the time of transfer on the COC form. The original COC form will be transported with the samples to the laboratory. All samples will be shipped to the testing laboratories in either coolers or shipping containers sealed with custody seals.

Each laboratory will designate a sample custodian who will be responsible for receiving samples and documenting their progress through the laboratory analytical process. The sample custodian for each laboratory will establish the integrity of the custody seals upon sample

arrival at the laboratory. The laboratory sample custodian will also ensure that the COC and sample tracking forms are properly completed, signed, and initialed upon receipt of the samples.

When the laboratory receives the samples, the laboratory sample custodian will conduct an inventory by comparing sample labels to those on the COC document. The custodian will enter the sample number into a laboratory tracking system by task code and sample designation. The custodian will assign a unique laboratory number to each sample and will be responsible for distributing the samples to the appropriate analyst or for storing samples at the correct temperature in an appropriate secure area.

3.4 STATION NUMBERING

All stations will be assigned a unique identification code based on a designation scheme designed to suit the needs of the field personnel, data management, and data users. Station numbers will include "SS" to indicate soil samples followed by a one-letter code for the receptor group of concern to be assessed at a given location (G = general concern, H = human concern). All station numbers will then be followed by a two-digit number (e.g., 01, 02, or 03). The station numbers will increase as the stations move north to south (downstream). An example station number for the 2010 soil study would be SS_H-01, reflecting the first soil sample station designation for human health concerns.

Station numbers will not be recorded on sample labels or COC forms to prevent analytical laboratories from seeing the relationships between samples and stations.

3.5 SAMPLE IDENTIFIERS

Each soil sample from a given station will also have a unique label identifier. Sample identifiers will be established before field sampling begins and will be assigned to each sample as it is collected. Sample identifiers consist of codes designed to fulfill three purposes: 1) to identify related samples (i.e., field split samples) to ensure proper data analysis and interpretation; 2) to obscure the relationships between samples so that laboratory analysis will be unbiased by presumptive similarities between samples; and 3) to track individual sample containers to ensure that the laboratory receives all of the material associated with a single sample. To accomplish these purposes, each container is assigned a sample number and a tag number. These codes and their uses are described below:

- A sample identifier for each surface sample will be created as follows: the station number (e.g., SS_H-01), followed by a two-letter code for the kind of sample collected at a given location (SS = surface soil grab sample, CR = core subsurface soil sample). In addition, core samples for chemical analyses will also have a final alpha character

attached to the sample identifier that will distinguish between the different sample intervals of the core (e.g., A = 12 to 24 in. [30 to 61cm], B = 24 in. to 36 in. [61 to 90cm]). Two-digit sample numbers will follow the station-sample identifier. Example identifiers for a subsurface soil sample would be SS_G-20-CR1A. If a second core was required at a given station to obtain the required sample volume, then example sample identifiers for this second core would be SS_G-20-CR1Aa, SS_G-20-CR1Ab, and so on.

- The sample number is an arbitrary number assigned to each soil sample collected for a specific station identifier. All subsamples of a composited field sample will have the same sample number. Each field split sample and each field triplicate will have a different sample number, and the sample numbers of related field QC samples may not share any content. The sample number appears on the sample containers and the COC forms.
- A unique numeric sample tag number will be attached to each sample container. If the amount of material (i.e., everything associated with a single sample number) is too large for a single container, each container will have the same sample number and a different sample label with a unique sample tag number. A sample will also be split between containers if a different preservation technique is used for each container (i.e., because different analyses will be conducted). The sample tag number will appear on the COC forms. Tag numbers are used by laboratories only to confirm that they have received all of the containers that were filled and shipped. Data are reported by sample number.

Sample numbers will be assigned sequentially in the field, and sample labels will be preprinted with tag numbers.

For equipment rinse blanks, identical sample numbering for a designated sampling station will be used, with the exception that sample suffix designators will not be "SS" or "CR," but rather "1S" or "1C" to relate to stainless-steel spoon/bowl or Lexan core collection. Blanks ending in "1R" pertain to a non-equipment rinse blank. An example blank sample is SSRB-G-06_1S, which reflects an equipment rinse blank for stainless-steel spoon and bowl sample collection.

4 FIELD DATA MANAGEMENT AND REPORTING PROCEDURES

During field operations, effective data management is critical to providing consistent, accurate, and defensible data and data products. Daily field records (a combination of field log books, field forms, if any, and COC forms) will comprise the main documentation for field activities. Upon completion of sampling, field notes, data sheets (if any), and COC forms will be scanned to create an electronic record. Field data will be manually entered into the project database. One hundred percent of the transferred data will be verified based on hard copy records. Electronic QA checks to identify anomalous values will also be conducted following entry.

5 REFERENCES

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FIGURES

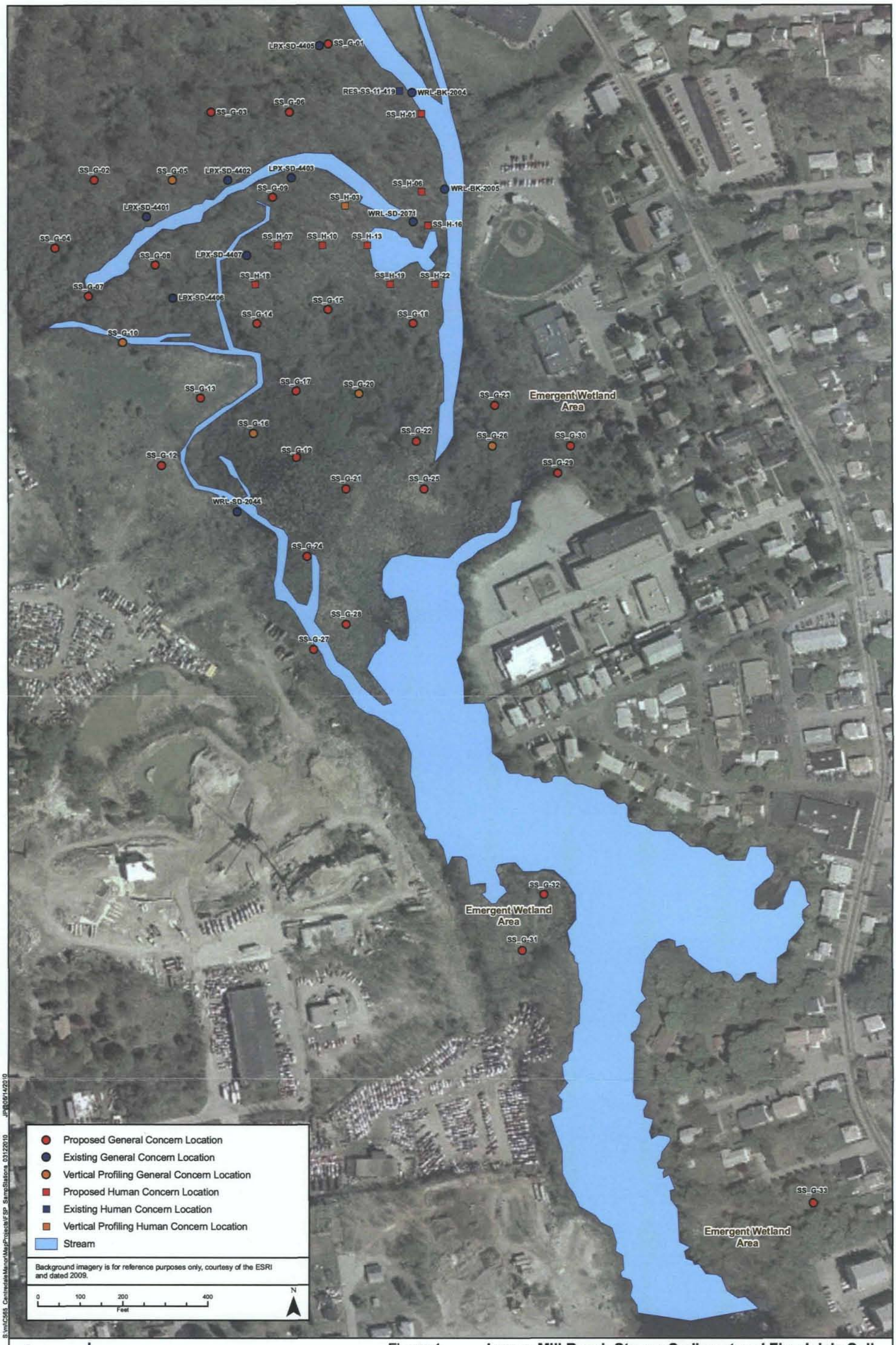

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Figure 1. **Lyman Mill Reach Stream Sediment and Floodplain Soil Sampling Locations**

Centredale Manor Restoration Project

TABLES

Table A-1. Sample Containers, Preservation, and Holding Time Requirements

Matrix	Container [a] Type	Container [a] Size	Laboratory	Parameter	Preservation	Holding Time	Sample Size [b]
Soil							
	WMG	8 oz	TBD	TOC	4±2°C	28 days	1 g
	WMG	8 oz	TBD	Metals	4±2°C	6 months	10 g
	WMG	16 oz	TBD	Grain Size	4±2°C	6 months	100 g
	WMG	8 oz	TBD	Percent solids	4±2°C	6 months	10 g
	WMG	8 oz	TBD	pH	4±2°C	14 days [e]	20 g
	WMG	8 oz	TBD	Dioxins/furans	4±2°C/Deep frozen (-20°C) [c]/ -10°C [d]	1 year/1 year [f]	10 g
	WMG	16 oz	TBD	Organochlorine pesticides	4±2°C	14 days/40 days [f]	25 g
	WMG	16 oz	TBD	PCBs (Aroclors)	4±2°C	14 days/40 days [f]	25 g
	WMG	16 oz	TBD	SVOCs	4±2°C	14 days/40 days [f]	25 g
Equipment Rinsate Blanks							
	HDPE	500 mL	TBD	Metals	5 mL of 1:1 HNO_3; 4±2°C	6 months	100 mL
	AG	1 L	TBD	Dioxins/furans	4±2°C	1 year/1 year [f]	1 L
	AG	1 L	TBD	Organochlorine pesticides	4±2°C	7 days/40 days [f]	1 L
	AG	1 L	TBD	PCBs (Aroclors)	4±2°C	7 days/40 days [f]	1 L
	AG	1 L	TBD	SVOCs	4±2°C	7 days/40 days [f]	1 L

Notes:

AG = amber glass
HDPE = high density polyethylene
PCB = polychlorinated biphenyl
SVOC = semivolatile organic compound
TOC = total organic carbon
WMG = wide mouth glass

[a] The size and number of containers may be modified by the analytical laboratory.
[b] Sample sizes may be modified once laboratory selection is made.
[c] Samples will be shipped to the laboratory on ice at 4±2°C. Once received at the laboratory, samples will be stored at -20°C.
[d] Extracts will be stored at -10°C.
[e] Published holding time does not exist. Holding time shown is based on best professional judgment.
[f] Holding time for samples prior to extraction/holding time for extracts.

Table A-2. Field Sample Collection Matrix

				Chemistry Grab and Core Samples				Equipment Rinse Blanks				
				Metals, TOC, pH, Percent Solids (8oz WMG) [a]	SVOCs, PCBs (Aroclors), Organo-chlorine Pesticides (16oz WMG) [a]	Dioxins/Furans (8oz WMG) [a]	Grain Size 16oz WMG[a]	Metals (500 mL HDPE) [a]	Dioxins/Furans (1L AG) [a]	Organo-chlorine Pesticides (1L AG) [a]	PCBs (Aroclors) (1L AG) [a]	SVOCs (1L AG) [a]
Station	Sample ID	Sample Type	Sample Group	4±2°C	4±2°C	4±2°C/ Deep frozen (-20°C)[b]/-10°C[c]	4±2°C	5 mL of 1:1 HNO3; 4±2°C	4±2°C	4±2°C	4±2°C	4±2°C
SS_G-01	SS_G-01-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-01	SS_G-01-SS1	QA-field split	General area	☐	☐	☐	☐					
SS_G-02	SS_G-02-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-03	SS_G-03-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-04	SS_G-04-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-05	SS_G-05-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-05	SS_G-05-CR1A	Core	General area	☐	☐	☐	☐					
SS_G-05	SS_G-05-CR1B	Core	General area	☐	☐	☐	☐					
SS_G-06	SS_G-06-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-06	SSRB-G-06_1S	Equipment rinse blank	General area					☐	☐	☐	☐	☐
SS_G-06	SSRB-G-06_1C	Equipment rinse blank	General area					☐	☐	☐	☐	☐
SS_G-06	SSRB_G-06-1R	Rinsate blank	NA					☐	☐	☐	☐	☐
SS_G-07	SS_G-07-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-08	SS_G-08-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-09	SS_G-09-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-10	SS_G-10-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-10	SS_G-10-CR1A	Core	General area	☐	☐	☐	☐					
SS_G-10	SS_G-10-CR1B	Core	General area	☐	☐	☐	☐					
SS_G-12	SS_G-12-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-13	SS_G-13-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-14	SS_G-14-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-15-1	SS_G-15-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-16	SS_G-16-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-16	SS_G-16-CR1A	Core	General area	☐	☐	☐	☐					
SS_G-16	SS_G-16-CR1A	QA-field split	General area	☐	☐	☐	☐					

Table A-2. Field Sample Collection Matrix

				Chemistry Grab and Core Samples				Equipment Rinse Blanks				
				Metals, TOC, pH, Percent Solids (8oz WMG) [a]	SVOCs, PCBs (Aroclors), Organo-chlorine Pesticides (16oz WMG) [a]	Dioxins/Furans (8oz WMG) [a]	Grain Size 16oz WMG[a]	Metals (500 mL HDPE) [a]	Dioxins/Furans (1L AG) [a]	Organo-chlorine Pesticides (1L AG) [a]	PCBs (Aroclors) (1L AG) [a]	SVOCs (1L AG) [a]
						4±2°C/ Deep frozen (-20°C)[b]/-10°C[c]		5 mL of 1:1 HNO3;				
Station	Sample ID	Sample Type	Sample Group	4±2°C	4±2°C		4±2°C	4±2°C	4±2°C	4±2°C	4±2°C	4±2°C
SS_G-16	SS_G-16-CR1B	Core	General area	☐	☐	☐	☐					
SS_G-17	SS_G-17-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-18	SS_G-18-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-19	SS_G-19-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-20	SS_G-20-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-20	SS_G-20-CR1A	Core	General area	☐	☐	☐	☐					
SS_G-20	SS_G-20-CR1B	Core	General area	☐	☐	☐	☐					
SS_G-21	SS_G-21-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-22	SS_G-22-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-23	SS_G-23-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-24	SS_G-24-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-25	SS_G-25-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-26	SS_G-26-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-26	SS_G-26-CR1A	Core	General area	☐	☐	☐	☐					
SS_G-26	SS_G-26-CR1B	Core	General area	☐	☐	☐	☐					
SS_G-27	SS_G-27-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-28	SS_G-28-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-29	SS_G-29-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-30	SS_G-30-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-31	SS_G-31-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-32	SS_G-32-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_G-33	SS_G-33-SS1	Surface soil	General area	☐	☐	☐	☐					
SS_H-01	SS_H-01-SS1	Surface soil	HHRA	☐	☐	☐	☐					
SS_H-03	SS_H-03-SS1	Surface soil	HHRA	☐	☐	☐	☐					
SS_H-03	SS_H-03-CR1A	Core	HHRA	☐	☐	☐	☐					
SS_H-03	SS_H-03-CR1B	Core	HHRA	☐	☐	☐	☐					
SS_H-06	SS_H-06-SS1	Surface soil	HHRA	☐	☐	☐	☐					

Table A-2. Field Sample Collection Matrix

				Chemistry Grab and Core Samples				Equipment Rinse Blanks				
				Metals, TOC, pH, Percent Solids (8oz WMG) [a]	SVOCs, PCBs (Aroclors), Organo-chlorine Pesticides (16oz WMG) [a]	Dioxins/Furans (8oz WMG) [a]	Grain Size 16oz WMG[a]	Metals (500 mL HDPE) [a]	Dioxins/Furans (1L AG) [a]	Organo-chlorine Pesticides (1L AG) [a]	PCBs (Aroclors) (1L AG) [a]	SVOCs (1L AG) [a]
Station	Sample ID	Sample Type	Sample Group	4±2°C	4±2°C	4±2°C/ Deep frozen (-20°C)[b]/-10°C[c]	4±2°C	5 mL of 1:1 HNO3; 4±2°C	4±2°C	4±2°C	4±2°C	4±2°C
SS_H-07	SS_H-07-SS1	Surface soil	HHRA	☐	☐	☐	☐					
SS_H-10	SS_H-10-SS1	Surface soil	HHRA	☐	☐	☐	☐					
SS_H-13	SS_H-13-SS1	Surface soil	HHRA	☐	☐	☐	☐					
SS_H-13	SS_H-13-SS1	QA-field split	HHRA	☐	☐	☐	☐					
SS_H-16	SS_H-16-SS1	Surface soil	HHRA	☐	☐	☐	☐					
SS_H-18	SS_H-18-SS1	Surface soil	HHRA	☐	☐	☐	☐					
SS_H-19	SS_H-19-SS1	Surface soil	HHRA	☐	☐	☐	☐					
SS_H-22	SS_H-22-SS1	Surface soil	HHRA	☐	☐	☐	☐					
SS_H-22	SS_H-22-SS1	PE sample	HHRA	☐	☐	☐	☐					
			Total	58	58	58	58	3	3	3	3	3

Notes:

AG = amber jar
HDPE = high density polyethylene
HHRA = human health risk assessment
NA = not applicable
PCB = polychlorinated biphenyls
PE = performance evaluation
QA = quality assurance
SVOC = semivolatile organic compound
TOC = total organic carbon
WMG = wide mouth glass

Station ID:

CR = sediment cores
SS = soil surface samples
Equipment blanks ending in C = Lexan cores
Equipment blanks ending in S = stainless steel bowls and spoons
Equipment blanks ending in R = rinsate
Beige highlight = extra sample volume to include matrix spike/matrix spike duplicate analysis
Blue highlight = equipment rinse blanks
Green highlight = chemistry grab and core samples
Purple highlight = field replicate

[a] The size and number of containers may be modified by the analytical laboratory.
[b] Samples will be shipped to the laboratory on ice at 4±2°C. Once received at the laboratory samples will be stored at -20°C.
[c] Extracts will be stored at -10°C.

Table A-3. Station Coordinates, Sample Type, Sampling Interval, and Corresponding Analysis

Station Number	Sample Type	Sampling Interval [a]	Analysis	Coordinates [b,c] X	Coordinates [b,c] Y
General Area Stations					
SS_G-01	Surface sediment	0–30 cm	All COPCs	333083.9503	279316.2944
SS_G-02	Surface sediment	0–30 cm	All COPCs	332532.8484	278998.1156
SS_G-03	Surface sediment	0–30 cm	All COPCs	332808.3994	279157.2050
SS_G-04	Surface sediment	0–30 cm	All COPCs	332440.9981	278839.0262
SS_G-05	Surface sediment	0–30 cm	All COPCs	332716.5491	278998.1156
SS_G-05	Subsurface sediment (core)	30–61 cm	All COPCs	332716.5491	278998.1156
SS_G-05	Subsurface sediment (core)	61–91 cm	All COPCs	332716.5491	278998.1156
SS_G-06	Surface sediment	0–30 cm	All COPCs	332992.1000	279157.2050
SS_G-07	Surface sediment	0–30 cm	All COPCs	332518.7560	278725.1877
SS_G-08	Surface sediment	0–30 cm	All COPCs	332676.7860	278799.0920
SS_G-09	Surface sediment	0–30 cm	All COPCs	332952.3370	278958.1814
SS_G-10	Surface sediment	0–30 cm	All COPCs	332599.6796	278616.3496
SS_G-10	Subsurface sediment (core)	30–61 cm	All COPCs	332599.6796	278616.3496
SS_G-10	Subsurface sediment (core)	61–91 cm	All COPCs	332599.6796	278616.3496
SS_G-12	Surface sediment	0–30 cm	All COPCs	332690.6998	278327.7043
SS_G-13	Surface sediment	0–30 cm	All COPCs	332782.5501	278486.7937
SS_G-14	Surface sediment	0–30 cm	All COPCs	332915.1051	278661.6932
SS_G-15	Surface sediment	0–30 cm	All COPCs	333081.6265	278693.9832
SS_G-16	Surface sediment	0–30 cm	All COPCs	332906.6760	278403.4221
SS_G-16	Subsurface sediment (core)	30–61 cm	All COPCs	332906.6760	278403.4221
SS_G-16	Subsurface sediment (core)	61–91 cm	All COPCs	332906.6760	278403.4221
SS_G-17	Surface sediment	0–30 cm	All COPCs	333006.9554	278502.6037
SS_G-18	Surface sediment	0–30 cm	All COPCs	333282.5063	278661.6932
SS_G-19	Surface sediment	0–30 cm	All COPCs	333008.2350	278346.8761
SS_G-20	Surface sediment	0–30 cm	All COPCs	333155.2478	278496.2827
SS_G-20	Subsurface sediment (core)	30–61 cm	All COPCs	333155.2478	278496.2827
SS_G-20	Subsurface sediment (core)	61–91 cm	All COPCs	333155.2478	278496.2827
SS_G-21	Surface sediment	0–30 cm	All COPCs	333123.9901	278272.2876
SS_G-22	Surface sediment	0–30 cm	All COPCs	333289.4695	278384.6807
SS_G-23	Surface sediment	0–30 cm	All COPCs	333473.9095	278468.5960
SS_G-24	Surface sediment	0–30 cm	All COPCs	333032.1397	278113.1982
SS_G-25	Surface sediment	0–30 cm	All COPCs	333307.6907	278272.2876

Table A-3. Station Coordinates, Sample Type, Sampling Interval, and Corresponding Analysis

Station Number	Sample Type	Sampling Interval [a]	Analysis	Coordinates [b,c] X	Coordinates [b,c] Y
SS_G-26	Surface sediment	0–30 cm	All COPCs	333468.2713	278373.2611
SS_G-26	Subsurface sediment (core)	30–61 cm	All COPCs	333468.2713	278373.2611
SS_G-26	Subsurface sediment (core)	61–91 cm	All COPCs	333468.2713	278373.2611
SS_G-27	Surface sediment	0–30 cm	All COPCs	333048.2123	277894.8614
SS_G-28	Surface sediment	0–30 cm	All COPCs	333123.9901	277954.1088
SS_G-29	Surface sediment	0–30 cm	All COPCs	333622.0616	278309.2555
SS_G-30	Surface sediment	0–30 cm	All COPCs	333651.9719	278373.2611
SS_G-31	Surface sediment	0–30 cm	All COPCs	333538.0000	277183.0000
SS_G-32	Surface sediment	0–30 cm	All COPCs	333588.1601	277315.6201
SS_G-33	Surface sediment	0–30 cm	All COPCs	334219.1480	276588.1845
Human Health Exposure Stations					
SS_H-01	Surface sediment	0–30 cm	All COPCs	333301.8074	279153.2880
SS_H-03	Surface sediment	0–30 cm	All COPCs	333122.5636	278936.8699
SS_H-03	Subsurface sediment (core)	30–61 cm	All COPCs	333122.5636	278936.8699
SS_H-03	Subsurface sediment (core)	61–91 cm	All COPCs	333122.5636	278936.8699
SS_H-06	Surface sediment	0–30 cm	All COPCs	333301.8074	278970.3746
SS_H-07	Surface sediment	0–30 cm	All COPCs	332964.1560	278845.4132
SS_H-10	Surface sediment	0–30 cm	All COPCs	333069.7611	278845.4132
SS_H-13	Surface sediment	0–30 cm	All COPCs	333175.3662	278845.4132
SS_H-16	Surface sediment	0–30 cm	All COPCs	333317.1536	278891.4299
SS_H-18	Surface sediment	0–30 cm	All COPCs	332911.3534	278753.9565
SS_H-19	Surface sediment	0–30 cm	All COPCs	333228.1687	278753.9565
SS_H-22	Surface sediment	0–30 cm	All COPCs	333333.7738	278753.9565

Notes:

COPC = chemical of potential concern

[a] 0–30 cm = 0–12 in.; 30–61 cm = 12–24 in.; 61–91 cm = 24–36 in.

[b] NAD 1983; State Plane Rhode Island

[c] Coordinates provided correspond to proposed station locations represented on Figure 1. Coordinates for actual station locations will be collected in the field.

Table A-4. Number of Locations Sampled [a]

Sample Group	Sampling Method and Depth	Number of Locations	Sample Locations	Analytes	Study Elements
Surface soil, general concern, all COPCs	Surface 30 cm (0–12 in.)	32	Selected locations in the Oxbow Area	All COPCs, pH, organic carbon, grain size, and percent solids	General concern
Surface soil, human concern, all COPCs	Surface 30 cm (0–12 in.)	10	Selected locations in the Oxbow Area	All COPCs, pH, organic carbon, grain size, and percent solids	Human health exposure concern
Vertical profile, general concern, all COPCs	Grab sampler, 30–61 cm (12–24 in.) and 61–91 cm (24–36 in.)	5	Selected locations in the Oxbow Area	All COPCs, pH, organic carbon, grain size, and percent solids	General concern
Vertical profile, human concern, all COPCs	Grab sampler, 30–61 cm (12–24 in.) and 61–91 cm (24–36 in.)	1	Selected locations in the Oxbow Area	All COPCs, pH, organic carbon, grain size, and percent solids	Human health exposure concern

Notes:
COPC = constituent of potential concern

[a] Numbers do not include field quality control samples, and cores, which generate more than one "sample," are counted only once.

ATTACHMENT A1

HEALTH AND SAFETY PLAN

SAMPLING AND ANALYSIS PLAN: SUPPLEMENTAL INVESTIGATION OF THE LYMAN MILL REACH SEDIMENT AND FLOOD PLAIN SOILS

ATTACHMENT A1
SITE-SPECIFIC HEALTH AND SAFETY PLAN

Centredale Manor Restoration Project Superfund Site in North Providence, Rhode Island

Prepared for
Emhart Industries, Inc.
c/o Sullivan & Worcester, LLP
1666 K Street, NW
Washington, DC 20006

Prepared by

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June 21, 2010

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Exhibit 3. Material Safety Data Sheets
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ACRONYMS AND ABBREVIATIONS

CFR	Code of Federal Regulations
CPR	cardiopulmonary resuscitation
CRZ	contamination reduction zone
HAZWOPER	Hazardous Waste Operations and Emergency Response
HSP	health and safety plan
IDLH	immediately dangerous to life and health
IDW	investigation-derived waste
Integral	Integral Consulting Inc.
OSHA	Occupational Safety and Health Administration
PCB	polychlorinated biphenyl
PEL	permissible exposure limit
PFD	personal flotation device
PPE	personal protective equipment
SSO	site safety officer
STEL	short-term exposure limit
VOC	volatile organic compound
USEPA	U.S. Environmental Protection Agency

SITE-SPECIFIC HEALTH AND SAFETY PLAN APPROVAL

This site-specific health and safety plan (HSP) has been reviewed and approved for the Supplemental Investigation of the Lyman Mill Reach Sediment and Flood Plain Soils, Centredale Manor Restoration Project Superfund Site in North Providence, Rhode Island.

Project Manager Date

Corporate Health and Safety Officer Date

SITE-SPECIFIC HEALTH AND SAFETY PLAN ACKNOWLEDGMENT

In the absence of an appropriate subcontractor or consultant health and safety plan (HSP), and with the express approval of the corporate health and safety officer, the subcontractor or consultant may utilize the Integral Consulting Inc. (Integral) site-specific HSP, provided there is written concurrence from the subcontractor that it will directly administer the plan for its employees. The Integral HSP is a minimum standard for the site and will be strictly enforced for all Integral personnel and subcontractors where applicable.

I have reviewed the HSP prepared by Integral, dated June 21, 2010, for the Supplemental Investigation of the Lyman Mill Reach Sediment and Flood Plain Soils, Centredale Manor Restoration Project Superfund Site in North Providence, Rhode Island. I understand the purpose of the plan, and I consent to adhere to its policies, procedures, and guidelines while an employee of Integral or its subcontractors or consultants. I have had an opportunity to ask questions regarding this plan, which have been answered satisfactorily by Integral.

Date	Name (print)	Signature	Company

Date	Name (print)	Signature	Company

1 INTRODUCTION

This site-specific health and safety plan (HSP) provides the general health and safety provisions to protect workers from potential hazards during field activities associated with the supplemental sampling investigation at the Centredale Manor Restoration Project Superfund Site, North Providence, Rhode Island. The field activities include surface and/or subsurface soil sampling using hand and power tools (stainless steel shovel, trowel, hand auger, or electric impact hand corer).

It is the policy of Integral Consulting Inc. (Integral) to provide a safe and healthful work environment that is compliant with applicable regulations. No aspect of the work is more important than protecting the health and safety of all workers.

This site-specific HSP has been prepared to identify potential site hazards to the extent possible based on information available to Integral. Integral cannot guarantee the health or safety of any person entering this site. Because of the potentially hazardous nature of this site and the activity occurring thereon, it is not possible to discover, evaluate, and provide protection for all possible hazards that may be encountered. Strict adherence to the health and safety guidelines set forth herein will reduce, but not eliminate, the potential for injury and illness at this site. The health and safety guidelines in this plan were prepared specifically for this site and should not be used on any other site without prior evaluation by trained health and safety personnel.

A copy of this HSP must be in the custody of the field crew during field activities. All individuals performing fieldwork must read, understand, and comply with this plan before undertaking field activities. Once the information has been read and understood, the individual must sign the Site-Specific Health and Safety Plan Acknowledgment form provided as part of this plan. The signed form will become part of the project file.

This plan may be modified at any time based on the judgment of the Integral site safety officer (SSO) in consultation with the corporate health and safety officer and Integral project manager or designee. Any modification will be presented to the onsite team during a safety briefing and will be recorded in the field logbook.

2 SITE EMERGENCY PROCEDURES

2.1 EMERGENCY CONTACT INFORMATION

Site Emergency Form and Emergency Phone Numbers

Category	Information
Chemicals of Potential Concern	Dioxins/furans, arsenic, PCB Aroclors®, chlordane, and DDx
Minimum Level of Protection	Level D
Site(s) Location Address	Centredale Manor Restoration Project Superfund Site in North Providence, Rhode Island. Oxbow area at the flood plain of Lyman Mill Pond. Near No. 90 Allendale Avenue, North Providence, RI. Coordinates [41° 51' 1.92"N, 71° 28' 57.3"W]

Emergency Phone Numbers

Ambulance	911
Fire	911
Police	911
Poison Control	911 and then 1-800-222-1212, if appropriate

Project-Specific Health and Safety Officers' Phone Numbers

Integral Field Lead and Integral Site Safety Officer (SSO)	Matt Behum	Office: (410) 573-1982 ext. 12 Cell: (951) 640-4758
Integral Corporate Health and Safety Officer	Eron Dodak	Office: (503) 284-5545 ext. 14 Cell: (503) 407-2933
Integral Project Manager	Patrick Gwinn	Office: (207) 874-9000 ext. 206 Cell: (207) 798-9578
Client Contact – Emhart Industries Inc.	Linda Biagioni	Office: (410) 716-2341 Cell:

Reporting Oil And Chemical Spills

National Response Center	1-800-424-8802
State Emergency Response System	(401) 222-3070
EPA Environmental Response Team	(201) 321-6600

Note: In the event of any emergency, contact both the Integral and Emhart Industries project managers.

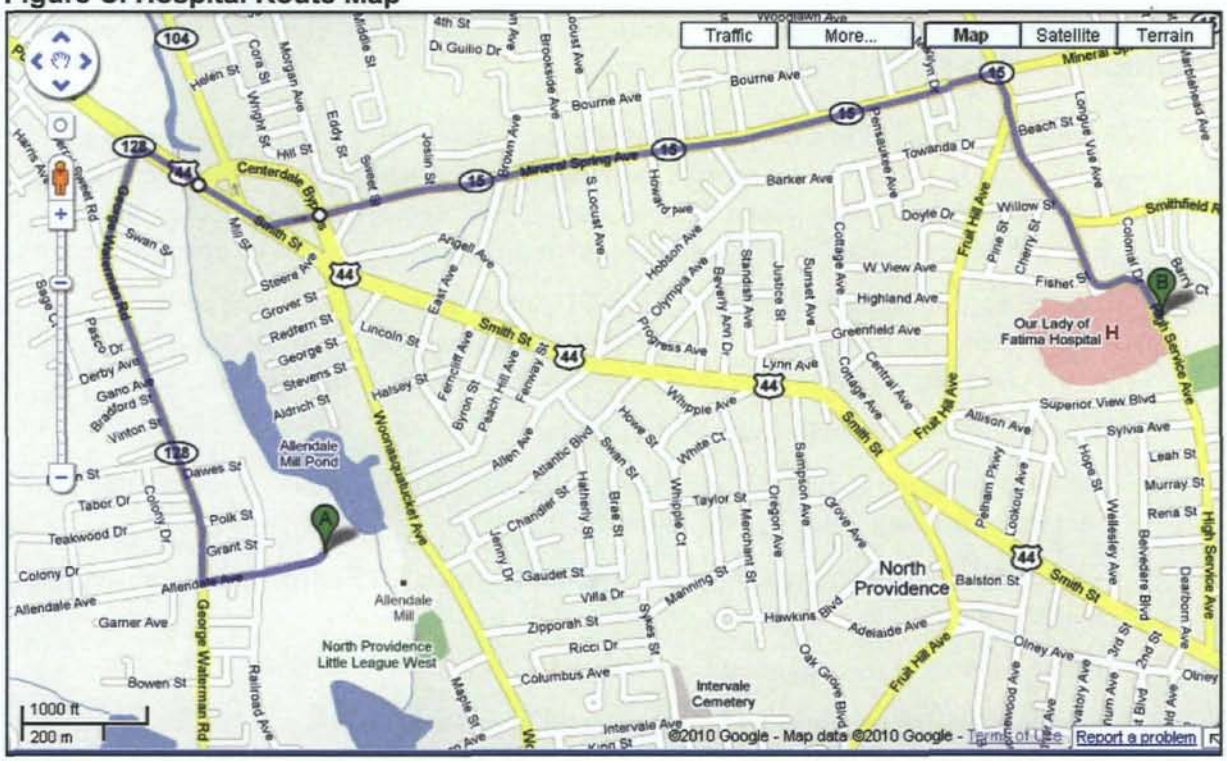
Site Location Map

Site Boat Access

Hospital Information

Category	Information
Hospital Name	Our Lady of Fatima
Address	200 High Service Avenue
City, State	North Providence, RI 02904
Phone	(401) 456-3000
Emergency Phone	(401) 456-3400

Figure C. Hospital Route Map



Driving Directions from Site to Hospital

1. Head **southwest** on **Allendale Ave** toward **Railroad Ave** (go 0.2 mile)
2. Take the 2nd **right** onto **George Waterman Rd/RI-128 N** (go 0.7 mile)
3. Turn **right** at **Putnam Pike/US-44 E**, Continue to follow US-44 E (go 0.2 mile)
4. Turn **left** at **Mineral Spring Ave** (go 1.1 miles)
5. Turn **right** at **Smithfield Rd** (go 322ft)
6. Slight **left** to stay on **Smithfield Rd** (go 0.2 mile)

7. Slight **right** at **High Service Ave** (go 0.2 mile)
8. Arrive at **200 High Service Area** (Total 2.7 miles).

NOTE: The map and traffic directions are provided for planning purposes only. Weather conditions, construction projects, closures, or other events may cause road conditions or directions to differ from the instructions described above. The emergency route should be checked before commencing field work. The maps and hospital route are also provided in Exhibit 1.

2.2 EMERGENCY RESPONSE PROCEDURES

In the event of an emergency, immediate action must be taken by the first person to recognize the event. Use the following steps as a guideline:

1. Survey the situation to ensure that it is safe for you and the victim. Do not endanger your own life. Do not enter an area to rescue someone who has been overcome unless properly equipped and trained. Ensure that all protocols are followed. If applicable, review Material Safety Data Sheets to evaluate response actions for chemical exposures.
2. Call the appropriate emergency number (911) or direct someone else to do this immediately (see the Emergency Contact Information Table). Explain the physical injury, chemical exposure, fire, or release and the location of the incident.
3. Have someone retrieve the nearest first aid kit.
4. Decontaminate the victim without delaying life-saving procedures (see Section 10.2 for emergency decontamination procedures).
5. Administer first aid and cardiopulmonary resuscitation (CPR), if properly trained, until emergency responders arrive.
6. Notify the project manager and the field lead.
7. Complete the appropriate incident investigation reports.

2.3 FIRST AID AND CPR GUIDELINES

Personnel qualified and current in basic first aid and/or CPR procedures may perform those procedures as necessary. Personnel qualified and current in basic first aid and/or CPR are protected under Good Samaritan policies as long as they only perform the basic tasks that they were taught. Do not perform first aid and/or CPR tasks if you have not been trained in first aid and/or CPR.

2.4 INJURY MANAGEMENT/INCIDENT NOTIFICATION

Observe the following injury management/incident notification procedures and practices:

2.4.1 Injury Management

1. Once a personal injury incident is discovered, the first action will be to ensure that the injured party receives appropriate medical attention.
2. If it is safe to do so, the nearest workers will immediately assist a person who shows signs of medical distress or who is involved in an accident.
3. Render first aid and call 911 or the appropriate emergency number as soon as possible.
4. Escort the injured person to the occupational clinic or hospital or arrange for an ambulance.
5. Proceed immediately to Notification Requirements, below.

2.4.2 Notification Requirements

Directly after caring for an injured person, the work crew supervisor will be summoned. The work crew supervisor will immediately make contact with the project manager or other designated individual to alert them of the medical emergency and advise them of the following:

- Location of the victim at the work site
- Nature of the emergency
- Whether the victim is conscious
- Specific conditions contributing to the injury, if known

The project manager will contact upper line management, including the corporate health and safety officer. The corporate health and safety officer will facilitate the incident investigation.

All client requirements pertinent to personal injury incident reporting will also be adhered to.

2.5 INCIDENT OTHER THAN PERSONAL INJURY

All incidents including, but not limited to, fire, explosion, property damage, or environmental release will be responded to in accordance with the site-specific Health and Safety Plan. In general, this includes securing the site appropriate to the incident, turning control over to the emergency responders, or securing the site and summoning appropriate remedial personnel or equipment. Integral will immediately notify the client of any major incident, fire, equipment or

property damage, or environmental incident with a preliminary report. A full report will be provided within 72 hours.

2.6 NEAR-MISS REPORTING

All near-miss incidents (those that could have reasonably lead to an injury, environmental release, or other incident) must also be reported to the field lead and/or project manager immediately so they can take action to ensure that such conditions that lead to the near-miss incident can be readily corrected in order to prevent future occurrences.

2.7 SPILLS AND RELEASES OF HAZARDOUS MATERIALS

When required, notify the National Response Center and local state agencies. The following information should be provided to the National Response Center:

- Name and telephone number
- Name and address of facility
- Time and type of incident
- Name and quantity of materials involved, if known
- Extent of injuries
- Possible hazards to human health or the environment outside of the facility

The emergency telephone number for the National Response Center is 1-800-424-8802. If hazardous waste has been released or produced through control of the incident, ensure that:

- Waste is collected and contained
- Containers of waste are removed or isolated from the immediate site of the emergency
- Treatment or storage of the recovered waste, contaminated soil or surface water, or any other material that results from the incident or its control is provided
- No waste that is incompatible with released material is treated or stored in the facility until cleanup procedures are completed
- Ensure that all emergency equipment used is decontaminated, recharged, and fit for its intended use before operations are resumed.

A copy of this HSP must be in the possession of the field crew at all times during field work.

3 ROLES AND RESPONSIBILITIES AND PROJECT BACKGROUND

All personnel and visitors on this site must comply with the requirements of this HSP. The specific responsibilities and authority of management, safety and health, and other personnel on this site are detailed in the following paragraphs.

3.1 REGULATORY FRAMEWORK

The State of Rhode Island operates under federal Occupational Safety and Health Administration (OSHA) regulations, which cover all occupational safety and health concerns for the state (Exhibit 2). This site-specific HSP follows the federal OSHA Hazardous Waste Operations and Emergency Response (HAZWOPER; 29 Code of Federal Regulations [CFR] 1910.120) regulations.

3.2 PROJECT MANAGER AND OTHER KEY CONTACTS

	Name (and Affiliation)	Work (and/or Cell) Telephone	Home Telephone
Project manager	Patrick Gwinn (Integral)	(207)874-9000 ext 206 (work) (207) 798-9578 (cell)	(207) 926-3099
Client contact	Linda Biagioni (Emhart)	(410) 716-3208 (work)	

3.2.1 Site Safety Officer/Site Supervisor

The SSO and site supervisor have full responsibility and authority to develop and implement this HSP and to verify compliance. They report to the project manager and are onsite or readily accessible to the site during all work operations. They have the authority to halt site work if unsafe conditions are detected and are responsible for assessing site conditions and directing and controlling emergency response activities. The specific responsibilities of the SSO are as follows:

- Managing the safety and health functions on this site
- Serving as the site's point of contact for safety and health matters
- Assessing site conditions for unsafe acts and conditions and providing corrective action
- Ensuring that all Integral employees and subcontractors know and follow the HSP

- Ensuring that daily work schedules and tasks are appropriate for the required levels of effort and weather conditions
- Confirming local emergency response phone numbers and locations
- Conducting and documenting the initial and daily health and safety briefings
- Evaluating and modifying the level of protective apparel and safety equipment, as necessary, based on site conditions
- Ensuring that the field team observes all necessary decontamination procedures
- Conducting inspections on a weekly basis to determine the effectiveness of the HSP.

If the site supervisor determines that site conditions are unsafe, he or she has the authority to suspend field operations until the problem is corrected. The site supervisor can modify HSP procedures in consultation with the SSO. Any changes must be documented in the field logbook and field staff must be immediately informed of the change. The project manager and Integral's corporate health and safety officer must be notified of any changes to the HSP within 12 hours.

3.2.2 Site Workers

Site workers are responsible for complying with this HSP, using the proper personal protective equipment (PPE), reporting unsafe acts and conditions, and following the work and safety and health instructions of the project manager and SSO. All site workers have the authority and are encouraged to suspend field operations and leave the environment if they feel conditions have become unsafe.

3.2.3 Buddy System

The buddy system will be used at the site at all times. The buddy system is a system of organizing employees into field teams in such a manner that each employee of the field team is designated to be observed by at least one other employee in the field team. The purpose of the buddy system is to provide rapid assistance to employees in the event of an emergency.

3.3 SITE BACKGROUND AND TASK DESCRIPTION

The Centredale Manor Restoration Project Superfund Site is located at 2072 and 2074 Smith Street in North Providence, Rhode Island. The entire site consists of approximately 9.04 acres and encompasses the surface water, sediment, and floodplain soil areas of the Woonasquatucket River, extending southerly from Route 44 to the Allendale Dam and further below to the Lyman Mill Dam. Prior to 1936, Centredale Worsted Mills, a woolens mill, occupied the site. In 1943, Atlantic Chemical Company began operating on a portion of the site. In 1953, Atlantic Chemical Company changed its name to Metro-Atlantic, Inc. and operated on

a portion of the site until the early 1968. From 1952 until the early 1970s, New England Container Company, Inc. operated a drum reconditioning facility on a portion of the site. In 1972, a fire destroyed most of the structures at the site. In 1976 and 1983, respectively, Brook Village Associates Limited Partnership and Centerdale Manor Associates each constructed an apartment building, providing affordable housing to the elderly on the properties located at 2072 and 2074 Smith Street.

Site Information

Site name	Centredale Manor Restoration Project Superfund Site
Location	North Providence, Rhode Island
Owners/tenants	To be determined
Hazardous waste site?	Yes
Industrial waste site?	No
Topography	Personnel will be working on the flood plain at Lyman Mill Pond. The area is relatively flat and composed of Adrian muck and Podunk fine sandy loam.
Site access	The site will be accessed via vehicle to the nearest parking area and then on foot to the sampling site. Some areas may be accessed by boat.
Site activity	Surface and/or subsurface soil collection (surface grabs/hand cores) sampling using hand and power tools.
Nearest drinking water/sanitary facilities	The sampling team will bring drinking water with them, which will be left outside the exclusion and contaminant reduction zones (i.e., in the vehicle). Sanitary facilities will be available at nearby gas stations or similar facilities.
Nearest telephone	Matt Behum (Integral) Cell Phone: (951) 640-4758
Task description	Surface and subsurface soil samples will be collected from station locations within the oxbow flood plain area at the Lyman Mill Pond portion of the Centredale Manor Restoration Project Superfund Site in North Providence, Rhode Island. A stainless steel hand auger, shovel, or trowel will be used to collect surface soil samples. Subsurface soil samples will be collected by using a stainless steel hand corer down to 40 inches.

3.4 DEFINITIONS

Contamination reduction zone (CRZ):	Area between the exclusion and support zones that provides a transition between contaminated and clean zones
Exclusion zone:	Any area of the site where hazardous substances are present, or are reasonably suspected to be present, and pose an exposure hazard to personnel
HAZWOPER:	Hazardous Waste Operations and Emergency Response standard, as described in 29 CFR Part 1910.120

OSHA:	Occupational Safety and Health Administration
Support zone:	Any area of the site, so designated, that is outside the exclusion and contamination reduction zones

4 CHEMICAL HAZARD EVALUATION

Below is a summary of chemicals detected in the soils of the oxbow flood plain portion of the site, which is where soil sampling will be conducted (MACTEC 2006).

Chemical	Maximum Detected Concentration
PCBs (ng/g dry wt)	
Total PCBs Aroclor®	3,583
TCDD/TCDF (pg/g dry wt)	
2,3,7,8-TCDD	3,140
2,3,7,8-TCDF	19.3
2,3,7,8-TEQ	3,160
Pesticides (ng/g dry wt)	
4,4'-DDD	26.58
4,4'-DDE	42.36
4,4'-DDT	2.76
a-BHC	0.22
a-chlordane	30.08
Aldrin	0.14
b-BHC	0.13
d-BHC	0.16
Dieldrin	0.16
Endosulfan I	0.17
Endosulfan II	1.16
Endosulfan sulfate	0.19
Endrin	0.16
Endrin aldehyde	0.21
Endrin ketone	0.16
g-chlordane	2.1
Heptachlor	0.15
Heptachlor epoxide	0.14
Lindane	0.14
Methoxychlor	0.22
Technical Chlordane	16.2

Chemical	Maximum Detected Concentration
Metals (µg/g dry wt)	
Antimony	7.01
Arsenic	12.8
Barium	514
Beryllium	7.9
Cadmium	8.25
Chromium	104
Cobalt	22
Copper	357
Lead	1,835
Manganese	859
Molybdenum	17.3
Nickel	32.4
Selenium	2.22
Silver	11.1
Thallium	1.04
Vanadium	71.1
Zinc	1,867

Representative chemicals of concern, applicable chemical properties, and potential exposure routes are presented in the following table. In addition, the table lists the properties of other chemicals that may be used at the site during the course of the supplemental soil investigation (i.e., gasoline). The table also lists the chemical properties and OSHA's permissible exposure limit (PEL), short-term exposure limit (STEL), and immediately dangerous to life and health (IDLH) level. Some chemicals used during equipment decontamination or sample preservation may volatilize and the potential exists that these chemicals may enter the field crew's breathing zone and be inhaled. The decontamination chemicals will be handled in open air and at sufficient distance from the breathing zone to minimize the potential inhalation hazard.

4.1 CHEMICAL PROPERTIES

Chemical of Concern	Concentration (Site Maximum)	Medium	OSHA PEL (mg/m^3)	OSHA STEL (mg/m^3)	OSHA IDLH (mg/m^3)	Odor Threshold (ppm)	Carcinogen or Other Hazard
PCDD/PCDF	4.27 ng/kg	Soil	--	--	--	--	Ca
PCDD/PCDF (2,3,7,8-TCDD TEQ)	4,291 ng/kg	Soil	--	--	--	--	Ca
Total polychlorinated biphenyls (PCBs), Aroclor 1254	3.58 mg/kg	Soil	0.5 mg/m^3; NIOSH 0.001	0.001	5	NA	Ca
Chlordane	30.08 ng/g	Soil	0.5	--	100	NA	Ca
4,4-DDD	26.58 ng/g	Soil	--	--	--	NA	
4,4-DDE	42.36 ng/g	Soil	--	--	--	NA	
4,4-DDT	2.76 ng/g	Soil	1; NIOSH 0.5	--	500	NA	Ca
Arsenic	12.8 mg/kg	Soil	0.01; NIOSH	0.002 (NIOSH ceiling)	5	Odorless	Ca
Gasoline[a]	Product	Product	--	1480	--	--	Flammable liquid, Ca
Liquinox/Alconox[a]	Product	Product	--	--	--	--	--

Source: Batelle 2005

Notes: -- = none established
Ca = carcinogen
IDLH = immediately dangerous to life and health
mg/kg = milligrams per kilogram
mg/m^3 = milligrams per cubic meter
NA = not available
ng/g = nanograms per gram
ng/kg = nanograms per kilogram
NIOSH = National Institute for Occupational Safety and Health
PEL = permissible exposure limit
ppm = parts per million
STEL = short-term exposure limit

[a] Material Safety Data Sheets can be found in Exhibit 3.

4.1.1 Potential Chemical Exposure Routes at Site

Potential Chemical Exposure Route	Likely	Possible	Unlikely
Inhalation		X[a]	X[b]
Ingestion			X
Skin absorption		X	
Skin contact		X	
Eye contact		X	
If likely, describe safety precautions			

[a] Decontamination chemicals, preservatives, and gasoline. Carbon monoxide in exhaust fumes of power generator is a toxic gas, but, being colorless, odorless, tasteless, and non-irritating, it is very difficult for people to detect. Field personnel will stand upwind when using a power generator.
[b] Soil

Chemical Characteristics	Yes	No
Corrosive		X
Ignitable	X[a]	X[b]
Reactive	X[a]	X[b]
Volatile	X[a]	X[b]
Radioactive		X
Explosive		X
Biological agent		X
Particulates or fibers		X

Notes:
Gasoline is volatile and field personnel will stand upwind when using power generator.
These chemicals will not be used unless area is well ventilated.
Keep chemicals away from ignition sources at all times.

[a]Gasoline from power generator
[b]Soil

5 PHYSICAL HAZARD EVALUATION

5.1 SUMMARY OF ACTIVITIES AND POTENTIAL HAZARDS

Activity	Potential Hazard
Surface and subsurface soil sampling	Uneven terrain/tripping, heat stress, noise, falling objects, heavy equipment, material handling, adverse weather, plant/animal hazards, drowning
Sample and investigation-derived waste (IDW) handling	Uneven terrain/tripping, heat stress, noise, material handling, adverse weather, plant/animal hazards

The following table presents possible physical hazards that are expected to be present during field activities.

Possible Physical Hazard	Yes	No	Proposed Safety Procedure
Heavy equipment	X		Stay back from operating equipment (i.e., impact hammer); wear reflective safety vests, ear plugs, and safety goggles; coordinate and maintain eye contact with equipment operator.
Material handling	X		Lift properly; seek assistance if necessary; do not overfill coolers or boxes.
Drowning	X		Wear a personal flotation device (PFD) at all times while on boat. Participate in boat safety meeting.
Adverse weather	X		Seek shelter during electrical storms; work in adverse weather conditions only with proper training and equipment.
Plant/animal hazards	X		Presence of poison ivy. Red fox, raccoon, and skunk. Use insect repellent if mosquitoes are persistent.
Uneven terrain/tripping	X		Snags and fallen dead wood are common. Area with several flooded pools. Wear properly fitting boots, hip waders, or chest waders. If working near river, wear a PFD.
Noise	X		Wear ear protection when working around heavy equipment and other noise sources.
Heat stress	X		Follow heat stress information (Exhibit 4); potential for heat stress will depend on season.
Falling objects		X	Wear hard hats near overhead hazards (i.e., winch).
Drill rigs		X	Avoid all pinch points; do not operate or stand near during electrical storms; stay a safe distance (25 ft) from power lines, level drill rig.
Other Traffic		X	A traffic control plan is not required since the site is located on a flood plain away from roads.

Notes: If confined space entry is required, personnel must first obtain a confined space entry permit. Integral personnel are not trained or authorized to enter confined spaces under any circumstances.

6 PERSONAL PROTECTIVE EQUIPMENT AND SAFETY EQUIPMENT

The following sections address PPE and safety equipment required for completing the field activities.

6.1 LEVEL OF PROTECTION REQUIRED FOR SITE ACTIVITIES

Site Activity	Level of Protection	
	Initial	Contingency[a]
Surface and subsurface soil sampling near river or on boat	MD*	Leave site
Surface and subsurface soil sampling in flood plain	MD	Leave site
Sample handling	D	Leave site
Decontamination of sampling equipment	D	Leave site

[a] Based on unexpected change in site conditions

6.2 LEVELS OF PROTECTION FOR PERSONAL PROTECTIVE EQUIPMENT

Protection Level	Required	Personal Protective Equipment
Level D	X	Long pants and shirt or work coveralls, steel toed boots, latex or nitrile gloves, and eye protection. Hard hat and hearing protection as needed.
Level MD	X	Same as Level D with modification (M) of addition of brightly colored traffic safety vest. When sampling in flooded areas wear hip or chest waders.
Level MD*	X	Same as Level MD with addition of PFD.

6.2.1 Respirator Information

Is there potential for a respirator to be donned during fieldwork? Yes _____ No X

6.3 SAFETY EQUIPMENT

First Aid Kit is mandatory and must include adhesive band-aids, gauze, tape, gloves, CPR shield, and triangle bandage. **Check items required for the site:**

- ☐ Aerosol meter
- ☐ Air sampling pumps
- ☒ Cellular phone
- ☐ Cool vests
- ☐ Detector tubes and pump (e.g., Draeger®)
- ☒ Drinking water
- ☒ Emergency blanket
- ☒ Eyewash solution
- ☒ Fire extinguisher
- ☐ H_2S meter
- ☐ Respirator, cartridges, and supplies
- ☒ Global positioning system
- ☒ Insect repellent
- ☐ LEL/O_2 meter
- ☒ Personal flotation device
- ☐ Photoionization detector
- ☐ Radiation meter
- ☐ Radio sets
- ☐ Stopwatch for monitoring heart rate
- ☒ Sunscreen
- ☐ Survival kit
- ☐ Thermoscan® thermometer (or equivalent) for heat stress monitoring
- ☐ Windsock
- ☐ Other: _____

7 AIR MONITORING

Air monitoring will be conducted when entering previously uncharacterized sites, when working in the vicinity of chemicals or spills, when opening containers and well casings, and prior to opening and entering confined spaces. Air monitoring must be conducted to identify potentially hazardous environments and determine reference or background concentrations. Air monitoring can be used to augment judgment in defining exclusion zones. Air monitoring may also be conducted to evaluate the concentration of chemicals in samples.

The primary constituents of concern at the site are PCBs and dioxins. There is a chance for dust to become airborne during soil sampling activities. However, sampling will be done by advancing a hand auger into the soil. Drilling will not take place. The majority of the subsurface soils are expected to be moist or wet, and the volume extracted from the ground during sampling is limited, which will minimize the potential for dust generation. Exposure to airborne particulates will be assessed by visual observation. Activities producing dust will either be curtailed or modified (e.g., dust suppression with potable water sprays).

If the generation of visible dust particles during sample collection cannot be curtailed, work must stop and workers must leave the area. The field coordinator must contact the project manager and wait for further instructions. The project manager will consult with the corporate health and safety officer and determine appropriate actions for field sampling crew. The project manager will then contact the field coordinator and give instructions on how to proceed with field work

8 HEALTH AND SAFETY TRAINING AND EMERGENCY PLANNING

8.1 HEALTH AND SAFETY TRAINING AND MEDICAL MONITORING

State and federal laws establish training requirements for workers at uncontrolled hazardous waste sites (including areas where accumulations of hazardous waste create a threat to the health and safety of an individual, the environment, or both). Integral and subcontractor personnel are required to complete the following training requirements prior to working at the site.

OSHA requires medical monitoring for personnel potentially exposed to chemical hazards in concentrations in excess of the PEL for more than 30 days per year and for personnel who must use respiratory protection for more than 30 days per year. Integral requires medical monitoring for all employees potentially exposed to chemical hazards.

Training and Medical Requirements[a]

Personnel	No Training	24-hour	40-hour[b]	Supervisor[c]	First Aid/CPR[d]	Medical Monitoring
Matt Behum			X	X	X	X

[a] Note that non-Integral subcontractors and personnel may have requirements that are more stringent than those listed above. These are minimum training and monitoring requirements to work on this site.
[b] Must have current 8-hour refresher if it has been more than a year since the 40-hour training.
[c] At least one person on site must be OSHA HAZWOPER supervisor trained if this is a hazardous waste site.
[d] At least one member of each team of two or more people onsite must be first aid/CPR trained.

8.2 SITE SAFETY MEETINGS

Site safety meetings must be held before beginning new tasks or when new staff begin work at the site. Site safety meetings should be held at a minimum of once a week and should be held daily on large projects. Tailgate safety meetings should occur every morning during review of the day's work plan, covering specific hazards that may be encountered that day. Additional meetings will be held at any time health and safety concerns are raised by any of the personnel. Attendance and topics covered are to be documented in the field logbook.

8.3 EMERGENCY PLANNING AND TELEPHONE NUMBERS

In case of any emergency affecting the site, all affected personnel must immediately evacuate the work area and report to the SSO at the following predetermined location.

DESIGNATED ASSEMBLY LOCATION: Field vehicle

In case of injury, field personnel should take precautions to protect the victim from further harm and notify local or facility emergency services. In remote areas, it will be necessary to have first aid-trained personnel on the field team. The victim may require decontamination prior to treatment—requirements will vary based on site conditions.

Emergency medical care will be provided by:

- [X] Local emergency medical provider (i.e., fire department)
- [ ] Facility emergency medical provider
- [ ] First aid-trained field staff (for remote areas only)

Emergency Telephone Numbers

Local Resources	Name	Telephone	Notified Prior to Work (Yes/No)?
Fire	North Providence Fire Department	911	No
Police	North Providence Police Department	911	No
Ambulance	North Providence	911	No
Hospital	Our Lady of Fatima Hospital Emergency Department	(401) 456-3400	No
Site phone	Field cell phone	(951) 640-4758	NA
Directions to the hospital:	Consult map and driving instructions in Section 2.1 and Exhibit 1. The hospital is located at 200 High Service Avenue, Providence, RI.		

Corporate Resources	Name	Work/Cell Telephone	Home Telephone
Integral Corporate Health and Safety Officer	Eron Dodak	Work: (503) 284-5545 Cell: (503) 407-2933	NA
Medical consultant	Dr. Calvin Jones (HealthForce Partners)	Work: (425) 806-5700	NA

In case of serious injuries, death, or other emergency, the Integral corporate health and safety officer must be notified immediately. To contact the Integral corporate health and safety officer (or delegate), try calling the phone numbers listed above.

9 WORK ZONES

Exclusion zone: The area immediately around the sampling activities. The sampling sites will most likely be on areas where normal foot traffic does not occur (e.g. wetland area). However, where transient foot traffic is possible, the sampling site will be marked with orange traffic cones or caution tape (a minimum 15 ft radius around the sampling location).

Contamination reduction zone (CRZ): A CRZ will be located inside the exclusion zone, marked off at the edge of the exclusion zone.

Support zone: The area outside of the exclusion zone and CRZ.

Controls to be used to prevent entry by unauthorized persons: Sampling staff will police the area for people approaching the exclusion zone. All people will be instructed to remain outside of the marked area.

10 DECONTAMINATION AND PERSONAL HYGIENE

10.1 EQUIPMENT DECONTAMINATION

After sampling is completed, the exclusion zone will be used as the CRZ for decontamination activities.

To minimize or prevent personal exposure to hazardous materials, all personnel working in the exclusion zone and CRZ will comply with the following decontamination procedures:

- All personnel will wash soil and chemicals from their raingear or remove Tyvek® coveralls before leaving the exclusion zone. All gloves, Tyvek, rain gear, and rubber boots will be removed prior to entering the field vehicle.

Decontamination equipment required at the site includes the following:

- Buckets or tubs
- Distilled/deionized water
- Site water
- Scrub brushes
- Liquinox® or Alconox® detergent
- Garden sprayer
- Plastic bags
- Foil
- Paper towels
- Garbage bags

All non-disposable components of the sampling equipment (e.g., stainless steel spoons and bowls) that contact the sediment or surface water will be decontaminated using the following steps:

1. Rinse with site water/tap water
2. Wash with Alconox or Liquinox detergent
3. Rinse with site water/tap water
4. Rinse with distilled/deionized water using a garden sprayer (compositing equipment only)
5. Allow to air dry
6. Wrap up compositing equipment in aluminum foil.

If samples are collected that include obvious oily contamination, the sampling equipment used to collect and process them will be decontaminated using a separate decontamination station dedicated to heavily impacted equipment. This equipment will undergo a second decontamination sequence.

10.2 EMERGENCY DECONTAMINATION PROCEDURES FOR PERSONNEL

In case of an emergency, gross decontamination procedures will be speedily implemented, if possible. If a life-threatening injury occurs and the injured person cannot undergo decontamination procedures, the medical facility will be informed that the injured person has not been decontaminated and given information regarding the most probable chemicals of concern.

Decontamination procedures will only be used if practical and if they will not further injure the person. The SSO will make the determination on whether or not to decontaminate the injured person. The following steps will be followed for decontaminating injured personnel:

- Wash the gross contamination from the injured person's PPE (e.g., Tyvek coveralls, work boots, etc.) with a Liquinox or Alconox solution followed by a rinse with tap or deionized/distilled water

Remove PPE if feasible without further injuring the person.

10.3 STUDY-DERIVED WASTES

Any excess phosphate-free, detergent-bearing liquid wastes from decontamination or any sample remaining after processing will be deposited in the vicinity of the collection area. Any dry waste (e.g., contaminated boots, bibs, Tyvek suits, contaminated soils) present at the end of the sampling event will be segregated and containerized (e.g., D.O.T-approved 55-gallon steel drums) and properly disposed of in accordance with applicable regulations by a subcontractor specialized in hazardous waste removal. The subcontractor will be required to have, at a minimum, a drum management service that provides the following:

- Proper waste identification including full analytical capability
- Pick up and disposal of a broad range of hazardous wastes
- Safe and proper transportation
- Environmentally sound treatment and disposal
- Regularly scheduled service visits with manifest and label preparation.

All disposable materials used for sample collection and processing, such as paper towels and gloves, will be placed in heavyweight garbage bags or other appropriate containers. Disposable supplies that do not contain site soil will be removed from the site by sampling personnel and disposed of in a solid waste landfill.

10.4 PERSONAL HYGIENE

The following personal hygiene practices will be used at the site to reduce exposure to chemicals:

- Long hair will be secured away from the face so it does not interfere with any activities.
- All personnel leaving potentially contaminated areas will wash their hands, forearms, and faces in the CRZ prior to entering any clean areas or eating areas.
- Personnel leaving potentially contaminated areas will shower (including washing hair) and change to clean clothing as soon as possible after leaving the site.
- No person will eat, drink, or chew gum or tobacco in the exclusion zone or CRZ. Drink containers and drinking of replacement fluids for heat stress control will be permitted only in support areas. Smoking is prohibited in all areas of the site because of the potential for contaminating samples and for health and safety reasons.

11 VEHICLE SAFETY, SPILL CONTAINMENT, AND SHIPPING INSTRUCTIONS

11.1 VEHICLE SAFETY

Integral's vehicle safety program requires the following:

- All vehicles are to be operated in a safe manner and in compliance with statutory traffic regulations and ordinances
  - Operators are to practice defensive driving and drive in a courteous manner
- Operators are required to have a valid driver's license and liability insurance (per local state laws)
  - Seat belts are to be worn by the driver and all passengers
- No persons are allowed to ride in the back of any trucks or vans, unless equipped with seatbelts
- Vehicles are to be driven in conformance with local speed limits
- Personnel who are impaired by fatigue, illness, alcohol, illegal or prescription drugs, or who are otherwise physically unfit, are not allowed to drive
- Personnel are to avoid using cellular phones or engaging in other distractions while driving
- All Integral-owned field vehicles are to be maintained in a safe and clean condition
- All Integral-owned field vehicles are to be equipped with the following:
  - First-aid kit
  - Fire extinguisher
  - Flares
  - Spare tire and jack
  - Other equipment as required for the project (e.g., tire chains, towing cable, tools, cellular phone or radio)
- Motor vehicle accidents are to be reported to the responsible law enforcement agency, the Integral human resources manager, and the Integral corporate health and safety officer on the same day of occurrence
- Employees who have experienced work-related vehicle accidents or citations may be required to complete a defensive driving program.

11.2 SPILL CONTAINMENT

Detergent used for decontamination will be dispensed from capped containers directly into wash tubs or specific laboratory safety squirt bottles that have been permanently marked with the name of the decontamination chemical and that have screw caps. Gasoline will be carefully poured into the generator's fuel tank from an approved gasoline container. Secondary containment (e.g., large plastic bin) will be used while decontamination chemicals are poured into the squirt bottles and when gasoline is poured into the generator's fuel tank to capture any overfill/spills.

In case of a significant bulk liquid spill, the National Response Center (800-424-8802) will be notified by the SSO or the project manager.

11.3 SHIPPING INFORMATION

Federal laws and international guidelines place restrictions on what materials may be shipped by passenger and cargo aircraft. In addition, 49 CFR regulates labeling, manifesting, and shipment of all packages containing potentially hazardous materials.

A 24-hour emergency response number (on any shipping documents such as a Uniform Hazardous Waste Manifest, Shipper's Declaration of Dangerous Goods, etc.) is required for shipments of all dangerous or hazardous goods. Integral does not have a 24-hour emergency contact number for dangerous or hazardous goods shipment. No dangerous or hazardous goods may be shipped by Integral until an account is set up with a 24-hour emergency response service such as CHEM-TEL. If any hazardous or dangerous goods need to be shipped for a project, they must be shipped directly to the site by the supplier. Any hazardous or dangerous goods that are not used in the course of the field effort must remain at the site.

In the course of this field investigation, the following items will be shipped to and from the site as shown below:

Item	Hazardous Constituent	Quantity	Packaging	How Shipped
Samples	None	Soil solid matrix samples	Coolers	Fed Ex
Other	None			

The samples will be prepared and labeled for shipment in accordance with the sampling and analysis plan developed for the site.

12 TASK-SPECIFIC SAFETY PROCEDURES

12.1 SOIL SAMPLING

Avoid getting soil, gasoline, and decontamination chemicals on your clothes or skin. Exercise care when lifting, assembling, and decontaminating equipment. Get help when lifting heavy sampling equipment or coolers of samples. Always stay clear of the electric impact handheld corer and be aware of its location. Keep in eye contact with the core operator. Keep equipment organized. Keep gasoline away from ignition sources at all times. Always refuel machinery in well ventilated areas and stand upwind while refueling.

Before sampling begins, if area appears dusty, surface soils slated for sampling will be wetted down using a garden sprayer filled with potable or distilled water. If it is necessary to kneel, new 6-mil plastic sheeting will be placed on the ground adjacent to the station so the sampling personnel can kneel on the ground without contacting the soils. If any visible dust is observed during sampling activities, the soils will be wetted down again. Once the sampling is completed, the plastic sheeting and disposable gloves will be disposed of properly.

Wear a PFD when transporting samples and sampling equipment on a boat. Wear rubber boots or waders if collecting samples in shallow water without the use of a boat. Walk slowly and deliberately to make sure the sediment is firm enough to support your weight. If it is not, special shoes such as Mudders™ may be necessary. Always use the buddy system when working near bodies of water.

12.1.1 Small Boat Safety

The stability of a small boat is related directly to the below-water hull shape and to the height of the center of gravity. Therefore, all weight, including the crew, should be near the centerline and as low in the boat as possible. The following steps will help prevent boat injuries.

- Participate in the health and safety meeting and listen to the boat operator instructions.
- Keep your shoulders inside the gunnels. Center yourself in the boat keeping your center of gravity low in the boat.
- Maintain three points of contact. Keep both hands and at least one foot, or both feet and at least one hand, in contact with the boat at all times.
- Load the boat properly. Check the load capacity plate and keep within its limits. Keep gear weight centered both from side to side and bow to stern.
- Integral staff are not allowed to operate small boats equipped with outboards.
- Use the buddy system when on board.

12.2 SAMPLE AND INVESTIGATION-DERIVED WASTE HANDLING

Get help when lifting coolers full of samples and lift with your legs instead of your back. Always get help moving IDW containers and make sure the lids are securely fastened. If possible, use a hand truck or drum dolly when moving IDW containers. Wear nitrile gloves under thick leather gloves since the drum lids may have sharp edges.

13 REFERENCES

Battelle. 2005. Interim Final Remedial Investigation Centredale Manor Restoration Project Superfund Site North Providence, Rhode Island. Contract No. DACW33-01-D-0004. Delivery Order No. 01. Prepared for U.S. EPA Region 1, Boston, MA, and U.S. Army Corps of Engineers, New England District, Concord, MA. Prepared by Battelle, Duxbury, MA. June 30, 2005.

MACTEC. 2006. Addendum to the Interim Final Baseline Risk Assessment: Oxbow Area, Centredale Manor Restoration Project Superfund Site, North Providence, Rhode Island. Contract No. DACW33-01-D-0004. Delivery Order No. 01. Prepared for U.S. Army Corps of Engineers, New England District, Concord, MA. Prepared by MACTEC Engineering and Consulting, Inc. Wakefield, MA, under contract to Battelle, Duxbury, MA. August.

EXHIBIT 1

SITE MAP AND HOSPITAL ROUTE

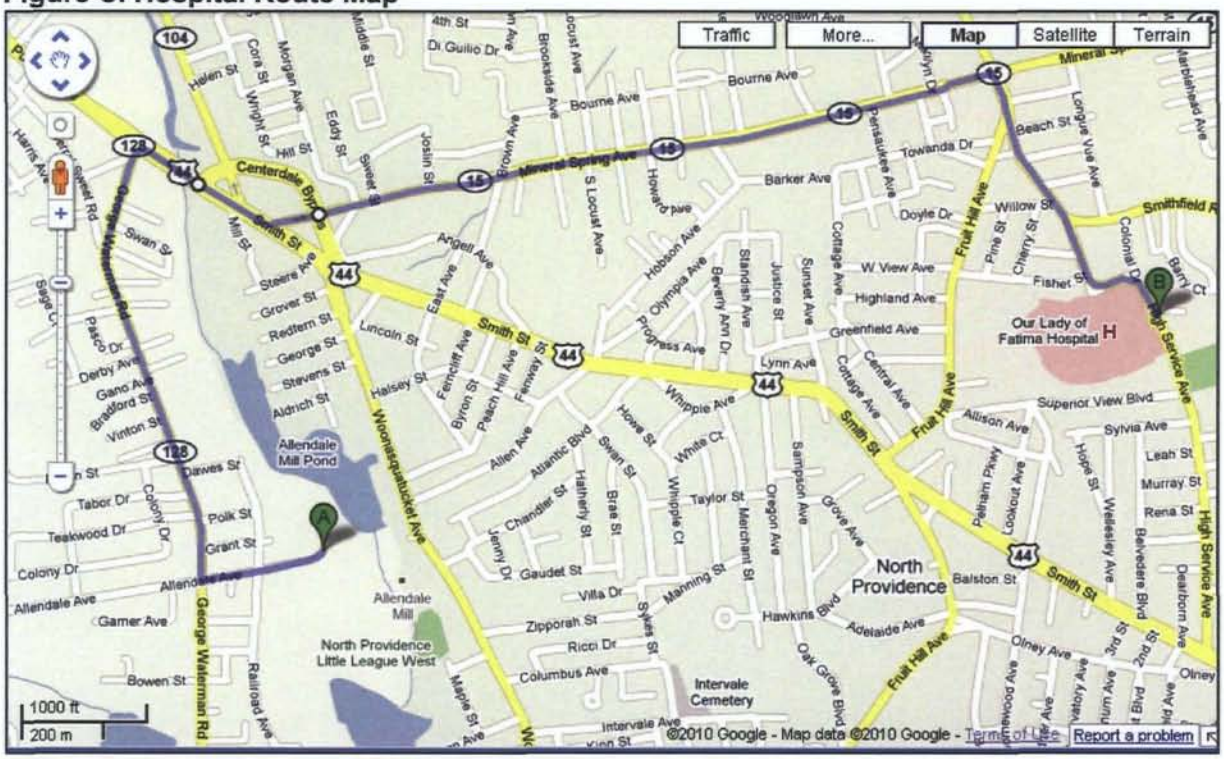
Site Location Map

Site Boat Access

Hospital Information

Category	Information
Hospital Name	Our Lady of Fatima
Address	200 High Service Avenue
City, State	North Providence, RI 02904
Phone	(401) 456-3000
Emergency Phone	(401) 456-3400

Figure C. Hospital Route Map



Driving Directions from Site to Hospital

1. Head **southwest** on **Allendale Ave** toward **Railroad Ave** (go 0.2 mile)
2. Take the 2nd **right** onto **George Waterman Rd/RI-128 N** (go 0.7 mile)
3. Turn **right** at **Putnam Pike/US-44 E**, Continue to follow US-44 E (go 0.2 mile)
4. Turn **left** at **Mineral Spring Ave** (go 1.1 miles)
5. Turn **right** at **Smithfield Rd** (go 322ft)
6. Slight **left** to stay on **Smithfield Rd** (go 0.2 mile)
7. Slight **right** at **High Service Ave** (go 0.2 mile)
8. Arrive at **200 High Service Area** (Total 2.7 miles).

NOTE: The map and traffic directions are provided for planning purposes only. Weather conditions, construction projects, closures, or other events may cause road conditions or directions to differ from the instructions described above. The emergency route should be checked before commencing field work.

EXHIBIT 2

REGULATORY NOTICE

You Have a Right to a Safe and Healthful Workplace.

IT'S THE LAW!

- You have the right to notify your employer or OSHA about workplace hazards. You may ask OSHA to keep your name confidential.
- You have the right to request an OSHA inspection if you believe that there are unsafe and unhealthful conditions in your workplace. You or your representative may participate in the inspection.
- You can file a complaint with OSHA within 30 days of discrimination by your employer for making safety and health complaints or for exercising your rights under the *OSH Act.*
- You have a right to see OSHA citations issued to your employer. Your employer must post the citations at or near the place of the alleged violation.
- Your employer must correct workplace hazards by the date indicated on the citation and must certify that these hazards have been reduced or eliminated.
- You have the right to copies of your medical records or records of your exposure to toxic and harmful substances or conditions.
- Your employer must post this notice in your workplace.

The *Occupational Safety and Health Act of 1970 (OSH Act)*, P.L. 91-596, assures safe and healthful working conditions for working men and women throughout the Nation. The Occupational Safety and Health Administration, in the U.S. Department of Labor, has the primary responsibility for administering the *OSH Act.* The rights listed here may vary depending on the particular circumstances. To file a complaint, report an emergency, or seek OSHA advice, assistance, or products, call 1-800-321-OSHA or your nearest OSHA office: • Atlanta (404) 562-2300 • Boston (617) 565-9860 • Chicago (312) 353-2220 • Dallas (214) 767-4731 • Denver (303) 844-1600 • Kansas City (816) 426-5861 • New York (212) 337-2378 • Philadelphia (215) 861-4900 • San Francisco (415) 975-4310 • Seattle (206) 553-5930. Teletypewriter (TTY) number is 1-877-889-5627. To file a complaint online or obtain more information on OSHA federal and state programs, visit OSHA's website at **www.osha.gov**. If your workplace is in a state operating under an OSHA-approved plan, your employer must post the required state equivalent of this poster.

1-800-321-OSHA
www.osha.gov

U.S. Department of Labor • Occupational Safety and Health Administration • OSHA 3165

Usted Tiene el Derecho a un Lugar de Trabajo Seguro y Saludable.

¡LO ESTABLECE LA LEY!

- Tiene el derecho de notificar a su empleador o a la OSHA sobre cualquier peligro en su lugar de trabajo. Puede pedir a la OSHA que mantenga su nombre en reserva.
- Tiene el derecho de solicitar una inspección de la OSHA si considera que existen condiciones peligrosas y poco saludables en su lugar de trabajo. Usted o su representante puede participar en la inspección.
- Puede presentar un reclamo a OSHA durante un plazo de 30 días si su empleador lo discrimina por presentar reclamos de seguridad y sanidad o por ejercer sus derechos de acuerdo con la Ley.
- Tiene el derecho de ver las citaciones de la OSHA enviadas a su empleador. Su empleador debe colocar las citaciones en un lugar visible en el sitio de la supuesta infracción o cerca de él.
- Su empleador debe corregir los peligros en el lugar de trabajo dentro del plazo indicado en la citación y debe certificar que dichos peligros se hayan reducido o eliminado.
- Tiene el derecho de recibir copias de su historial médico o de los registros de su exposición a sustancias o condiciones tóxicas y peligrosas.
- Su empleador debe colocar este aviso en un lugar visible de su lugar de trabajo.

La Ley de Seguridad y Salud Ocupacionales de 1970 (la Ley), P.L. 91-596, garantiza condiciones ocupacionales seguras y saludables para los hombres y las mujeres que desempeñen algún trabajo en toda la Nación. La Administración de Seguridad y Salud Ocupacionales (OSHA), dependiente del Departamento del Trabajo de los Estados Unidos, es la responsable principal de supervisar la Ley. Los derechos que se indican en este documento pueden variar según las circunstancias particulares. Para presentar un reclamo, informar sobre una emergencia o pedir consejo, asistencia o productos de la OSHA, llame al 1-800-321-OSHA o a la oficina de la OSHA más cercana a usted: • Atlanta (404) 562-2300 • Boston (617) 565-9860 • Chicago (312) 353-2220 • Dallas (214) 767-4731 • Denver (303) 844-1600 • Ciudad de Kansas (816) 426-5861 • Nueva York (212) 337-2378 • Filadelfia (215) 861-4900 • San Francisco (415) 975-4310 • Seattle (206) 553-5930. El número TTY es 1-877-889-5627. Para presentar un reclamo en línea u obtener más información sobre los programas federales y estatales de la OSHA, visite el sitio Web de la OSHA en www.osha.gov. Si su lugar de trabajo se encuentra en un estado que funciona según un plan aprobado por la OSHA, su empleador debe colocar en un sitio visible el equivalente estatal de este afiche.

1-800-321-OSHA
www.osha.gov

Departmento del Trabajo de los E.E. UU. • **Administracion de Seguridad y Salud Ocupacionales** • **OSHA 3167**

EXHIBIT 3

MATERIAL SAFETY DATA SHEETS

MATERIAL SAFETY DATA SHEET

Conventional Gasoline

1. PRODUCT AND COMPANY IDENTIFICATION

Product Name:	Conventional Gasoline
Synonyms:	Gasoline, Unleaded, Conventional (All Grades); Gasoline, Low Sulfur Unleaded (All Grades)
Intended Use:	Fuel
Responsible Party:	ConocoPhillips 600 N. Dairy Ashford Houston, Texas 77079-1175
MSDS Information:	800-762-0942 MSDS@conocophillips.com
Customer Service:	800-527-5476
Technical Information:	800-527-5476

Emergency Overview

24 Hour Emergency Telephone Numbers:
Spill, Leak, Fire or Accident Call CHEMTREC:
North America: (800) 424-9300
Others: (703) 527-3887 (collect)

California Poison Control System: (800) 356-3219

Health Hazards/Precautionary Measures: Cancer hazard. Contains benzene. Causes skin irritation. Aspiration hazard if swallowed. Can enter lungs and cause damage. Use with ventilation adequate to keep exposure below recommended limits, if any. Avoid breathing vapor or mist. Avoid contact with eyes, skin and clothing. Wash thoroughly after handling. Wear appropriate personal protective equipment. Do not taste or swallow.

Physical Hazards/Precautionary Measures: Extremely flammable liquid and vapor. Vapor can cause flash fire. Keep away from heat, sparks, flames, static electricity or other sources of ignition.

Appearance:	Clear to amber
Physical Form:	Liquid
Odor:	Gasoline

NFPA 704 Hazard Class:

Health:	1 (Slight)
Flammability:	3 (High)
Instability:	0 (Least)

2. COMPOSITION / INFORMATION ON INGREDIENTS

HAZARDOUS COMPONENTS					
Component / CAS No:	**Percent (%)**	**ACGIH:**	**OSHA:**	**NIOSH:**	**Other:**
Gasoline NONE	100	300 ppm TWA 890 mg/m^3 TWA 500 ppm STEL 1480 mg/m^3 STEL	NE	NE	---
Xylenes 1330-20-7	0 - 21	100 ppm TWA 434 mg/m^3 TWA 150 ppm STEL 651 mg/m^3 STEL	100 ppm TWA 435 mg/m^3 TWA	900 ppm IDLH	---
Toluene 108-88-3	0 - 15	50 ppm TWA SKIN	200 ppm TWA 300 ppm CEIL 500 ppm 10 min. peak	500 ppm IDLH	---
Ethyl Benzene 100-41-4	0 - 5	100 ppm TWA 434 mg/m^3 TWA 125 ppm STEL 543 mg/m^3 STEL	100 ppm TWA 435 mg/m^3 TWA	800 ppm IDLH	---
Benzene 71-43-2	0 - 5	0.5 ppm TWA-SKIN 2.5 ppm STEL-SKIN	1 ppm TWA ppm STEL	500 ppm IDLH	---
1,2,4-Trimethyl Benzene 95-63-6	0 - 5	25 ppm TWA 123 mg/m^3 TWA	NE	NE	Mixed Isomers
n-Hexane 110-54-3	0 - 4	50 ppm TWA SKIN	500 ppm TWA	1100 ppm IDLH	---
Cyclohexane 110-82-7	0 - 2	100 ppm TWA	300 ppm TWA 1050 mg/m^3 TWA	1300 ppm IDLH	---

Note: State, local or other agencies or advisory groups may have established more stringent limits. Consult an industrial hygienist or similar professional, or your local agencies, for further information.

1%=10,000 PPM.
NE=Not Established

Contains benzene. If exposure concentrations exceed the 0.5 ppm action level, OSHA requirements for personal protective equipment, exposure monitoring, and training may apply (29CFR1910.1028). Also see Section 4.

3. HAZARDS IDENTIFICATION

Potential Health Effects

Eye: Contact may cause mild eye irritation including stinging, watering, and redness.

Skin: Skin irritant. Contact may cause redness, itching, burning, and skin damage. Prolonged or repeated contact can worsen irritation by causing drying and cracking of the skin, leading to dermatitis (inflammation). Not acutely toxic by skin absorption, but prolonged or repeated skin contact may be harmful (see Section 11).

Inhalation (Breathing): Low to moderate degree of toxicity by inhalation.

Ingestion (Swallowing): Low degree of toxicity by ingestion. ASPIRATION HAZARD - This material can enter lungs during swallowing or vomiting and cause lung inflammation and damage.

Signs and Symptoms: Effects of overexposure may include irritation of the respiratory tract, irritation of the digestive tract, nausea, vomiting, flushing, blurred vision, transient excitation followed by signs of nervous system depression (e.g., headache, drowsiness, dizziness, loss of coordination, disorientation and fatigue), tremors, respiratory failure, unconsciousness, convulsions, death.

Cancer: This material is a possible cancer hazard (see Sections 11 and 15).

Target Organs: Inadequate evidence available for this material. See Section 11 for target-organ toxicity information of individual components, if any.

Developmental: No harm to the fetus was observed in laboratory animal studies.

Other Comments: Reports have associated repeated and prolonged occupational overexposure to solvents with permanent brain and nervous system damage (sometimes referred to as Solvent or Painters' Syndrome). Intentional misuse by deliberately concentrating and inhaling this material may be harmful or fatal.

Pre-Existing Medical Conditions: Exposure to high concentrations of this material may increase the sensitivity of the heart to certain drugs. Persons with pre-existing heart disorders may be more susceptible to this effect (see Section 4 - Note to Physicians).

Conditions aggravated by exposure may include respiratory (asthma-like) disorders, skin disorders.

4. FIRST AID MEASURES

Eye: If irritation or redness develops, move victim away from exposure and into fresh air. Flush eyes with clean water. If symptoms persist, seek medical attention.

Skin: Remove contaminated shoes and clothing, and flush affected area(s) with large amounts of water. If skin surface is damaged, apply a clean dressing and seek medical attention. If skin surface is not damaged, cleanse affected area(s) thoroughly by washing with mild soap and water. If irritation or redness develops, seek medical attention.

Inhalation (Breathing): If respiratory symptoms or other symptoms of exposure develop, move victim away from source of exposure and into fresh air. If symptoms persist, seek immediate medical attention. If victim is not breathing, clear airway and immediately begin artificial respiration. If breathing difficulties develop, oxygen should be administered by qualified personnel. Seek immediate medical attention.

Ingestion (Swallowing): Aspiration hazard: Do not induce vomiting or give anything by mouth because this material can enter the lungs and cause severe lung damage. If victim is drowsy or unconscious and vomiting, place on the left side with the head down. If possible, do not leave victim unattended and observe closely for adequacy of breathing. Seek medical attention.

Notes to Physician: Epinephrine and other sympathomimetic drugs may initiate cardiac arrhythmias in persons exposed to high concentrations of hydrocarbon solvents (e.g., in enclosed spaces or with deliberate abuse). The use of other drugs with less arrhythmogenic potential should be considered. If sympathomimetic drugs are administered, observe for the development of cardiac arrhythmias.

Federal regulations (29 CFR 1910.1028) specify medical surveillance programs for certain exposures to benzene above the action level or PEL (specified in Section (i)(1)(i) of the Standard). In addition, employees exposed in an emergency situation shall, as described in Section (i)(4)(i), provide a urine sample at the end of the shift for measurement of urine phenol.

5. FIRE-FIGHTING MEASURES

Flammable Properties:

Flash Point:	-49°F / -45°C
Test Method:	Test Method Unknown
OSHA Flammability Class:	Flammable Liquid
LEL%:	1.4
UEL%:	7.6
Autoignition Temperature:	833°F / 444°C

Unusual Fire & Explosion Hazards: This material is extremely flammable and can be ignited by heat, sparks, flames, or other sources of ignition (e.g., static electricity, pilot lights, mechanical/electrical equipment, and electronic devices such as cell phones, computers, calculators, and pagers which have not been certified as intrinsically safe). Vapors may travel considerable distances to a source of ignition where they can ignite, flash back, or explode. May create vapor/air explosion hazard indoors, in confined spaces, outdoors, or in sewers. If container is not properly cooled, it can rupture in the heat of a fire. Vapors are heavier than air and can accumulate in low areas.

Extinguishing Media: Dry chemical, carbon dioxide, or foam is recommended. Water spray is recommended to cool or protect exposed materials or structures. Carbon dioxide can displace oxygen. Use caution when applying carbon dioxide in confined spaces. Water may be ineffective for extinguishment, unless used under favorable conditions by experienced fire fighters.

Fire Fighting Instructions: For fires beyond the incipient stage, emergency responders in the immediate hazard area should wear bunker gear. When the potential chemical hazard is unknown, in enclosed or confined spaces, or when explicitly required by DOT, a self contained breathing apparatus should be worn. In addition, wear other appropriate protective equipment as conditions warrant (see *Section 8*).

Isolate immediate hazard area, keep unauthorized personnel out. Stop spill/release if it can be done with minimal risk. Move undamaged containers from immediate hazard area if it can be done with minimal risk.

Water spray may be useful in minimizing or dispersing vapors and to protect personnel. Cool equipment exposed to fire with water, if it can be done with minimal risk. Avoid spreading burning liquid with water used for cooling purposes.

6. ACCIDENTAL RELEASE MEASURES

Extremely flammable. Keep all sources of ignition and hot metal surfaces away from spill/release. The use of explosion-proof electrical equipment is recommended.

Stay upwind and away from spill/release. Notify persons down wind of the spill/release, isolate immediate hazard area and keep unauthorized personnel out. Stop spill/release if it can be done with minimal risk. Wear appropriate protective equipment including respiratory protection as conditions warrant (see Section 8).

Prevent spilled material from entering sewers, storm drains, other unauthorized drainage systems, and natural waterways. Dike far ahead of spill for later recovery or disposal. Use foam on spills to minimize vapors (see Section 5). Spilled material may be absorbed into an appropriate absorbent material.

Notify fire authorities and appropriate federal, state, and local agencies. Immediate cleanup of any spill is recommended. If spill of any amount is made into or upon navigable waters, the contiguous zone, or adjoining shorelines, notify the National Response Center (phone number 800-424-8802).

7. HANDLING AND STORAGE

Handling: Open container slowly to relieve any pressure. Bond and ground all equipment when transferring from one vessel to another. Can accumulate static charge by flow or agitation. Can be ignited by static discharge. The use of explosion-proof electrical equipment is recommended and may be required (see appropriate fire codes). Refer to NFPA-704 and/or API RP 2003 for specific bonding/grounding requirements.

Do not enter confined spaces such as tanks or pits without following proper entry procedures such as ASTM D-4276 and 29CFR 1910.146. The use of appropriate respiratory protection is advised when concentrations exceed any established exposure limits (see Sections 2 and 8).

Wash thoroughly after handling. Do not wear contaminated clothing or shoes. Keep contaminated clothing away from sources of ignition such as sparks or open flames. Use good personal hygiene practices.

"Empty" containers retain residue and may be dangerous. Do not pressurize, cut, weld, braze, solder, drill, grind, or expose such containers to heat, flame, sparks, or other sources of ignition. They may explode and cause injury or death. "Empty" drums should be completely drained, properly bunged, and promptly shipped to the supplier or a drum reconditioner. All containers should be disposed of in an environmentally safe manner and in accordance with governmental regulations.

Before working on or in tanks which contain or have contained this material, refer to OSHA regulations, ANSI Z49.1, and other references pertaining to cleaning, repairing, welding, or other contemplated operations.

Storage: Keep container(s) tightly closed. Use and store this material in cool, dry, well-ventilated areas away from heat, direct sunlight, hot metal surfaces, and all sources of ignition. Post area `No Smoking or Open Flame.` Store only in approved containers. Keep away from any incompatible material (see Section 10). Protect container(s) against physical damage. Outdoor or detached storage is preferred. Indoor storage should meet OSHA standards and appropriate fire codes.

Portable Containers: Static electricity may ignite gasoline vapors when filling portable containers. To avoid static buildup do not use a nozzle lock open device. Use only approved containers for the storage of gasoline. Place the container on the ground before filling. Keep the nozzle in contact with the container during filling. Do not fill any portable container in or on a vehicle or marine craft.

8. EXPOSURE CONTROLS / PERSONAL PROTECTION

Engineering controls: If current ventilation practices are not adequate to maintain airborne concentrations below the established exposure limits (see Section 2), additional engineering controls may be required. Where explosive mixtures may be present, electrical systems safe for such locations must be used (see appropriate electrical codes).

Personal Protective Equipment (PPE):

Respiratory: The use of respiratory protection is advised when concentrations are expected to exceed the established exposure limits (see Section 2). Depending on the airborne concentration, use a respirator with appropriate cartridges (NIOSH certified) or supplied-air equipment.

If benzene values equal or exceed applicable exposure limits the use of respiratory protection should comply with the requirements in OSHA 29 CFR 1910.1028-Benzene.

Skin: The use of gloves impervious to the specific material handled is advised to prevent skin contact, possible irritation, and skin damage (see glove manufacturer literature for information on permeability). Depending on conditions of use, apron and/or arm covers may be necessary.

Eye/Face: Approved eye protection to safeguard against potential eye contact, irritation, or injury is recommended. Depending on conditions of use, a face shield may be necessary.

Other Protective Equipment: A source of clean water should be available in the work area for flushing eyes and skin. Impervious clothing should be worn as needed.

Suggestions for the use of specific protective materials are based on readily available published data. Users should check with specific manufacturers to confirm the performance of their products.

9. PHYSICAL AND CHEMICAL PROPERTIES

Note: Unless otherwise stated, values are determined at 20°C (68°F) and 760 mm Hg (1 atm).

Appearance:	Clear to amber
Physical Form:	Liquid
Odor:	Gasoline
Odor Threshold:	No data
pH:	Not applicable
Vapor Pressure (mm Hg):	7-25 psi @ 100°F (38°C), 350-750 mm Hg
Vapor Density (air=1):	No data
Boiling Point:	80-440°F / 26-227°C
Melting/Freezing Point:	No data
Solubility in Water:	Negligible
Partition Coefficient (n-octanol/water) (Kow):	No data
Specific Gravity:	0.72 - 0.75 @ 60°F
Bulk Density:	6.17 lbs/gal
Percent Volatile:	100%
Evaporation Rate (nBuAc=1):	>1
Flash Point:	-49°F / -45°C
Test Method:	Test Method Unknown
LEL%:	1.4
UEL%:	7.6
Autoignition Temperature:	833°F / 444°C

10. STABILITY AND REACTIVITY

Stability: Stable under normal ambient and anticipated storage and handling conditions of temperature and pressure. Extremely flammable liquid and vapor. Vapor can cause flash fire.

Conditions to Avoid: Avoid all possible sources of ignition (see Sections 5 and 7).

Materials to Avoid (Incompatible Materials): Contact with strong oxidizing agents such as acids, chlorine, dichromates, or permanganates can cause fire or explosion.

Hazardous Decomposition Products: The use of hydrocarbon fuel in an area without adequate ventilation may result in hazardous levels of combustion products (e.g., oxides of carbon, sulfur and nitrogen, benzene and other hydrocarbons) and/or dangerously low oxygen levels. See Section 11 for additional information on hazards of engine exhaust.

Hazardous Polymerization: Will not occur.

11. TOXICOLOGICAL INFORMATION

Chronic Data:

Gasoline - NONE

Carcinogenicity: Two year inhalation studies of wholly vaporized unleaded gasoline produced increased incidences of kidney tumors in male rats and liver tumors in female mice. Follow-up studies suggest that occurrence of the kidney tumors may be linked to alpha-2-u-globulin nephropathy, and most likely unique to the male rat. Epidemiology data collected from a study of more than 18,000 petroleum marketing and distribution workers showed no increased risk of leukemia, multiple myeloma, or kidney cancer from gasoline exposure. Unleaded gasoline has been identified as a possible carcinogen by IARC. Because solvent extracts of gasoline exhaust particulates caused skin cancer in laboratory animals, IARC has categorized gasoline engine exhaust as a possible human cancer hazard.

Target Organs: A two year inhalation study of wholly vaporized unleaded gasoline produced nephropathy in male rats, characterized by the accumulation of alpha-2-u- globulin in epithelial cells of the proximal tubules, and necrosis and hyperplasia of surrounding cells. Follow-up studies suggest that these changes are unique to the male rat. Although prolonged exposure to n-hexane, a component of gasoline, has resulted in adverse male reproductive effects in experimental animal studies, no adverse male reproductive effects were found in studies conducted with gasoline.

Developmental: No evidence of developmental toxicity was found in pregnant laboratory animals (rats and mice) exposed to up to 9,000 ppm vapor of unleaded gasoline via inhalation.

Xylenes - 1330-20-7

Target Organs: Rats exposed to 800, 1000 or 1200 ppm 14 hours daily for 6 weeks demonstrated high frequency hearing loss. Another study in rats exposed to 1800 ppm 8 hours daily for 5 days demonstrated middle frequency hearing loss.

Developmental: Both mixed xylenes and the individual isomers produced limited evidence of developmental toxicity in laboratory animals. Inhalation and oral administration of xylene resulted in decreased fetal weight, increased incidences of delayed ossification, skeletal variations and resorptions.

Toluene - 108-88-3

Target Organs: Epidemiology studies suggest that chronic occupational overexposure to toluene may damage color vision. Subchronic and chronic inhalation studies with toluene produced kidney and liver damage, hearing loss and central nervous system (brain) damage in laboratory animals. Intentional misuse by deliberate inhalation of high concentrations of toluene has been shown to cause liver, kidney, and central nervous system damage, including hearing loss and visual disturbances.
Developmental: Exposure to toluene during pregnancy has demonstrated limited evidence of developmental toxicity in laboratory animals. The effects seen include decreased fetal body weight and increased skeletal variations in both inhalation and oral studies.

Ethyl Benzene - 100-41-4

Carcinogenicity: Rats and mice exposed to 0, 75, 250, or 750 ppm ethyl benzene in a two year inhalation study demonstrated limited evidence of kidney, liver, and lung cancer. Ethyl benzene has been listed as a possible human carcinogen by IARC. Ethyl benzene has not been listed as a carcinogen by NTP, or OSHA.
Target Organs: In rats and mice exposed to 0, 75, 250, or 750 ppm ethyl benzene in a two year inhalation study there was mild damage to the kidney (tubular hyperplasia), liver (eosinophilio foci, hypertrophy, necrosis), thyroid (hyperplasia) and pituitary (hyperplasia).

Benzene - 71-43-2

Carcinogenicity: Benzene is known to cause cancer of the blood-forming organs in humans, including acute myelogenous leukemia. It has been identified as a human carcinogen by NTP, IARC and OSHA.
Target Organs: Prolonged or repeated exposures to benzene vapors can cause damage to the blood and blood forming organs, including disorders like leukopenia, thrombocytopenia, and aplastic anemia.
Developmental: Exposure to benzene during pregnancy demonstrated limited evidence of developmental toxicity in laboratory animals. The effects seen include decreased body weight and increased skeletal variations in rodents. Alterations in hematopoeisis have been observed in the fetuses and offspring of pregnant mice.
Mutagenic Effects: Benzene exposure has resulted in chromosomal aberrations in human lymphocytes and animal bone marrow cells, and DNA damage in mammalian cells in vitro.

n-Hexane - 110-54-3

Target Organs: Excessive exposure to n-hexane can result in peripheral neuropathies. The initial symptoms are symmetrical sensory numbness and paresthesias of distal portions of the extremities. Motor weakness is typically observed in muscles of the toes and fingers but may also involve muscles of the arms, thighs and forearms. The onset of these symptoms may be delayed for several months to a year after the beginning of exposure. The neurotoxic properties of n-hexane are potentiated by exposure to methyl ethyl ketone and methyl isobutyl ketone. Prolonged exposure to high concentrations of n-hexane (>1,000 ppm) has resulted in decreased sperm count and degenerative changes in the testes of rats but not those of mice.

Acute Data:

Gasoline - CAS: NONE
Dermal LD50 = >5 ml/kg (Rabbit)
LC50 = > 4500 ppm (Rat)
Oral LD50 = 18.75 ml/kg. (Rat)

Xylenes - CAS: 1330-20-7
Dermal LD50 = >3.16 ml/kg (Rabbit)
LC50 = 5000 ppm/4 hr. (Rat)
Oral LD50 = 4300 mg/kg (Rat)

Toluene - CAS: 108-88-3
Dermal LD50 = 14 g/kg (Rabbit)
LC50 = 8,000 ppm; 49 g/m^3 (4-hr., Rat)
Oral LD50 = 2.5 - 7.9 g/kg (Rat)

Ethyl Benzene - CAS: 100-41-4
Dermal LD50 = 17,800 mg/kg (Rabbit)
LC50 = 4000 ppm/4 hr.; 13367 ppm (Rat)
Oral LD50 = 3500 mg/kg (Rat)

Benzene - CAS: 71-43-2
Dermal LD50 = > 9400 mg/kg (Rabbit)
LC50 = 10000 ppm/7hr. (Rat)
Oral LD50 = 930 mg/kg (Rat)

1,2,4-Trimethyl Benzene - CAS: 95-63-6
Dermal LD50 = No data available
LC50 = 18 gm/m^3/4hr (Rat)
Oral LD50 = 3-6 g/kg (Rat)

n-Hexane - CAS: 110-54-3
Dermal LD50 = >2,000 mg/kg (Rabbit)
LC50 = >3,367 ppm (4-hr., Rat)
Oral LD50 = = 25,000 g/kg (Rat): 28.7 g/kg(Rat)

Cyclohexane - CAS: 110-82-7
Dermal LD50 = >180.2 g/kg (Rabbit)
LC50 = 18,500 ppm (Rabbit)
Oral LD50 = >12.7 g/kg (Rat)

12. ECOLOGICAL INFORMATION

The individual hydrocarbon components of this material are differentially soluble in water with aromatic hydrocarbons tending to be more water soluble than aliphatic hydrocarbons. If spilled, the more volatile components will evaporate rapidly. Factors such as local environmetnal conditions (temperature, wind, soil type, mixing or wave action in water, etc), photo-oxidation, biodegradation and adsorption onto suspended sediments, contribute to the weathering of spilled material. Because of their differential solubility, the occurrence of hydrocarbons in groundwater will be at different proportions than the parent material.

The potential for bioaccumulation and/or long term persistence of these materials in the environment is low to non-existent. In laboratory soil column experiments, the half-time of unleaded gasoline was reported as 1.2 to 2.7 days in sand, loam or clay soils. Microorganisms prsent in sediments and in the water are capable of degrading gasoline and naphtha containing hydrocarbons. Simpler hydrocarbons are more readily degraded than complex molecules. Adaption of bacteria in gasoline-contaminated groundwater to the soluble constituents has been reported.

In general, naphtha streams exhibit some short-term toxicity to freshwater and marine organisms, especially under closed vessel or flow-through exposure conditions in the laboratory. The components most likely causing toxicity are also highly volatile and can be readily biodegraded by microorganisms.

13. DISPOSAL CONSIDERATIONS

This material, if discarded as produced, is not a RCRA "listed" hazardous waste. However, it should be fully characterized for ignitability (D001) and benzene (D018) prior to disposal (40 CFR 261). Use which results in chemical or physical change or contamination may subject it to regulation as a hazardous waste. Along with properly characterizing all waste materials, consult state and local regulations regarding the proper disposal of this material.

Container contents should be completely used and containers should be emptied prior to discard. Container rinsate could be considered a RCRA hazardous waste and must be disposed of with care and in full compliance with federal, state and local regulations. Larger empty containers, such as drums, should be returned to the distributor or to a drum reconditioner. To assure proper disposal of smaller empty containers, consult with state and local regulations and disposal authorities.

14. TRANSPORTATION INFORMATION

DOT

Shipping Description: Gasoline, 3 UN1203, II
Bulk Package/Placard Marking: Flammable/1203
Non-Bulk Package Marking: Gasoline, UN1203
Non-Bulk Package Labeling: Flammable
Packaging - References (Exceptions, Non-Bulk, Bulk): 49 CFR 173.150, 173.202, 173.242
Hazardous Substance: None

Emergency Response Guide: 128

IMDG

Shipping Description: UN1203, Gasoline, 3, II (<-45°C)
Non-Bulk Package Marking: Gasoline, UN 1203
Labels: Flammable
Placards/Marking (Bulk): Flammable/1203
Packaging - Non-Bulk: P001
EMS: F-E, S-E

ICAO/IATA

UN/ID #: UN1203
Proper Shipping Name: Gasoline
Hazard Class/Division: 3
Packing Group: II
Subsidiary risk: None
Non-Bulk Package Marking: Gasoline, UN1203
Labels: Flammable
ERG Code: 3H

	LTD. QTY.	Passenger Aircraft	Cargo Aircraft Only
Packaging Instruction #:	Y305	305	307
Max. Net Qty. Per Package:	1 L	5 L	60 L

15. REGULATORY INFORMATION

U.S. Regulations:

EPA SARA 311/312 (Title III Hazard Categories)

Acute Health: Yes
Chronic Health: Yes
Fire Hazard: Yes
Pressure Hazard: No
Reactive Hazard: No

SARA - Section 313 and 40 CFR 372:

This material contains the following chemicals subject to the reporting requirements of SARA 313 and 40 CFR 372:
Xylenes.....1330-20-7.....0 - 21%
Toluene.....108-88-3.....0 - 15 %
Ethyl Benzene.....100-41-4.....0 - 5%
Benzene.....71-43-2.....0 - 5%
1,2,4-Trimethyl Benzene.....95-63-6.....0 - 5%
n-Hexane.....110-54-3.....0 - 4%
Cyclohexane.....110-82-7.....0 - 2%

EPA (CERCLA) Reportable Quantity (in pounds):

EPA's Petroleum Exclusion applies to this material - (CERCLA 101(14)).

CERCLA/SARA - Section 302 Extremely Hazardous Substances and TPQs (in pounds):

This material contains the following chemicals subject to the reporting requirements of SARA 302 and 40 CFR 372:
-- None Known --

California Proposition 65:

Warning: This material contains the following chemicals which are known to the State of California to cause cancer, birth defects or other reproductive harm, and are subject to the requirements of California Proposition 65 (CA Health & Safety Code Section 25249.5):
Benzene -- Cancer, Developmental and Reproductive Toxicant
Toluene -- Developmental Toxicant
Unleaded Gasoline (wholly vaporized) -- Cancer

Carcinogen Identification:

Unleaded gasoline has been identified as a carcinogen by IARC. For carcinogenicity information on individual components, see Section 11.

TSCA:
All components are listed on the TSCA inventory.

Canadian Regulations:
This product has been classified in accordance with the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all the information required by the CPR.

Domestic Substances List: Listed
WHMIS Hazard Class:
B2 - Flammable Liquids
D2A - Materials Causing Other Toxic Effects - Very Toxic Material
D2B - Materials Causing Other Toxic Effects - Toxic Material

16. OTHER INFORMATION

Issue Date:	25-Oct-2005
Previous Issue Date:	1-Aug-2005
Revised Sections or Basis for Revision:	Periodic review and update
MSDS Code:	251720

Disclaimer of Expressed and implied Warranties:
The information presented in this Material Safety Data Sheet is based on data believed to be accurate as of the date this Material Safety Data Sheet was prepared. HOWEVER, NO WARRANTY OF MERCHANTABILITY, FITNESS FOR ANY PARTICULAR PURPOSE, OR ANY OTHER WARRANTY IS EXPRESSED OR IS TO BE IMPLIED REGARDING THE ACCURACY OR COMPLETENESS OF THE INFORMATION PROVIDED ABOVE, THE RESULTS TO BE OBTAINED FROM THE USE OF THIS INFORMATION OR THE PRODUCT, THE SAFETY OF THIS PRODUCT, OR THE HAZARDS RELATED TO ITS USE. No responsibility is assumed for any damage or injury resulting from abnormal use or from any failure to adhere to recommended practices. The information provided above, and the product, are furnished on the condition that the person receiving them shall make their own determination as to the suitability of the product for their particular purpose and on the condition that they assume the risk of their use. In addition, no authorization is given nor implied to practice any patented invention without a license.

ALDRICH CHEMICAL CO INC. -- LIQUI-NOX PHOSPHATE-FREE DETERGENT, 24302-7 -- 6810-00N016648

========== Product Identification ==========

Product ID:LIQUI-NOX PHOSPHATE-FREE DETERGENT, 24302-7
MSDS Date:01/09/1990
FSC:6810
NIIN:00N016648
MSDS Number: BQTFQ
=== Responsible Party ===
Company Name:ALDRICH CHEMICAL CO INC.
Address:1001 W. ST. PAUL AVE
Box:355
City:MILWAUKEE
State:WI
ZIP:53201
Country:US
Info Phone Num:414-273-3850/FAX -4979
Emergency Phone Num:414-273-3850
CAGE:60928
=== Contractor Identification ===
Company Name:ALDRICH CHEMICAL CO INC
Address:1001 WEST ST PAUL AVE
Box:355
City:MILWAUKEE
State:WI
ZIP:53233
Country:US
Phone:414-273-3850
CAGE:60928

========== Composition/Information on Ingredients ==========

Ingred Name:LIQUI-NOX, PHOSPHATE-FREE DETERGENT

========== Hazards Identification ==========

LD50 LC50 Mixture:NONE SPECIFIED BY MANUFACTURER.
Routes of Entry: Inhalation:YES Skin:YES Ingestion:YES
Reports of Carcinogenicity:NTP:NO IARC:NO OSHA:NO
Health Hazards Acute and Chronic:ACUTE: MAY BE HARMFUL BY INHALATION, INGESTION, OR SKIN ABSORPTION. MAY CAUSE EYE IRRITATION. MAY CAUSE SKIN IRRITATION. TO THE BEST OF OUR KNOWLEDGE, THE CHEMICAL, PHYSICAL, AND TOXICOLOGICAL PROPERT IES HAVE NOT BEEN THOROUGHLY INVESTIGATED.
Explanation of Carcinogenicity:NOT RELEVANT
Effects of Overexposure:SEE HEALTH HAZARDS.
Medical Cond Aggravated by Exposure:NONE SPECIFIED BY MANUFACTURER.

========== First Aid Measures ==========

First Aid:EYE: IMMEDIATELY FLUSH EYES WITH COPIOUS AMOUNTS OF WATER FOR AT LEAST 15 MIN. SKIN: IMMEDIATELY WASH SKIN WITH SOAP AND COPIOUS AMOUNTS OF WATER. INHAL: REMOVE TO FRESH AIR. IF NOT BREATHING GIVE ART F RESP. IF BREATHING IS DIFFICULT, GIVEOXYGEN. CALL A PHYSICIAN. WASH CONTAMINATED CLOTHING BEFORE REUSE. INGEST: GET MD IMMEDIATELY .

========== Fire Fighting Measures ==========

Extinguishing Media:WATER SPRAY, CARBON DIOXIDE, DRY CHEMICAL POWDER, ALCOHOL OR POLYMER FOAM.
Fire Fighting Procedures:WEAR NIOSH/MSHA APPROVED SCBA AND FULL PROTECTIVE EQUIPMENT TO PREVENT CONTACT WITH SKIN AND EYES.
Unusual Fire/Explosion Hazard:NONE SPECIFIED BY MANUFACTURER.

========== Accidental Release Measures ==========

Spill Release Procedures:WEAR NIOSH/MSHA APPROVED RESP, CHEMICAL SAFETY GOGGLES, RUBBER BOOTS AND HEAVY RUBBER GLOVES. ABSORB ON SAND OR VERMICULITE AND PLACE IN CLOSED CONTAINERS FOR DISPOSAL. VENTILATE AREA AND WASH SPILL S ITE AFTER MATERIAL PICKUP IS COMPLETE.
Neutralizing Agent:NONE SPECIFIED BY MANUFACTURER.

========== Handling and Storage ==========

Handling and Storage Precautions:KEEP TIGHTLY CLOSED. STORE IN A COOL DRY PLACE. AVOID INHALATION. AVOID CONTACT WITH EYES, SKIN AND CLOTHING. AVOID PROLONGED OR REPEATED EXPOSURE.
Other Precautions:NONE SPECIFIED BY MANUFACTURER.

========== Exposure Controls/Personal Protection ==========

Respiratory Protection:NIOSH/MSHA APPROVED RESPIRATOR.
Ventilation:MECHANICAL EXHAUST REQUIRED.
Protective Gloves:COMPATIBLE CHEMICAL-RESISTANT GLOVES.
Eye Protection:CHEMICAL SAFETY GOGGLES.
Other Protective Equipment:SAFETY SHOWER AND EYE BATH.
Work Hygienic Practices:WASH THOROUGHLY AFTER HANDLING.
Supplemental Safety and Health
WASTE DISP: AND NEUTRALIZATION REACTIONS MAY GENRATE HEAT & FUMES WHICH CAN BE CONTROLLED BY THE RATE OF ADDITION. OBSERVE ALL FEDERAL, STATE AND LOCAL LAWS.

========== Physical/Chemical Properties ==========

HCC:N1
Spec Gravity:1.051
Appearance and Odor:NONE SPECIFIED BY MANUFACTURER.

========== Stability and Reactivity Data ==========

Stability Indicator/Materials to Avoid:YES
STRONG OXIDIZING AGENTS.
Stability Condition to Avoid:NONE SPECIFIED BY MANUFACTURER.
Hazardous Decomposition Products:NATURE OF DECOMPOSITION PRODUCTS NOT KNOWN.

========== Disposal Considerations ==========

Waste Disposal Methods:SML QTYS: CAUTIOUSLY ADD TO A LRG STIRRED EXCESS OF WATER. ADJUST THE PH TO NEUTRAL, SEPARATE ANY INSOLUBLE SOLIDS OR LIQUIDS & PACKAGE THEM FOR HAZARDOUS-WASTE DISP. FLUSH THE AQUEOUS SOLN DOWN THE D RAIN W/PLENTY OF WATER. THE HYDROLYSIS (SUPP DATA)

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ALCONOX MSDS

Section 1 : MANUFACTURER INFORMATION

Product name: Alconox

Supplier: Same as manufacturer.

Manufacturer: Alconox, Inc.
30 Glenn St.
Suite 309
White Plains, NY 10603.

Manufacturer emergency phone number: 800-255-3924.
813-248-0585 (outside of the United States).

Manufacturer: Alconox, Inc.
30 Glenn St.
Suite 309
White Plains, NY 10603.

Supplier MSDS date: 2005/03/09

D.O.T. Classification: Not regulated.

Section 2 : HAZARDOUS INGREDIENTS

C.A.S.	CONCENTRATION %	Ingredient Name	T.L.V.	LD/50	LC/50
25155-30-0	10-30	SODIUM DODECYLBENZENESULFONATE	NOT AVAILABLE	438 MG/KG RAT ORAL 1330 MG/KG MOUSE ORAL	NOT AVAILABLE
497-19-8	7-13	SODIUM CARBONATE	NOT AVAILABLE	4090 MG/KG RAT ORAL 6600 MG/KG MOUSE ORAL	2300 MG/M3/2H RAT INHALATION 1200 MG/M3/2H MOUSE INHALATION
7722-88-5	10-30	TETRASODIUM PYROPHOSPHATE	5 MG/M3	4000 MG/KG RAT ORAL 2980 MG/KG MOUSE ORAL	NOT AVAILABLE
7758-29-4	10-30	SODIUM PHOSPHATE	NOT AVAILABLE	3120 MG/KG RAT ORAL 3100 MG/KG MOUSE ORAL >4640 MG/KG RABBIT DERMAL	NOT AVAILABLE

Section 2A : ADDITIONAL INGREDIENT INFORMATION

Note: (supplier).
CAS# 497-19-8: LD50 4020 mg/kg - rat oral.
CAS# 7758-29-4: LD50 3100 mg/kg - rat oral.

Section 3 : PHYSICAL / CHEMICAL CHARACTERISTICS

Physical state: Solid

Appearance & odor: Almost odourless.
White granular powder.

Odor threshold (ppm): Not available.

Vapour pressure (mmHg): Not applicable.

Vapour density (air=1): Not applicable.

By weight: Not available.

Evaporation rate (butyl acetate = 1): Not applicable.

Boiling point (°C): Not applicable.

Freezing point (°C): Not applicable.

pH: (1% aqueous solution).
9.5

Specific gravity @ 20 °C: (water = 1).
0.85 - 1.10

Solubility in water (%): 100 - > 10% w/w

Coefficient of water\oil dist.: Not available.

VOC: None

Section 4 : FIRE AND EXPLOSION HAZARD DATA

Flammability: Not flammable.

Conditions of flammability: Surrounding fire.

Extinguishing media: Carbon dioxide, dry chemical, foam.
Water
Water fog.

Special procedures: Self-contained breathing apparatus required.
Firefighters should wear the usual protective gear.

Auto-ignition temperature: Not available.

Flash point (°C), method: None

Lower flammability limit (% vol): Not applicable.

Upper flammability limit (% vol): Not applicable.

Not available.

Sensitivity to mechanical impact: Not applicable.

Hazardous combustion products: Oxides of carbon (COx).
Hydrocarbons.

Rate of burning: Not available.

Explosive power: None

Section 5 : REACTIVITY DATA

Chemical stability: Stable under normal conditions.

Conditions of instability: None known.

Hazardous polymerization: Will not occur.

Incompatible substances: Strong acids.
Strong oxidizers.

Hazardous decomposition products: See hazardous combustion products.

Section 6 : HEALTH HAZARD DATA

Route of entry: Skin contact, eye contact, inhalation and ingestion.

Effects of Acute Exposure

Eye contact: May cause irritation.

Skin contact: Prolonged contact may cause irritation.

Inhalation: Airborne particles may cause irritation.

Ingestion: May cause vomiting and diarrhea.
May cause abdominal pain.
May cause gastric distress.

Effects of chronic exposure: Contains an ingredient which may be corrosive.

LD50 of product, species & route: > 5000 mg/kg rat oral.

LC50 of product, species & route: Not available for mixture, see the ingredients section.

Exposure limit of material: Not available for mixture, see the ingredients section.

Sensitization to product: Not available.

Carcinogenic effects: Not listed as a carcinogen.

Reproductive effects: Not available.

Teratogenicity: Not available.

Mutagenicity: Not available.

Synergistic materials: Not available.

Medical conditions aggravated by exposure: Not available.

First Aid

Skin contact: Remove contaminated clothing.
Wash thoroughly with soap and water.
Seek medical attention if irritation persists.

Eye contact: Check for and remove contact lenses.
Flush eyes with clear, running water for 15 minutes while holding eyelids open: if irritation persists, consult a physician.

Inhalation: Remove victim to fresh air.
Seek medical attention if symptoms persist.

Ingestion: Dilute with two glasses of water.
Never give anything by mouth to an unconscious person.
Do not induce vomiting, seek immediate medical attention.

Section 7 : PRECAUTIONS FOR SAFE HANDLING AND USE

Leak/Spill: Contain the spill.
Recover uncontaminated material for re-use.
Wear appropriate protective equipment.
Contaminated material should be swept or shoveled into appropriate waste container for disposal.

Waste disposal: In accordance with municipal, provincial and federal regulations.

Handling procedures and equipment: Protect against physical damage.
Avoid breathing dust.
Wash thoroughly after handling.
Keep out of reach of children.
Avoid contact with skin, eyes and clothing.
Launder contaminated clothing prior to reuse.

Storage requirements: Keep containers closed when not in use.
Store away from strong acids or oxidizers.
Store in a cool, dry and well ventilated area.

Section 8 : CONTROL MEASURES

Precautionary Measures

Gloves/Type:

Neoprene or rubber gloves.

Respiratory/Type:

If exposure limit is exceeded, wear a NIOSH approved respirator.

Eye/Type:

Safety glasses with side-shields.

Footwear/Type: Safety shoes per local regulations.

Clothing/Type: As required to prevent skin contact.

Other/Type: Eye wash facility should be in close proximity.
Emergency shower should be in close proximity.

Ventilation requirements: Local exhaust at points of emission.

EXHIBIT 4

SAFETY PROCEDURES

HEAT EXHAUSTION

What happens to the body:

Headaches, dizziness, or light-headedness, weakness, mood changes, irritability or confusion, feeling sick to your stomach, vomiting, fainting, decreased and dark-colored urine, and pale, clammy skin.

What should be done:

- Move the person to a cool shaded area. Don't leave the person alone. If the person is dizzy or light-headed, lay him on his back and raise his legs about 6-8 inches. If the person is sick to his stomach, lay him on his side.
- Loosen and remove heavy clothing.
- Have the person drink some cool water (a small cup every 15 minutes) if he is not feeling sick to his stomach.
- Try to cool the person by fanning him. Cool the skin with a cool spray mist of water or wet cloth.
- If the person does not feel better in a few minutes call for emergency help (ambulance or call 911.)

(If heat exhaustion is not treated, the illness may advance to heat stroke.)

How to Protect Workers

- Learn the signs and symptoms of heat-induced illnesses and what to do to help the worker.
- Train workers about heat-induced illnesses.
- Perform the heaviest work during the coolest part of the day.
- Slowly build up tolerance to the heat and the work activity (usually takes up to 2 weeks.)
- Use the buddy system (work in pairs.)
- Drink plenty of cool water (one small cup every 15-20 minutes.)
- Wear light, loose-fitting, breathable (like cotton) clothing.
- Take frequent short breaks in cool, shaded areas (allow your body to cool down.)
- Avoid eating large meals before working in hot environments.
- Avoid caffeine and alcoholic beverages (these beverages make the body lose water and increase the risk of heat illnesses.)

Workers are at increased risk when...

- They take certain medications. Check with your doctor, nurse, or pharmacy to see if medicines you take affect you when working in hot environments.
- They have had a heat-induced illness in the past.
- They wear personal protective equipment.

HEAT STROKE - A Medical Emergency

What happens to the body:

Dry, pale skin (no sweating); hot red skin (looks like a sunburn); mood changes; irritability, confusion, and not making any sense; seizures or fits, and collapse (will not respond).

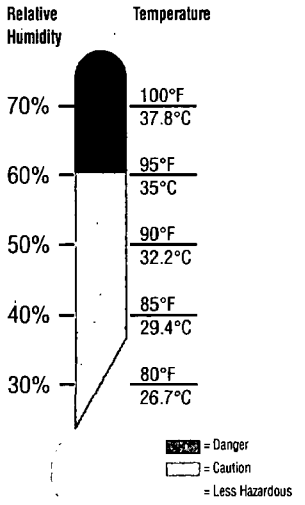
What should be done:

- Call for emergency help (i.e., ambulance or 911.)
- Move the person to a cool, shaded area. Don't leave the person alone. Lay him on his back and if the person is having seizures, remove objects close to him so he won't hit them. If the person is sick to his stomach, lay him on his side.
- Remove heavy and outer clothing.
- Have the person drink some cool water (a small cup every 15 minutes) if he is alert enough to drink anything and not feeling sick to his stomach.
- Try to cool the person by fanning him or her. Cool the skin with a cool spray mist of water, wet cloth, or wet sheet.
- If ice is available, place ice packs in armpits and groin area.

THE HEAT EQUATION

HIGH TEMPERATURE + HIGH HUMIDITY + PHYSICAL WORK = HEAT ILLNESS

When the body is unable to cool itself through sweating, **serious** heat illnesses may occur. The most severe heat-induced illnesses are **heat exhaustion** and **heat stroke**. If actions are not taken to treat heat exhaustion, the illness could progress to heat stroke and **death**.



Oregon Occupational Safety & Health Division
440-3333E-S(9/03)

ATTACHMENT A2

STANDARD OPERATING PROCEDURES

STANDARD OPERATING PROCEDURE (SOP) AP-01

SAMPLE PACKAGING AND SHIPPING

SCOPE AND APPLICATION

This SOP describes specific requirements for sample packaging and shipping to ensure the proper transfer and documentation of environmental samples collected during field operations. Procedures for the careful and consistent transfer of samples from the field to the laboratory are outlined herein. This SOP also presents the method to be used when packing samples that will either be hand delivered or shipped by commercial carrier to the laboratory.

EQUIPMENT AND SUPPLIES REQUIRED

Make sure that you have the equipment and supplies necessary to properly pack and ship environmental samples, including the following:

- Project-specific sampling and analysis plan (SAP)
- Project-specific field logbook
- Sealable airtight bags in assorted sizes (e.g., Ziploc®)
- Wet ice in doubled, sealed bags; frozen Blue Ice®; or dry ice
- Cooler(s)
- Bubble wrap
- Fiber-reinforced packing tape, clear plastic packing tape, and duct tape
- Scissors or knife
- Chain-of-custody (COC) forms
- COC seals
- Large plastic garbage bags (preferably 3 mil [0.003 in.] thick)
- Paper towels
- "Fragile," "This End Up," or "Handle With Care" labels
- Mailing labels
- Air bills for overnight shipment

PROCEDURE

Customize the logistics for sample packaging and shipping to each study. If necessary, transfer samples from the field to a local storage facility where they can be frozen or refrigerated. Depending on the logistics of the operation, field personnel may transport samples to the laboratory or use a commercial courier or shipping service. In the latter case, Integral field personnel must be aware of any potentially limiting factors to timely shipping, such as availability of overnight service and weekend deliveries to specific areas, and shipping regulations regarding "restricted articles" (e.g., dry ice, formalin) prior to shipping the samples.

SAMPLE PREPARATION

Take the following steps to ensure the proper transfer of samples from the field to the laboratories:

At the sample collection site:

1. Document all samples using the proper logbooks or field forms (see SOP AP-02), required sample container identification (i.e., sample labels with tag numbers), and COC form (example provided in SOP AP-03). Fill out the COC form as described in SOP AP-03, and use the sample labeling techniques provided in SOP AP-04.
2. Make all applicable laboratory quality control sample designations on the COC forms. Clearly identify samples that will be archived for future possible analysis. Label these samples as follows: "Do Not Analyze: Hold and archive for possible future analysis." Some laboratories interpret "archive" to mean that they should continue holding the residual sample after analysis.
3. Notify the laboratory contact and the Integral project quality assurance/quality control (QA/QC) coordinator that samples will be shipped and the estimated arrival time. Send copies of all COC forms to Integral's project QA/QC coordinator or project manager, as appropriate.
4. Keep the samples in the possession of the sampling personnel at all times. Lock and secure any temporary onsite sample storage areas to maintain sample integrity and COC requirements.
5. Clean the outside of all dirty sample containers to remove any residual material that may lead to cross-contamination.
6. Complete the COC form as described in SOP AP-03, and retain the back (pink) copy for project records prior to sealing the cooler. Check sample containers against the COC form to ensure all the samples that were collected are in the cooler.

7. Store each sample container in a sealed plastic bag that allows the sample label (example provided in SOP AP-03) to be read. Before sealing the bags, ensure that volatile organic analyte (VOA) vials are encased in a foam sleeve or in bubble wrap.
8. If the samples require storage at a specific temperature, place enough ice in the sample cooler to maintain the temperature (e.g., 4°C) throughout the sampling day.

At the sample processing area (immediately after sample collection) take the following steps:

1. If the samples require a specific storage temperature, then cool the samples and maintain the temperature prior to shipping. For example, place enough ice in each sample cooler to maintain the temperature at 4°C until processing begins at the testing laboratory.
2. Be aware of holding time requirements for project-specific analytes and arrange the sample shipping schedule accordingly.
3. Place samples in secure storage (i.e., locked room or vehicle) or keep them in the possession of Integral sampling personnel before shipment. Lock and secure any sample storage areas to maintain sample integrity and COC requirements.
4. Store samples in the dark (e.g., keep coolers shut).

At the sample processing area (just prior to shipping), do the following:

1. Check sample containers against the COC form to account for all samples intended for shipment.
2. Choose cooler(s) of appropriate size and make sure they are clean of gross contamination inside and out. If the cooler has a drain, close the drain and secure it with duct tape.
3. Line the cooler with bubble wrap and place a large plastic bag (preferably with a thickness of 3 mil), open, inside the cooler.
4. Individually wrap each glass container (which was sealed in a plastic bag at the collection site) in bubble wrap and secure with tape or a rubber band. Place the wrapped samples in the large plastic bag in the cooler, leaving room for ice to keep the samples cold (i.e., 4°C).
5. If temperature blanks have been provided by the testing laboratory, place one temperature blank in each sample cooler.
6. If the samples require a specific storage temperature, add enough wet ice or Blue Ice® to maintain that temperature during overnight shipping (i.e., 4°C). Always overestimate the amount of ice that will be required. Keep ice in a sealed plastic bag, which is placed in a second sealed plastic bag to prevent leakage. Avoid separating the samples from the ice with excess bubble wrap because it may insulate the samples from the ice. After adding all samples and ice to the cooler, use bubble wrap (or other

available clean packing material) to fill any empty space and prevent the samples from shifting during transport.

7. If possible, consolidate all VOA samples in a single cooler and ship them with (a) trip blank(s) if the project-specific QA project plan calls for them.
8. Sign, date, and include any tracking numbers provided by the shipper on the COC form. Remove the back (pink) copy of the original COC form and retain this copy for the project records.
9. Seal the rest of the signed COC form in a bag and tape the bag to the inside of the cooler lid. Each cooler should contain an individual COC form for the samples contained inside it. If time is short and it becomes necessary to combine all the samples onto a single set of COC forms and ship multiple coolers together, then indicate on the outside of the appropriate cooler, "Chain-of-Custody Inside."
10. After the cooler is sufficiently packed to prevent shifting of the containers, close the lid and seal it with fiber-reinforced packing tape. Tape the cooler around the opening, joining the lid to the bottom, and around the circumference of the cooler at both hinges.
11. As security against unauthorized handling of the samples, apply two COC seals across the opening of the cooler lid (provided with example field forms). Place one seal on the front right portion of the cooler and one on the back left. Be sure the seals are properly affixed to the cooler to prevent removal during shipment. Additional tape across the seal may be necessary if the outside of the cooler is wet.

SAMPLE SHIPPING

Hand Delivery to the Testing Laboratory

1. Notify the laboratory contact and the Integral project QA/QC coordinator that samples will be delivered to the laboratory and the estimated arrival time.
2. When hand-delivering environmental samples, make sure the testing laboratory receives them on the same day that they were packed in the coolers.
3. Fax or scan and e-mail copies of all COC forms to the Integral project QA/QC coordinator. Note: It may be necessary to photocopy the COC form on a slightly darker setting so the form is readable after it has been faxed. Never leave the original COC form in the custody of non-Integral staff.

Shipped by Commercial Carrier to the Laboratory

1. Apply a mailing label to the cooler with destination and return addresses, and add other appropriate stickers, such as "This End Up," "Fragile," and "Handle With Care." If the shipment contains multiple coolers, indicate on the mailing label the number of coolers that the testing laboratory should expect to receive (e.g., 1 of 2; 2 of 2). Place clear tape over the mailing label to firmly affix it to the cooler and to protect it from the weather. This is a secondary label in case the air bill is lost during shipment.
2. Fill out the air bill and fasten it to the handle tags provided by the shipper (or the top of the cooler if handle tags are not available).
3. If samples must be frozen (–20°C) during shipping, make sure that dry ice has been placed in the sample cooler. Be aware of any additional shipping, handling, and special labeling requirements that the shipper may require.
4. Make sure that benthic infauna samples have been preserved with formalin in the field prior to shipping. Be aware of any additional shipping, handling, and special labeling requirements that the shipper may require for these samples.
5. Notify the laboratory contact and the Integral project QA/QC coordinator that samples will be shipped and the estimated arrival date and time. If environmental samples must be shipped at 4°C or –20°C, choose overnight shipping for delivery next morning. Fax or scan and e-mail copies of all COC forms to the Integral project QA/QC coordinator. Note: It may be necessary to photocopy the COC form on a slightly darker setting so the form is readable after faxing. Never leave the original COC form in the custody of non-Integral staff.

STANDARD OPERATING PROCEDURE (SOP) AP-02

FIELD DOCUMENTATION

SCOPE AND APPLICATION

This SOP describes the Integral procedure for accurate record-keeping in the field for the purposes of ensuring that samples can be traced from collection to final disposition.

Document all information relevant to field operations properly to ensure that activities are accounted for in written records to the extent that someone not present at the site could reconstruct the activity without relying on the memory of the field crew. Several types of field documents are used for this purpose and should be consistently used by field personnel. Field documentation should include only a factual description of site-related activities and observations. Field personnel should not include superfluous comments or speculation regarding the field activities or observations.

FIELD LOGBOOKS

During field sampling events, field logbooks must be used to record all daily activities. The purpose of the field logbook is to document events and record data measured in the field to the extent that someone not present at the site could reconstruct the activity without relying on the memory of the field crew. The project manager (or designee) should issue a field logbook to the appropriate site personnel for the direction of onsite activities (e.g., reconnaissance survey team leader, sampling team leader). It is this designee's responsibility to maintain the site logbook while it is in his or her possession and return it to the project manager or turn it over to another field team.

Make entries in the field logbook as follows:

1. Document all daily field activities in indelible ink in the logbook and make no erasures. Make corrections with a single line-out deletion, followed by the author's initials and the date. The author must initial and date each page of the field logbook. The author must sign and date the last page at the end of each day, and draw a line through any blank space remaining on the page below the last entry.

2. Write the project name, dates of the field work, site name and location (city and state), and Integral job number on the cover of the field logbook. If more than one logbook is used during a single sampling event, then annotate the upper right-hand corner of the logbook (e.g., Volume 1 of 2, 2 of 2) to indicate the number of logbooks used during the field event. Secure all field logbooks when not in use in the field. The following is a list of the types of information that is appropriate for entry in the field notebook:
   - Project start date and end date
   - Date and time of entry (24-hour clock)
   - Time and duration of daily sampling activities
   - Weather conditions at the beginning of the field work and any changes that occur throughout the day, including the approximate time of the change (e.g., wind speed and direction, rain, thunder, wave action, current, tide, vessel traffic, air and water temperature, thickness of ice if present)
   - Name and affiliation of person making entries and other field personnel and their duties, including what times they are present
   - The location and description of the work area, including sketches, map references, and photograph log, if appropriate
   - Level of personal protection being used
   - Onsite visitors (names and affiliations), if any, including what times they are present
   - The name, agency, and telephone number of any field contacts
   - Notation of the coordinate system used to determine the station location
   - The sample identifier and analysis code for each sample to be submitted for laboratory analysis, if not included on separate field data sheets
   - All field measurements made (or reference to specific field data sheets used for this purpose), including the time of collection and the date of calibration, if appropriate
   - The sampling location name, date, gear, water depth (if applicable), and sampling location coordinates, if not included on separate field data sheets
   - For aquatic sampling, the type of vessel used (e.g., size, power, type of engine)
   - Specific information on each type of sampling activity
   - The sample type (e.g., groundwater, soil, surface sediment), sample number, sample tag number, and any preservatives used, if not included on separate field data sheets
   - Sample storage methods

- Cross-references of numbers for duplicate samples
- A description of the sample (source and appearance, such as soil or sediment type, color, texture, consistency, presence of biota or debris, presence of oily sheen, changes in sample characteristics with depth, presence/location/thickness of the redox potential discontinuity [RPD] layer, and odor) and penetration depth, if not included on separate field data sheets
- Estimate of length and appearance of recovered cores, if not included on separate field data sheets
- Photographs (uniquely identified) taken at the sampling location, if any
- Details of the work performed
- Variations, if any, from the project-specific sampling and analysis plan (SAP) or standard operating protocols and reasons for deviation
- Details pertaining to unusual events that might have occurred during sample collection (e.g., possible sources of sample contamination, equipment failure, unusual appearance of sample integrity, control of vertical descent of the sampling equipment)
- References to other logbooks or field forms used to record information (e.g., field data sheets, health and safety log)
- Any field results not appearing on the field data sheets (if used), including station identification and location, date, and time of measurement
- Sample shipment information (e.g., shipping manifests, chain-of-custody (COC) form numbers, carrier, air bill numbers, time addresses)
- A record of quantity of investigation-derived wastes (if any) and storage and handling procedures.

3. During the field day, as listed above, record in the logbook a summary of all site activities. Provide a date and time for each entry. The information need not duplicate anything recorded in other field logbooks or field forms (e.g., site health and safety officer's logbook, calibration logbook, field data sheets), but should summarize the contents of the other logbooks and refer to the pages in these logbooks for detailed information.
4. If measurements are made at any location, record the measurements and equipment used, or refer to the logbook and page number(s) or field forms on which they are recorded. All maintenance and calibration records for equipment should be traceable through field records to the person using the instrument and to the specific piece of instrumentation itself.

5. Upon completion of the field sampling event, the sampling team leader will be responsible for submitting all field logbooks to be copied. A discussion of copy distribution is provided below.

FIELD DATA FORMS

Occasionally, additional field data forms are generated during a field sampling event (e.g., groundwater monitoring form, sediment core profile form, water quality measurement form) to record the relevant sample information collected. For instructions regarding the proper identification of field data forms, sampling personnel should consult the project-specific SAP.

Upon completion of the field sampling event, the sampling team leader will be responsible for submitting all field data forms to be copied. A discussion of copy distribution is provided below.

PHOTOGRAPHS

In certain cases, photographs (print or digital) of sampling stations may be taken using a camera-lens system with a perspective similar to the naked eye. Ensure that photographs include a measured scale in the image, when practical. If you take photographs of sample characteristics and routine sampling activities, avoid using telephoto or wide-angle shots, because they cannot be used in enforcement proceedings. Record the following items in the field logbook for each photograph taken:

1. The photographer's name or initials, the date, the time of the photograph, and the general direction faced (orientation)
2. A brief description of the subject and the field work shown in the picture
3. For print photographs, the sequential number of the photograph and the roll number on which it is contained
4. For digital photographs, the sequential number of the photograph, the file name, the file location, and back-up disk number (if applicable).

Upon completion of the field sampling event, the sampling team leader is responsible for submitting all photographic materials to be developed (prints) or copied (disks). Place the prints or disks and associated negatives in the project files (at the Integral project manager's location). Make photocopies of photo logs and any supporting documentation from the field logbooks, and place them in the project files with the prints or disks.

EQUIPMENT CALIBRATION RECORDS

Record in the field logbook all equipment calibration records, including instrument type and serial number, calibration supplies used, calibration methods and calibration results, date, time, and personnel performing the calibration. Calibrate all equipment used during the investigation daily, at a minimum, in accordance with the manufacturers' recommendations.

DISTRIBUTION OF COPIES

At Integral offices, make two copies of all field logbooks and additional field data forms. Stamp the first copy with a "COPY" stamp, and place it in the project file to be available for general staff use. Stamp the second copy with a "FILE" stamp, and place it in the data management file with the laboratory data packages, to be used by the data management and quality assurance staff only. Place the original field logbooks and forms in a locked file cabinet.

SET-UP OF LOCKING FILE CABINET

Place each project in its own file folder in a locking file cabinet. On the folder label, include the project name and contract number. Each project folder will include up to six kinds of files:

- Field logbook(s)
- Additional field data forms
- Photographs
- COC forms
- Acknowledgment of Sample Receipt forms
- Archive Record form (to be completed only if samples are archived at an Integral field storage facility or Integral laboratory).

STANDARD OPERATING PROCEDURE (SOP) AP-03

SAMPLE CUSTODY

SCOPE AND APPLICATION

This SOP describes Integral procedures for custody management of environmental samples.

A stringent, established program of sample chain-of-custody will be followed during sample storage and shipping activities to account for each sample. The procedure outlined herein will be used with SOP AP-01, which covers sample packaging and shipping; SOP AP-02, which covers the use of field logbooks and other types of field documentation; and SOP AP-04, which covers sample labeling. Chain-of-custody (COC) forms ensure that samples are traceable from the time of collection through processing and analysis until final disposition. A sample is considered to be in a person's custody if any of the following criteria are met:

1. The sample is in the person's possession
2. The sample is in the person's view after being in his or her possession
3. The sample is in the person's possession and is being transferred to a designated secure area
4. The sample has been locked up to prevent tampering after it was in the person's possession.

At no time is it acceptable for samples to be outside of Integral personnel's custody unless the samples have been transferred to a secure area (i.e., locked up). If the samples cannot be placed in a secure area, then an Integral field team member must physically remain with the samples (e.g., at lunch time one team member must remain with the samples).

CHAIN-OF-CUSTODY FORMS

The COC form is critical because it documents sample possession from the time of collection through final disposition. The form also provides information to the laboratory regarding what analyses are to be performed on the samples that are shipped.

Complete the COC form after each field collection activity and before shipping the samples to the laboratory. Sampling personnel are responsible for the care and custody of the samples until they are shipped. The individuals relinquishing and receiving the samples must sign the

COC form(s), indicating the time and date of the transfer, when transferring possession of the samples.

A COC form consists of three-part carbonless paper with white, yellow, and pink copies. The sampling team leader keeps the pink copy. The white and yellow sheets are placed in a sealed plastic bag and secured inside the top of each transfer container (e.g., cooler). Field staff retain the pink sheet for filing at the Integral project manager's location. Each COC form has a unique four-digit number. This number and the samples on the form must be recorded in the field logbook. Integral also uses computer-generated COC forms. If computer-generated forms are used, then the forms must be printed in triplicate and all three sheets signed so that two sheets can accompany the shipment to the laboratory and one sheet can be retained on file. Alternatively, if sufficient time is available, the computer-generated forms will be printed on three-part carbonless paper.

Record on the COC form the project-assigned sample number and the unique tag number at the bottom of each sample label. The COC form also identifies the sample collection date and time, type of sample, project name, and sampling personnel. In addition, the COC form provides information on the preservative or other sample pretreatment applied in the field and the analyses to be conducted by referencing a list of specific analyses or the statement of work for the laboratory. The COC form is sent to the laboratory along with the sample(s).

PROCEDURES

Use the following guidelines to ensure the integrity of the samples:

1. Sign and date each COC form. Have the person who relinquishes custody of the samples also sign this form.
2. At the end of each sampling day and prior to shipping or storage, make COC entries for all samples. Check the information on the labels and tags against field logbook entries.
3. Do not sign the COC form until the team leader has checked the information for inaccuracies. Make corrections by drawing a single line through any incorrect entry, and then initial and date it. Make revised entries in the space below the entries. After making corrections, mark out any blank lines remaining on the COC form, using single lines that are initialed and dated. This procedure will prevent any unauthorized additions.

   At the bottom of each COC form is a space for the signatures of the persons relinquishing and receiving the samples and the time and date of the transfer. The time the samples were relinquished should match exactly the time they were received by another party. Under no circumstances should there be any time when custody of the samples is undocumented.

4. If samples are sent by a commercial carrier not affiliated with the laboratory, such as FedEx or United Parcel Service (UPS), record the name of the carrier on the COC form. Also enter on the COC form any tracking numbers supplied by the carrier. The time of transfer should be as close to the actual drop-off time as possible. After signing the COC forms and removing the pink copy, seal them inside the transfer container.
5. If errors are found after the shipment has left the custody of sampling personnel, make a corrected version of the forms and send it to all relevant parties. Fix minor errors by making the change on a copy of the original with a brief explanation and signature. Errors in the signature block may require a letter of explanation.
6. Provide a COC form and an Archive Record form for any samples that are archived internally at Integral.

Upon completion of the field sampling event, the sampling team leader is responsible for submitting all COC forms to be copied. A discussion of copy distribution is provided in SOP AP-02.

CUSTODY SEAL

As security against unauthorized handling of the samples during shipping, affix two custody seals to each sample cooler. Place the custody seals across the opening of the cooler (front right and back left) prior to shipping. Be sure the seals are properly affixed to the cooler so they cannot be removed during shipping. Additional tape across the seal may be prudent.

SHIPPING AIR BILLS

When samples are shipped from the field to the testing laboratory via a commercial carrier (e.g., FedEx, UPS), the shipper provides an air bill or receipt. Upon completion of the field sampling event, the sampling team leader will be responsible for submitting the sender's copy of all shipping air bills to be copied at an Integral office. A discussion of copy distribution is provided in SOP AP-02. Note the air bill number (or tracking number) on the applicable COC forms or, alternatively, note the applicable COC form number on the air bill to enable the tracking of samples if a cooler becomes lost.

ACKNOWLEDGMENT OF SAMPLE RECEIPT FORMS

In most cases, when samples are sent to a testing laboratory, an Acknowledgment of Sample Receipt form is faxed to the project QA/QC coordinator the day the samples are received by the laboratory. The person receiving this form is responsible for reviewing it, making sure that the laboratory has received all the samples that were sent, and verifying that the correct analyses were requested. If an error is found, call the laboratory immediately, and document

any decisions made during the telephone conversation, in writing, on the Acknowledgment of Sample Receipt form. In addition, correct the COC form and fax the corrected version to the laboratory.

Submit the Acknowledgment of Sample Receipt form (and any modified COC forms) to be copied. A discussion of copy distribution is provided in SOP AP-02.

ARCHIVE RECORD FORMS

On the rare occasion that samples are archived at an Integral office, it is the responsibility of the project manager to complete an Archive Record form. This form is to be accompanied by a copy of the COC form for the samples, and will be placed in a locked file cabinet. The original COC form remains with the samples in a sealed Ziploc® bag.

STANDARD OPERATING PROCEDURE (SOP) AP-04

SAMPLE LABELING

SCOPE AND APPLICATION

This SOP describes the general Integral procedures for labeling samples, and the three kinds of labels that can be used on a project (i.e., sample labels, sample tags, and internal sample labels). Consult the project-specific sampling and analysis plan (SAP) to determine the exact sample identifiers and sample labels that are required for a given project. If they are not specified in the SAP, then follow the designations below.

SAMPLE IDENTIFIERS

Before field sampling begins, establish sample identifiers to be assigned to each sample as it is collected. Sample identifiers consist of codes designed to fulfill three purposes: 1) to identify related samples (i.e., replicates) to ensure proper data analysis and interpretation, 2) to obscure the relationships between samples so that laboratory analysis will be unbiased by presumptive similarities between samples, and 3) to track individual sample containers to ensure that the laboratory receives all material associated with a single sample. To accomplish these purposes, each container may have three different codes associated with it: the sample identifier, the sample number, and the sample tag number. These codes and their use are described as follows:

- **Sample Identification Code**—The sample identification code (Sample ID) is a unique designation that identifies where and how the sample was collected. The sample identifier is recorded in the field logbook *only* and is not provided on the sample label or chain-of-custody (COC) form. The sample identifier is a multiple-part code. The first component begins with the letter abbreviation; for example, "SWNS" or "SWNB" to designate the surface water sample was collected from the near-surface or near-bottom of the water column. The second part could identify the sampling event; for example, "1" to designate Round 1 sampling. The third part could contain an abbreviation for whether the station is a single point (SP), a transect (TR), a composite (CO), or a vertically integrated station (VI). The station number would be the final component of the sample identifier. Use leading zeros for stations with numbers below 100 for ease of data management and correct data sorting.

  If appropriate, add a supplemental component to the sample identifier to code field

duplicate samples and splits. Use a single letter (i.e., a suffix of "A" and "B") to indicate field duplicates or splits in the final component of the sample identifiers. For equipment decontamination blanks, assign sequential numbers starting at 900 instead of station numbers. Use a sample type code that corresponds to the sample type for which the decontamination blank was collected. Additional codes may be adopted, if necessary, to reflect sampling equipment requirements (see project-specific SAP).

Examples of sample IDs are as follows:

- SWNS-1-SP-002: Surface water sample collected from the near-surface at a single point during Round 1 from Station 2.
- SWNB-1-TR-010-A: Duplicate surface water sample from the near-bottom transect during Round 1 from Station 10.

- **Sample Number**—The sample number is an arbitrary number assigned to each distinct sample or split that is shipped to the laboratory for separate analysis. The sample number appears on the sample containers and the COC forms. Each sample will be assigned a unique sample number. All aliquots of a composited field sample will have the same sample number. In cases where samples consist of multiple bottles from the same location, assign each bottle the same sample number and time. However, assign replicates from the same location different sample numbers and times. Sample numbers of related field replicates will not necessarily have any shared content.

  Each field split of a single sample will also have a different sample number and time. The sample number is generally a unique six-digit number that includes a two-digit media code and a four-digit number. The media code may be site-specific, but the Integral default codes are as follows:

  - SS—Surface soil
  - BH—Subsurface soil or rock (typically from borehole)
  - GW—Groundwater
  - SW—Surface water
  - PW—Pore water
  - SD—Sediment
  - BT—Biota or biological tissue

  The exact sample numbering scheme may vary from project to project. Variances in the sample numbering scheme will be described in the project-specific SAP for the field event. Example sample numbers are PW0001, PW0002, PW0003, etc.

- **Tag Number**—Attach a different tag number to each sample container. If the amount of material (i.e., everything associated with a single sample number) is too large for a single container, assign each container the same sample number and a different sample tag. A sample will also be split between containers if a different preservation technique is used for each container (i.e., because different analyses will be conducted).

  The sample tag number is a unique five- or six-digit number assigned to each sample label (or "tag") for multiple bottles per sample. Integral sample labels come with a preprinted sample tag number. The tag number provides a unique tracking number to a specific sample bottle. This allows for greater flexibility in tracking sample bottles and assists in field quality control when filling out documentation and shipping. Sample tags are not used by many other consultants, and there may be resistance from such firms during teaming situations. However, experience has shown that tags can be very valuable, both in the field and while processing data from field efforts.

  Record tag numbers on the COC form. Laboratories use tag numbers only to confirm that they have received all of the containers that were filled and shipped. Data are reported by sample number.

Assign sample numbers sequentially in the field; sample labels are preprinted with sequential tag numbers.

SAMPLE LABELS

Integral sample labels are designed to uniquely identify each individual sample container that is collected during a sampling event. Field sampling teams are provided with preprinted sample labels, which must be affixed to each sample container used. Fill out the labels at the time the samples are collected, documenting the following information:

- Sample number
- Site name or project number
- Date and time sample is collected
- Initials of the samplers
- Preservatives used, if any
- A unique number (commonly referred to as the "Tag Number") that is preprinted on the label consisting of five or six digits; used to identify individual containers.

SAMPLE TAGS

Integral sample tags are designed to be affixed to each container that is used for a sample. Sample tags are required only for environmental samples collected in certain U.S.

Environmental Protection Agency (EPA) regions (e.g., EPA Region 5). Field crews are provided with preprinted sample tags. Attach sample tags to each individual sample container with a rubber band or wire through a reinforced hole in the tag. Mark all sample tag entries with indelible ink. Fill out the tags at the time the samples are collected, documenting the following information:

- Sample number
- Site name or project number
- Date and time sample is collected
- Initials of the samplers
- Preservatives used, if any
- Type of analysis.

A space for the laboratory sample number (provided by the laboratory at log-in) will also be provided on the sample tag.

INTERNAL SAMPLE LABELS

For benthic infaunal samples, wash away the sediment from the sample and collect the remaining benthic infauna into a sample container. Affix sample label (as discussed above) to the outside of the sample container. In addition, place an internal sample label inside the sample container. This internal sample label is made of waterproof paper; be sure to make all internal sample label entries with pencil. Fill out the internal sample labels at the time the samples are collected, documenting the following information:

- Sample number
- Site name or project number
- Date and time sample is collected
- Initials of the samplers
- Preservative used (e.g., formalin).

STANDARD OPERATING PROCEDURE (SOP) AP-05

INVESTIGATION-DERIVED WASTE HANDLING

SCOPE AND APPLICATION

This SOP presents the method to be used for handling wastes generated during field sampling activities that could be hazardous. These wastes are referred to as investigation-derived waste and are subject to specific regulations.

All disposable materials used for sample collection and processing, such as paper towels and gloves, are not considered investigation-derived wastes and will be placed in heavyweight garbage bags or other appropriate containers. Disposable supplies will be removed from the site by sampling personnel and placed in a normal refuse container for disposal at a solid waste landfill.

EQUIPMENT AND REAGENTS REQUIRED

- 55-gallon drums (or appropriately sized waste container)
- Paint markers
- Tools (to open and close drum)
- Ziploc® bags
- Drum labels.

PROCEDURES

1. Place solid wastes that need to be containerized in properly labeled, DOT- approved, 55-gallon drums.
2. Properly close, seal, label, and stage all filled or partially filled drums before demobilization. Properly profile full drums and have them shipped off site to a RCRA Subtitle C facility.

3. Sampling activities generate personal protective equipment and miscellaneous debris that require disposal. Remove gross contamination from these items, and place the items in plastic bags. It is acceptable to store these items in plastic bags as an interim measure. At the end of each day, dispose of the bags at an appropriate solid waste facility dumpster.

STANDARD OPERATING PROCEDURE (SOP) AP-06

NAVIGATION AND STATION POSITIONING

SCOPE AND APPLICATION

This SOP describes procedures for accurate station positioning required to ensure quality and consistency in collecting samples and in data interpretation and analysis. Station positioning must be both absolutely accurate in that it correctly defines a position by latitude and longitude, and relatively accurate in that the position must be repeatable, allowing field crew to reoccupy a station location in the future (e.g., for long-term monitoring programs).

This SOP describes the most commonly used station positioning method, differential global positioning system (DGPS). Integral uses a Trimble Pathfinder™ Pro XRS DGPS for station positioning for many field efforts. The Pro XRS offers the submeter accuracy often required for documenting sampling station locations and for re-locating previously sampled stations. A comprehensive discussion of the Trimble Pathfinder™ Pro XRS DGPS is provided in Attachments 1, 2, and 3 of this SOP.

SUMMARY OF METHOD

Global positioning system (GPS) navigation is used to position the sampler at the desired location. GPS is a satellite-based system that receives positioning data at 1-second intervals from multiple satellites at known positions in space. Standard GPS is calculated to an accuracy of about 10 m.

One can obtain a higher accuracy of approximately 2 m by applying differential corrections to the standard GPS positioning data using DGPS. These differential corrections are applied by sending GPS differential corrections to the GPS receiver via radio transmission. If the sampling location is near the coastal U.S, the U.S. Coast Guard generates differential corrections that are transmitted via radio link to the GPS receiver. If a Coast Guard station is out of range of the sampling area, then a receiver may be set up at a known (i.e., surveyed) reference point on land, or real-time satellite differential signals can be purchased from a private company (e.g., OmniSTAR).

With the Pro XRS, GPS data can be gathered to submeter accuracy using a choice of differential correction sources (i.e., free beacon differential signals such as Coast Guard beacons or OmniSTAR) without establishing a reference station. Data must be corrected to gain submeter accuracy. Free beacon or base station signals allow differential corrections to be

performed after data collection by using a nearby beacon or base station logging data files. (Note: The station must be within 300 miles of the data collection location.) For satellite-based signals, a built-in virtual base station allows for real-time data correction, eliminating the need for post-processing data in some cases. However, postprocessing data corrections can obtain accuracies in the range of 30–50 cm. These accuracies are for the horizontal (northing and easting) component only. The vertical component (elevation) accuracy ranges from submeter to 3 times larger than the horizontal accuracy.

The GPS receiver displays and transmits differentially corrected positioning data to the computer using an integrated navigation software package (e.g., HYPACK, Terrasync). The computer data are typically displayed and recorded in World Geodetic System of 1984 (WGS-1984) geographic coordinates (latitude/longitude). However, the integrated navigation system can display and record information in other datums (e.g., UTM, NAD83). The integrated navigation system, acting as a data manager, displays the sampler's position relative to a target station location in plan view on a video screen. The resulting pictorial screen presentation, as well as numeric navigation data (e.g., range and bearing to the target sampling location) assists the vessel operator (when sampling on-water) in approaching and maintaining the station position while sampling.

SUPPLIES AND EQUIPMENT

- Cable
- GPS antenna
- Telemetry antenna (for differential corrections)
- GPS receiver
- Differential corrections receiver
- Computer and monitor
- Navigation software (e.g., Terrasync)
- Logbook or log sheets.

PROCEDURES

Obtain latitude and longitude coordinates at the locations where samples are collected. An average positioning objective is to accurately determine and record the positions of all sampling locations to within 2 m. Positioning accuracies on the order of 1–3 m can be achieved by avoiding the few minutes per day when the satellites are not providing the same level of signal. The GPS provides the operator with a listing of the time intervals during the

day when accuracies are decreased. Avoiding these times allows for better positioning accuracy.

On-Land Sampling Event

A backpack DGPS unit may be used to direct the sampling team to the proposed sampling location. To expedite field activities, enter the target station coordinates into the navigation system database prior to beginning sampling. Place the DGPS antenna as close as possible to where the sampling will occur. Once the sample(s) have been collected at the appropriate location, record the horizontal coordinates of the station in the field logbook.

On-Water Sampling Event

Mount the GPS antenna vertically at the outboard end of the vessel's boom, with the GPS antenna cable extended along the boom into the cabin. Mount the telemetry antenna for receiving differential corrections on a convenient fixture outside the cabin. Locate the GPS receiver, the differential corrections receiver, and the computer in the cabin. Orient the video screen for the computer to allow the vessel operator to observe on-screen positioning data from the helm.

Alternatively, use a backpack DGPS unit to position the sampling vessel (e.g., barge) over a proposed sampling location. Place the DGPS beacon as close as possible to where the drilling will occur (i.e., moon pool). Using the DGPS unit, direct the sampling vessel operator to the sample station location.

Once the sampling vessel is anchored at the appropriate location, record the horizontal coordinates of the station in the field logbook. To expedite field activities, enter the target station coordinates in the navigation system database prior to beginning sampling.

Positioning System Verification

GPS requires no calibration, as all signal propagation is controlled by the U.S. government (the Department of Defense for satellite signals and the U.S. Coast Guard for differential corrections). Verifying the accuracy of the GPS requires coordinates to be known for one (or more) horizontal control point within the study area. The GPS position reading at any given station can then be compared to the known control point. Verify the GPS accuracy at the beginning and end of each sampling day.

Station Positioning Activities

Use a consistent routine for each day's positioning activities. After confirming successful reception of differential signals, turn on the computer on, and the boot the software. Verify the accuracy of the system at a horizontal control point, as described in the previous section.

The sampling team proceeds to a target station location selected by the team leader. That station location is then selected from a number of preselected station locations that have been entered into the integrated navigation system database. Once the station has been selected, the positioning data are displayed on the computer screen or hand-held unit to assist in proceeding to the station and in maintaining the station position during sampling. A confirmed position is recorded electronically each time a sample collection is attempted. (This means that during sediment grab sampling and coring, the locations of both accepted and rejected grabs or cores are recorded.) Upon recovery of the sampling device, read the station position northing (y) and easting (x) coordinates from the archived computer file and record them in the field logbook or on log sheets as a backup to the computer record. Also record time and water depth, if applicable. Ancillary information recorded in the field logbook may include personnel operating the GPS, tidal phase, type of sampling activity, and time when coordinates were collected.

REFERENCES

Trimble Navigation Limited. 2001. TSC1 Asset Surveyor operation manual. Version 5.20. http://trl.trimble.com/dscgi/ds.py/Get/File-8145/Oper.pdf.

Trimble Navigation Limited. 2007. GPS tutorial. Accessed on January 12, 2007. http://www.trimble.com/gps/index.shtml.

ATTACHMENT 1
PRO XRS DESCRIPTION

The Pro XRS combines a high-performance GPS receiver and antenna, beacon differential receiver, and satellite differential receiver in one compact unit. It also includes Trimble's advanced Everest™ technology, which allows users to collect accurate position data near walls, water, vehicles, or other surfaces that reflect satellite signals. Reflected signals, also called multipath signals, make it difficult for GPS receivers to accurately determine position. Everest™ uses a patented technique to remove multipath signals before measurements are used to calculate position.

Equipment Required

The GPS Pathfinder™ Pro XRS consists of the following:

- GPS receiver in backpack casing (with system batteries and cables)
- Hand-held data logger (TSC1) and cable, *or* laptop computer with Terrasync software installed and cable. (Note: Terrasync procedures are described under separate cover.)
- Pro XRS antenna, range poles, and cable
- Compass and tape measure
- Spare 12-volt camcorder and 9-volt batteries (minimum of two each) (use only Kodak, Duracell, or Energizer 9-volt batteries)
- Battery charger and power cord.

Pro XRS Setup

Follow these procedures for the proper setup of the Pro XRS:

1. Ensure that connections between batteries, receiver, and data logger are correct and secure. The coaxial antenna cable connects from the GPS receiver port "ANT" to the base of the antenna. The TSC1 cable (a "pig-tail"-type cable) connects from the bottom or top of the TSC1 to the receiver port "B," where a 9-pin serial port dongle is attached. The dual Y-clip cables should be connected from the receiver to the batteries. Alternatively, if AC power is available (e.g., aboard a vessel), then the power cable for the battery charger can be attached directly to the receiver on some models.
2. Screw the three long antenna poles together (the shorter pole may be added if necessary for taller users). Screw on the antenna and connect its cable.
3. Put backpack and/or shoulder strap on. The pouch for the data logger should be in place around the waist strap or in the backpack.

4. Screw antenna to the attachments on the top of the backpack. Wind cord around pole, and ensure the antenna is secure. Please be aware of overhead hazards, especially if working near low-hanging power lines. Severe injury or death can result.

Basic Operation of the Pro XRS

Recording a Feature

Before beginning field use, ensure that all GPS configurations and settings are set correctly for the particular use of the Pro XRS and that an appropriate data dictionary is loaded onto the TSC1 (see Attachments 2 and 3 for typical settings). These steps outline the basic use of the GPS to document a sample position or any other defined "feature." Note that the TSC1 has both hard and soft keys that allow for its operation. The hard keys comprise all of the keys (e.g., letters and numbers) on its surface. The soft keys are the F1 through F5 hard keys. The function of these changes depending upon the context. These keys will be referred to with brackets around them (<soft-key>).

1. Turn data logger on outside in an open area. Wait for antenna to receive satellite signals. The display will read Recording Almanac, Too Few SVs, and PDOP Too High. Continue to wait until enough satellites (four) are acquired and the PDOP is below 5.0.
2. Ensure that the real-time settings are correct according to the parameters listed in Attachment 2.
3. Select **Data Collection**, and create a new rover file or open an existing file. This file should be named according to the format specified by the project GIS analyst. Note: If opening an existing file, press **<NEW>** to access the *Antenna Options* menu and *Start Feature* menu.
4. Enter the height of the antenna from the ground to the *Measurement Method* reference point shown in the *Antenna Options* menu and then press **ENTER** to bring up the *Start Feature* menu.
5. Pick the appropriate data dictionary to use with the rover file. Only one dictionary can be used with a rover file. Consult with the project GIS analyst to formulate the most appropriate data dictionary for the type of sampling you wish to perform. The data dictionary titled *Generic* contains only a comment field and is appropriate for simple navigation tasks. If using a data dictionary, make sure to become familiar with its attributes before recording information in the field.

6. Move to the location of the first feature for which you want to record the GPS position. Select the appropriate feature and press **ENTER** to begin logging. Log data points in accordance with the feature type. Point features should have at least 10 points collected at a stationary location. Line features should be collected while moving. If movement is stopped, press the **<PAUSE>** key. When movement starts again, press the **<RESUME>** key. Area features should be collected with enough points to define the outline of the area (e.g., a square building would have four single points, collected on each corner, and the **<PAUSE>** key would be used between each of the points).
7. Depending on the setup of the data dictionary, each feature may have one or more feature attributes. An attribute is used to record additional data associated with the feature. For example, the attributes assigned to a sediment sampling station could be the sample number, station ID, sampling gear, sediment color, odor, etc.
8. Use the **<PAUSE>** key while recording feature attributes to avoid too many data points being collected at one point feature. (Body movements while logging attributes for an extended time can decrease the accuracy of collection.) The **<PAUSE>** key must be used when recording attributes of a line or area feature because only one data point should be collected in a single location.
9. Once all attributes are entered and the feature data points are logged, press **ENTER** to complete and save the feature and move on to a new feature. Pressing **ESC** instead of **ENTER** will allow the user to abandon the logged feature without saving.
10. When all features in a given area have been recorded, from the *Data Collection* menu, press **ESC** to exit data capture and then press **<YES>** to close the file. Features are appended and saved to the file after each collection, so there is no need to "save" the file. When the Pro XRS is not in use, it should be turned off. If you need to come back to the same rover file later in the day, the rover file may be reopened at that time. Rover files may not be edited after 7 days from the first feature was created. Please consult the project GIS analyst for the best way to handle multi-week sampling projects.
11. At the end of each day, download the rover file to a PC using Pathfinder Office software.

Feature Collection Options

Offsets—The Pro XRS can collect a point or line feature while standing at a set distance away from the feature. This option may be necessary because of obstructions such as tree cover, buildings, or car traffic. For a point feature, measure the distance between the object you want recorded and the Pro XRS antenna. Use the compass to determine the bearing (e.g., west is 270°). The bearing is the direction the point should be moved for it to be located in the correct place (e.g., if you are due north of the feature, the bearing is south, or 180°; i.e., the position you want recorded is south of where you are standing). Estimate the inclination from the

feature to the GPS antenna (if altitude determination is critical, a clinometer should be used). The inclination is the degree angle up from the feature to the antenna (e.g., if the feature is 5° below the antenna position, enter −5°). During data capture, from within the feature, press the **<OFFSET>** button, and enter the distance, bearing, and inclination. Press **OK** to complete the feature. Note: This procedure describes an offset of a single feature. A constant offset may be applied to all features collected as well.

Nesting—While recording a line feature or an area feature, a point feature may be collected to avoid backtracking. While recording the line or area feature, press **<PAUSE>** and then **<NEST>**. The Pro XRS will prompt for collection of a new feature. Move to the feature, and collect data as for any other point feature. When the feature is complete, press **OK**. The Pro XRS is ready to resume collecting data as part of the line/area feature: press **<RESUME>**. (Remember to continue moving before pressing resume to avoid having multiple positions recorded in the same place in the line or area feature.)

Segmenting—While moving along a line feature, changing the attributes of that line may be necessary (e.g., because of a change in surface type from paved to dirt road). This change may be done without having to begin a new feature by pressing **<PAUSE>** and then ****. Change the appropriate attributes and then press **<RESUME>** to continue recording.

Repeat—This function allows the collection of a new feature with the same feature attributes as the previous feature. If features are not exactly the same, it also allows editing of the attributes.

Quickmark—Allows collection of point features while moving (e.g., from a car or a boat) by estimating the exact location. The use of this feature will not result in positionally accurate locations and is not recommended for most sampling operations.

Reviewing and Editing Features

It is possible to review or edit features collected in the field while still in the data capture mode. For example, it may be necessary to document the GPS location in the field logbook or to edit one of the feature's attributes. Without exiting data capture, press **<REVIEW>**. (If data capture is already complete, just press **<REVIEW>** and then select the appropriate rover file.) This step will display a list of data points including each feature collected. Scroll to the appropriate feature, and follow the steps below depending on the required action:

- To view the GPS location (e.g., lat/lon), press **<POS>**.
- To edit the attributes, press **ENTER**. Make any necessary edits to the attributes by scrolling through.
- To change or add an offset, press **<POS>** and then **<OFFSET>**. Make any necessary changes.
- To delete a feature collected in error, press **<DEL>**.

Navigating to an Existing Location

Waypoints

To use the Pro XRS to navigate to a previously established position, this position must be loaded into the data logger as a waypoint, present as a feature position in the data files, or generated in the field using the GPS unit. Waypoints may be entered into the TSC1 by:

- Entering coordinates manually
- Choosing previously recorded locations and importing them into the TSC1 by using Pathfinder Office
- Defining a location stored in a rover file saved to the TSC1 as a waypoint (see *Reviewing/Editing Features*, above)
- Creating a way point from the current position being shown by the operating GPS unit in the field.

Navigating

Usually you will use the *Navigation* module (accessed by pressing **MENU** followed by **Navigation**) to guide yourself to a target (waypoint or feature). You can also use the *Map* module (accessed by pressing **MENU** followed by **Map**) to:

1. Orient yourself in the area where you are working.
2. Get a general indication of the location of a feature or waypoint that you want to find.
3. Find or select features or waypoints to which you wish to navigate toward.
4. Plot a course from one place to another.
   a. While in the Map screen, the GPS cursor x shows the current position reported by the receiver and is always shown on the Map screen (Note: it may not always be within the visible part of the screen when panning or scrolling). The **<OPTIONS>** key can be used to hide or display the GPS trail (line of dots showing up to 60 previous positions), the heading showing the direction of travel, and other options on the map display.
   b. Select a feature by pressing **MENU**, Data Collection to reach the *Start Feature* screen, and then **<REVIEW>** to access all features contained in the data file. Highlight and select the desired feature by pressing the **<Target>** key, which adds a crossed flag to the feature. Reaccess the *Map* screen by selecting **MENU**, then **Map**, which will now show the highlighted feature with a crossed flag symbol on the Map screen. You can then start moving toward the feature, and the current position (shown by the x) will move closer to the target position as the user approaches.

c. There are two graphical modes of navigation with the Pro XRS in the TSC1 *Navigation* module. On both modes, text information appears on the right of the screen in the *Info* panels, which can be configured by the user. The graphical modes available are the *Directional Dial* screen or the *Road* screen, which can be toggled between using the **<Mode>** key.

d. To navigate, select a target and then a start position. Each of these positions can be features from an open data file or a waypoint. Access a list of available features or waypoints by pressing **<TARGET>** or **<START>**. Once the item has been chosen as a target, it will show the crossed flags symbol in the list. Once a target has been selected, *Distance to Go* appears at the bottom of the *Navigation* screen, which indicates the distance from the current GPS position to the target. Select a start position (not required but useful for calculating crosstrack error and other navigation information) by pressing **<START>**. A waypoint of the current GPS position can be created for use as the Start point by selecting **<CREATE>**. Once the Start position is selected, a flag symbol will appear next to the item in the list.

e. In the *Directional Dial* mode, an arrow will appear that will always point at the target. This is the bearing to go. (Note: You need to be moving for this to be accurate, as it will lock if you are moving too slowly or have stopped.) The triangle at the top represents the direction that you are going or heading. This triangle never moves, but by changing directions, you can line up the arrow with the triangle. When the two are aligned, you are heading in the direction of the target. When you are close to the target, a bull's-eye (two concentric circles) will appear at the edge of the screen. This is warning you that the unit will be switching to the close up screen. A proximity alarm will sound and the directional arrow will be replaced by the bull's-eye on the close up screen. Your current position will be shown by an x and the target by the bull's-eye. Move so that the x is in the same location as the bull's-eye.

f. In the *Road* mode, navigate by walking down a road. Your position is shown by a stick figure and is always positioned in the center of the screen. The target (crossed flags) shows the point to which you are navigating toward. Your heading is shown by the top center of the screen and the bearing to go is shown by the direction of the road, which will rotate as you change your heading. Change your heading until the road is pointing at the top of the screen (*Target* is also at the top of the screen) and the edges are parallel to the sides of the screen. As you move toward the target the screen zooms in, so the road appears to get wider.

Downloading Rover Files

Upon returning to the office, download all rover files from the TSC1 to a PC for post-processing. You will need the Trimble Pathfinder software installed on your computer. If you

are not using a field laptop that already has the program installed, contact your project GIS analyst for instructions on how to install the software.

Connect the TSC1 to your computer using the appropriate cables. In addition to the "pigtail" cable, you will also need a null modem (a 9-pin female-to-female cable) to plug into a PC serial port. Once connected, power up the TSC1 unit and navigate to *MENU>File Manager>File Transfer*. Then, open the Pathfinder software and navigate to the *Utilities>Data Transfer...* window from the menu bar. Select **GIS Datalogger** on COM1 (for most computer systems), and press the green **Connect** button. Download files from the TSC1 by selecting the **Receive** tab and choosing the data file type from the *Add* pulldown menu (Figure 1).

After downloading, remove all rover files and waypoints from the TSC1 to conserve memory. Rover files may be deleted from the *File Manager* menu as follows:

1. Select **MENU>File Manager>Delete File(s)**
2. Select the rover file to be deleted, and press **<ENTER>**
3. Confirm the deletion of this file by pressing **<YES>**.

Delete data dictionaries in the same manner by selecting **Data Dictionaries** from the *File Manager* menu. Delete waypoints by selecting **Utilities** from the *Main* menu and then by selecting **Waypoints,** followed by **<DEL>**.

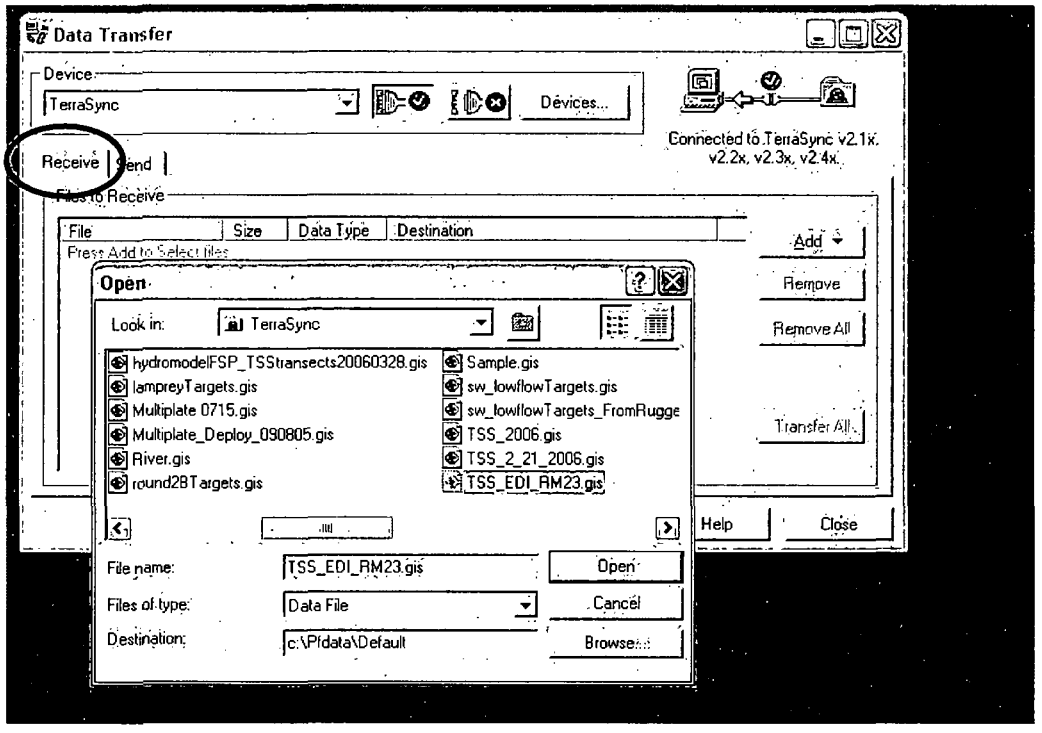


Figure 1. Transferring File from Terrasync

ATTACHMENT 2
TSC1 SETTINGS

The following are lists of menus that can be accessed through the TSC1 keypad. Please ensure that settings are correct before proceeding. Do not make changes to the settings unless necessary. Each menu will list all available subheadings, the correct setting, and the available **<soft-keys>** to access additional menus. Comments are included only where necessary.

GPS Rover Options

To access this menu, select **Configuration** from the main menu and then select **GPS Rover Options**. The table below lists logging options and settings.

Logging Options	Setting	Comment
Logging intervals		
Point feature	1s	
Line/area feature	2s–5s	depending upon speed of movement
Not in feature	None	
Velocity	None	
Confirm end feature	No	
Minimum pos	10	
Carrier Mode	Off	
Carrier phase min. time	10 minutes	
Dynamics code	Land	May be changed to sea or air, as appropriate
Audible click	Yes	
Log DOP data	Yes	
Log PPRT data	Yes	
Log QA/QC data	Yes	
Allow GPS update	Warn First	
Warning Distance	Any	
Position Mode Manual	3D	
Elevation Mask	15°	Should not go below 13° (accuracy decreases)
SNR Mask	6.0	Can raise to 7 if multi-path filtering is poor
PDOP Mask	5.0	Can be raised up to 8; reduces accuracy
PDOP Switch	6.0	

Real-Time Input Options

Access this menu from the GPS Rover *Options* menu by selecting **Real-Time Input**. The table below shows options and settings for real-time input.

Options	Setting	Comment
Preferred Correction Source		
	Choice 1	Integrated Beacon
	Choice 2	Integrated WAAS
	Choice 3	Use uncorrected GPS
Correction Age Limit	20s	

Antenna Options

Access this menu from the GPS rover *Options* menu by selecting **Antenna Options**. The table below shows antenna options and settings.

Option	Setting	Comment
Height	6 ft	Enter correct user antenna height using measurement method indicated below
Measure	Uncorrected	
Type	Integrated GPS/Beacon/Satellite	
Confirm	Per file	Can be changed to "Per feature" if antenna height varies and elevation is critical
Part Number	33580-50	Auto selected based on TYPE selected
Measurement	Bottom of Antenna	
Method	Mount	

ATTACHMENT 3
ADDITIONAL SETTINGS FOR THE TSC1

Additional TSC1 settings can be found in the *Configuration* menu. Items of particular importance are indicated in italics.

Configuration

This menu can be accessed by selecting **Configuration** from the main menu. The table below lists options and descriptions for the *Configuration* menu.

Options	Description
GPS base station options	For using a land base station or beacon for real time corrections
NMEA/TSIP output	Consult manual
Coordinate system	Changes coordinate system among latitude/longitude, UTM, and other coordinate systems. System can be converted, if necessary, after data capture by using Pathfinder Office software.
Map Display options	Change layers, scale, background files and items shown on the TSC1 screen during data collection
Navigation options	Changes Navigation parameters
Units and display	Changes various units, for example: length (e.g., feet, meters), altitude reference (e.g., MSL), *North reference* (i.e., true or magnetic). Units can be converted, if necessary, after data capture by using Pathfinder Office software.
Time and date	Changes to *local time*, 24-hour clock, date format, and other options
Quickmarks	Set-up parameters for use with Quickmarks.
Constant offset	Set-up parameters for use with a constant offset.
External sensors	Connections with external sensors.
Hardware (TSC1)	TSC1 settings such as beep volume, contrast, *internal and external battery status*, software version, free space.

Contrast and Backlighting

The TSC1 display can be viewed in various light settings. Press **FUNC**, then **L** to turn on the display backlight for viewing in dim lighting. Adjust the contrast by pressing **FUNC**, then **E** or **F**.

ATTACHMENT 4
PRE-SAMPLING ACTIVITIES BEFORE USE OF THE PRO XRS

Determination of Optimal Satellite-Use Time

Positioning accuracies on the order of ±1 to 3 m can be achieved by avoiding the few minutes per day when the satellites are not providing the same level of signal. The GPS unit provides the operator with a listing of the time intervals during the day when accuracies are decreased. Avoiding these time intervals permits the operator to maintain better positioning accuracy.

ATTACHMENT 5
MANAGING GPS DATA FROM TERRASYNC—A TUTORIAL

Currently, positional data collected in the field is most often done with a Trimble GPS unit (usually rented) interfaced with a laptop via Trimble's Terrasync software. The Terrasync software sometimes exhibits quirks that interfere with the smooth operation of data collection in otherwise stressful field conditions. This tutorial is meant to supplement the Terrasync software documentation and serve as a guide to field personnel to help them retrieve and collect geographic data as efficiently as possible with existing software.

Scope

This document is intended to be a reference for procedures involving the following:

- Fixing files that are more than 7 days old so that they can be updated
- Adding features in GPS Pathfinder software (companion to Terrasync) and then importing them as base files in Terrasync..

This document is not intended to be a comprehensive manual for using Terrasync or Pathfinder software. It is assumed that the reader has received at least some training on how to use the basic features of Terrasync and is competent at using MS Windows.

The Basics

GPS data collection currently relies on two pieces of complementary software:

- Terrasync—the interface for GPS navigation and data collection.
- Pathfinder Office—a multiuse piece of software that acts as a conduit between GIS data files (shape files) and Terrasync GPS files. Pathfinder can also be used as a simple map editor.

Installing the Correct Versions of Terrasync and Pathfinder

Important Note: This tutorial uses Pathfinder Office v. 3.00 and Terrasync v. 2.50. It is very important to use the proper versions of this software to avoid compatibility issues. These software versions should be included in the same folder as this tutorial, or can be obtained from GIS staff.

http://www.trimble.com/terrasync_ts.asp?Nav=Collection-4576

Key code for TerraSync
499043-00110-05273-EDD049BC

Pathfinder v.3.00
001533-00300-04152-0ee4d11f

Initial Setup of Terrasync/Pathfinder

Certain settings and configuration setups are needed before Pathfinder can talk to Terrasync. Whether you are installing this software for the first time or have an existing installation, check to make sure that these settings are in place.

1. Open Pathfinder Office and go to the *Utilities>Data Transfer...* menu. A dialog box should appear. This is the interface for communicating with Terrasync.
2. Click the **Devices** button, and then **New...** (Figure 1).
3. Click on **GIS Folder**.
4. Browse to the Terrasync data folder on your computer, which in most cases will be *C:\My Documents\TerraSync*.
5. In the next box, *Type* will be **Terrasync**, and *Version* will be **v. 2.1x, v.2.2x, v.2.3x,** and **v2.4x**.
6. At the prompt for a name that will display in the device list, enter **Terrasync**.
7. Go back to the Data Transfer dialog box, select **Terrasync** from the dropdown menu, press the **Connect** icon, and look for a green check mark indicating success.

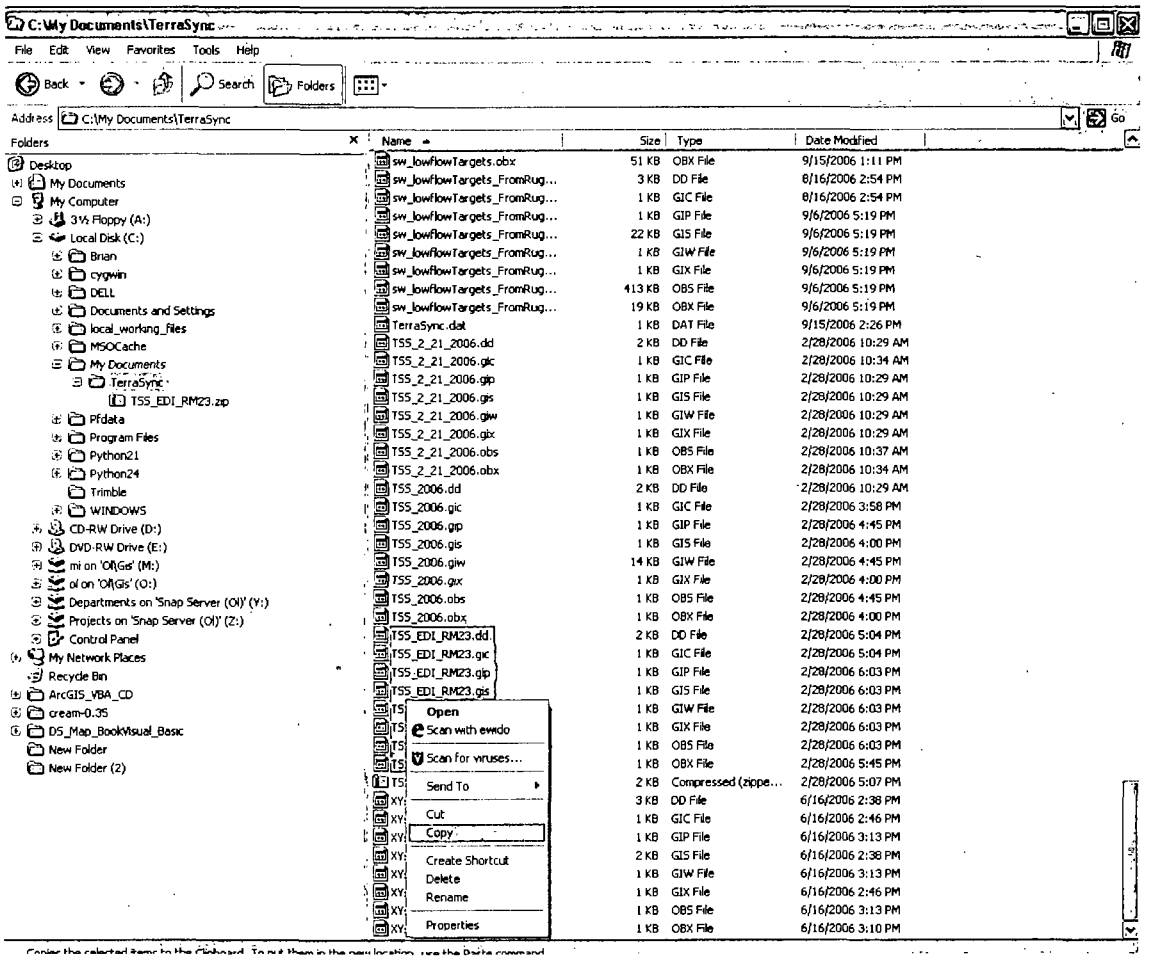


Figure 2. Selecting Files To Copy to a Different Directory

If this procedure does not work for you, you may have the wrong version of Pathfinder. For some unknown reason, with each version upgrade of Pathfinder, connectivity to older versions of Terrasync is lost. You can check what version of Pathfinder you have installed by going to the *Help>About GPS Pathfinder Office...* menu. To find out what version of Terrasync you have, go to *C:\Program Files\TerraSync*, right-click on **Terrasync.exe**, and choose the **Version** tab.

Handling Expired Files in Terrasync

One of the most common problems that field personnel will have to deal with is the 1-week expiration date when trying to collect data with Terrasync. This is a built-in function of Terrasync, and there is no simple way to work around it. The following instructions will guide you through the process to make the files usable. See Figure 3.

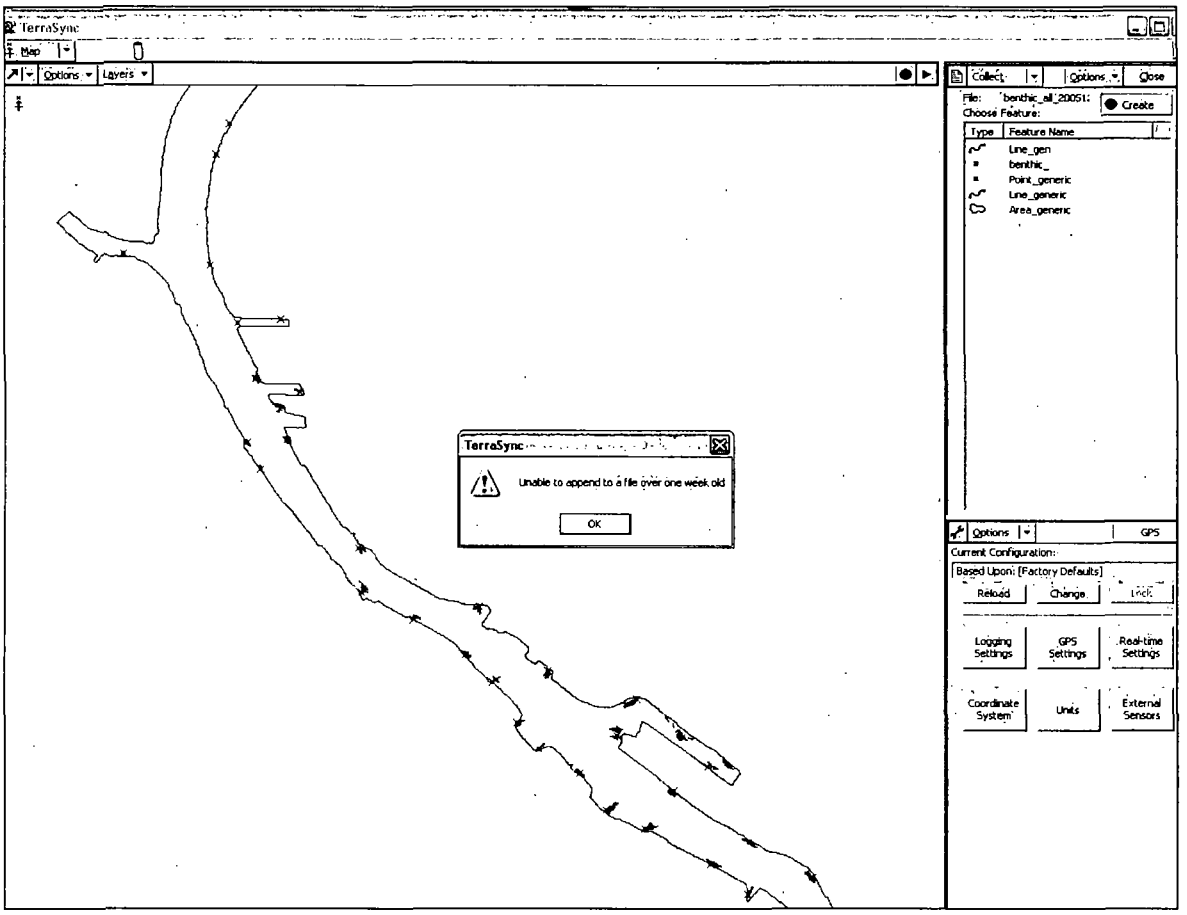


Figure 3. Notice That Terrasync File Older Than 1 Week Will Not Allow User To Collect Features (time begins to elapse when first feature is collected in the field, not when file is created)

Two options are available, depending on your needs. If you do not need to see the previously logged locations and need only to see the targets, use the original files provided by GIS staff (Option 1). If you need to see previously occupied locations in order to make decisions about where to go next, then transfer the file to Pathfinder and back again (Option 2).

Option 1: Move and replace logged files with original targets.

At the beginning of the field effort, you should receive a set of files with the target locations, most likely in a zip archive (.zip file extension). There will be six to eight files with the same name but with different extensions (Figure 4). These files will have to go into the *C:\My Documents\TerraSync* folder in order to be available to Terrasync.

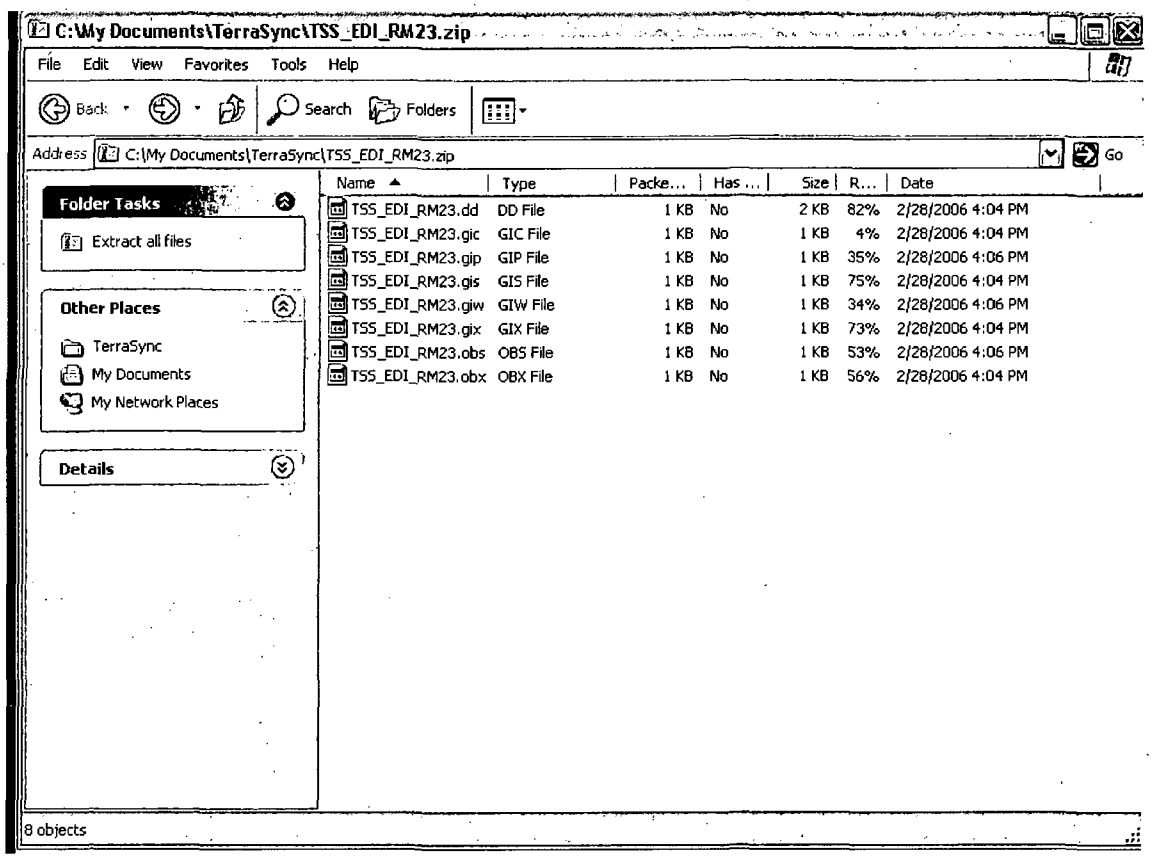


Figure 4. Example of File Set To Be Unzipped into the Terrasync Folder

After you unzip these files to Terrasync, keep this zip archive around in an easy-to-find place, such as your computer desktop, because the 1-week clock does not start until you begin collecting your first point in the field. You can use this unadulterated file again, as long as you make a copy of the work you did the previous week. The detailed steps are as follows:

1. Make sure you have the original files with the target locations available in a handy place. This will probably be the original zip archive. Also, be sure to close Terrasync while performing this process.
2. Navigate to C:\My Documents\TerraSync\ in Windows Explorer. Locate the files that you have been using the previous week. Note: It is crucial to get all of the small files associated with the data set. While it is useful to sort the files by date modified, you can miss some of the small files—it is highly recommended that you sort the files alphabetically.

3. Copy all of these files to a different directory, preferably one that is named appropriately to reflect the data and time period that you were collecting. For example: C:\Documents and Settings\bpointer\Desktop\lampreyTargets_20060925. These files contain the data you have collected the previous week and should be backed up and/or emailed to the appropriate project manager or GIS staff.
4. You can now safely replace the files you just copied with the ones from the original zip file. Right-click the zip archive, and click Extract All. When prompted to Select a folder to extract files to, browse to C:\My Documents\TerraSync. (Figure 5). If prompted about replacing existing files, select Yes to All. Note: It is crucial to make copies of the files first (see Step 3 above)—otherwise, you may lose the data.
5. You should now be able to open the file in Terrasync and begin logging as normal.

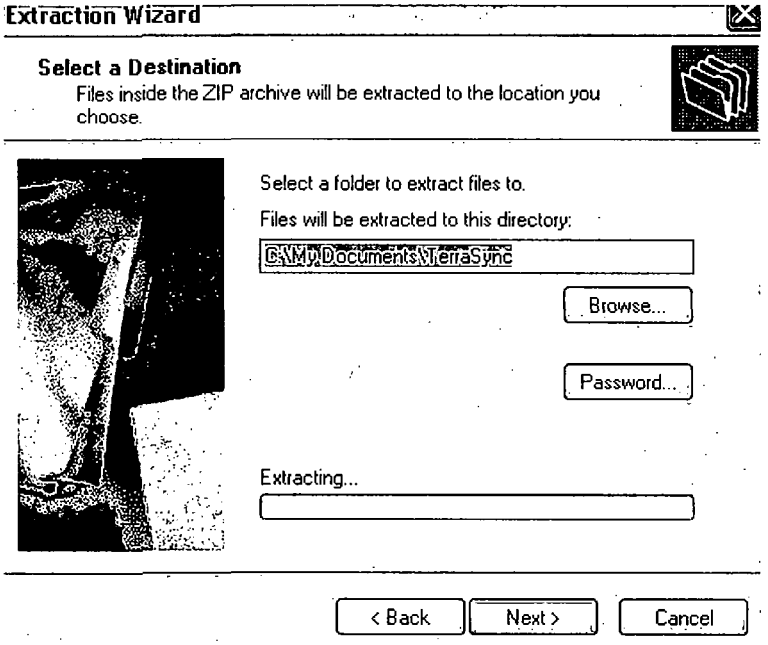


Figure 5. Extract (or copy) Original Target Files into the Terrasync Directory

Option 2: Transfer files back and forth from Terrasync.

If you need to be able to see the previously occupied positions from last week while positioning this week, you need to use Pathfinder to reset the file. This process will essentially combine the targets and actuals from last week into one file. However, this method has its drawbacks; once converted, the actuals from last week will not be able to be corrected, so a backup procedure similar to the one in the previous option should be carried out to maintain data integrity.

The steps for file transfer are as follows:

1. For good data management, back up the data files from the previous week using the procedure laid out in steps 1 through 3 in Option 1 above.
2. Close Terrasync and open up Pathfinder Office.
3. Go to the Utilities>Data Transfer menu or just click the icon on the left (Figure 6).
4. Ensure that the device listed is Terrasync. If not, follow the initial setup instructions at the beginning of this document. Most of the computers used for GPS logging are already setup for this.
5. There are two tabs, Receive and Send. Make sure that Receive is selected and then go to Add>Data File. Select the file(s) that you are using and select Open. The file should now be in the Files to Receive box. Click Transfer All and wait for the transfer to take place. If you have made the recommended backups, it is fine to replace any files.
6. Now select the Send tab (Figure 7), and go to Add>Data File. Select the file you just transferred (it will have the same name as the Terrasync file) and click Open. Now click Transfer All to move the file back to Terrasync.

   By transferring the file back and forth from Terrasync to Pathfinder, you have "reset the clock" and can now update the file for an additional 7 days. This file will have your targets and actual positions from the last week, so it is important to be aware of the features you are selecting for navigation.

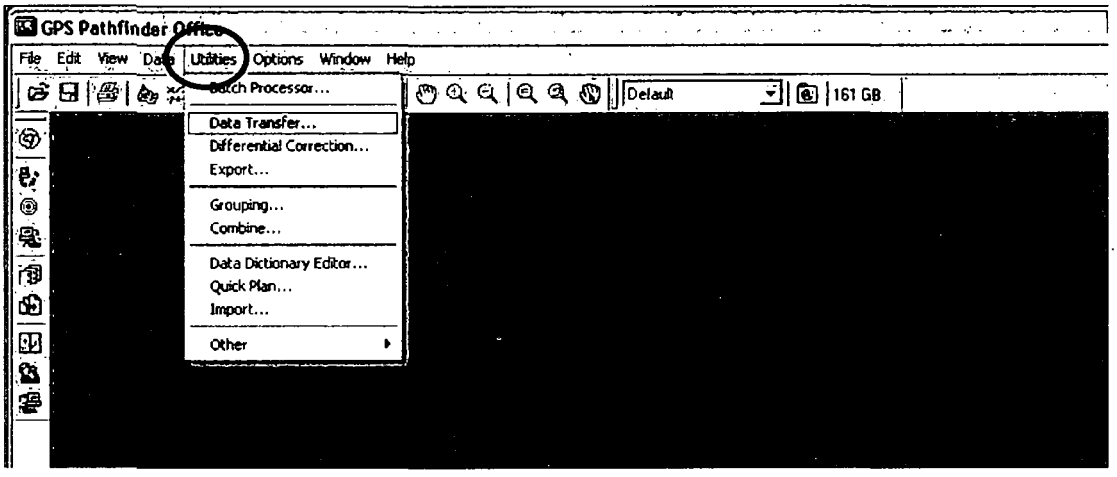


Figure 6. Data Transfer Menu

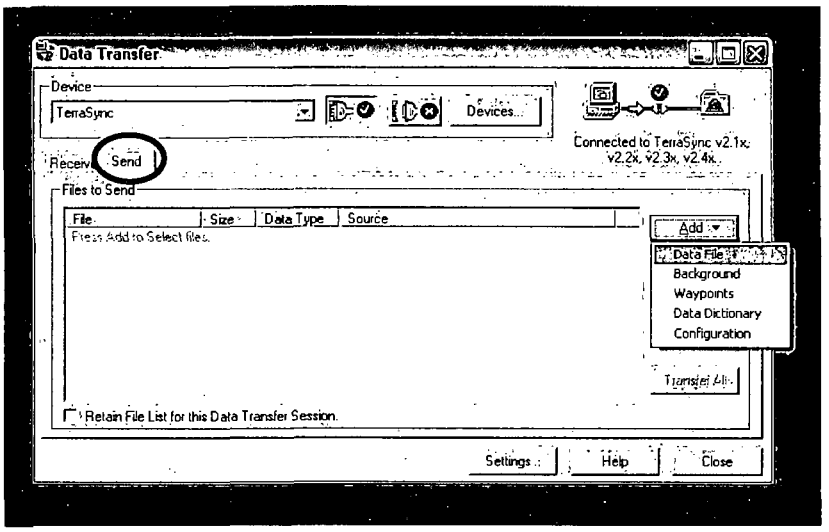


Figure 7. Sending Data File

STANDARD OPERATING PROCEDURE (SOP) SD-02

PREPARATION OF FIELD QUALITY CONTROL SAMPLES FOR SEDIMENTS

SCOPE AND APPLICATION

This SOP describes the purpose, preparation, and collection frequency of field duplicate samples, field replicate samples, matrix spike/matrix spike duplicates, equipment rinsate blanks, bottle blanks, trip blanks, temperature blanks, environmental blanks, and reference materials (i.e., a standard reference material, a certified reference material, or other reference material; for the purposes herein, all types of reference materials are referred to as standard reference material, or SRM) for sediment sampling efforts. Not all of the field quality control samples discussed in this SOP may be required for a given project. The specific field quality control samples will be identified in the project-specific field sampling plan (FSP) and quality assurance project plan (QAPP). For most projects, Integral's recommended field quality control samples are an equipment rinsate blank, a field duplicate, and trip blanks if samples are to be analyzed for volatile organic compounds (VOCs). Definitions of all potential quality control samples are described below.

As part of the quality assurance/quality control (QA/QC) program, all field quality control samples will be sent to the laboratories "blind." To accomplish this, field quality control samples will be prepared and labeled in the same manner as regular samples, with each quality control sample being assigned a unique sample number that is consistent with the numbering for regular samples. All of the containers with preservatives that are required to complete the field quality control sample for the applicable analyte list shall be labeled with the same sample number. The sample ID for field quality control samples should allow data management and data validation staff to identify them as such and should be recorded only in the field logbook. Under no circumstances should the laboratory be allowed to use reference materials, rinsate blanks, or trip blanks for laboratory quality control analysis (i.e., duplicates, matrix spike, and matrix spike duplicates). To prevent such an occurrence, regular samples should be selected and marked on the chain-of-custody/sampling analysis request (COC/SAR) form or the laboratory should be instructed to contact the project QA/QC coordinator to select appropriate samples for each sample group.

Field quality control samples will be prepared at least once per sampling event, and certain types will be prepared more often at predetermined frequencies. If the number of samples taken does not equal an integer multiple of the intervals specified in this SOP, the number of

field quality control samples is specified by the next higher multiple. For example, if a frequency of 1 quality control sample per 20 is indicated and 28 samples are collected, 2 quality control samples will be prepared. Field quality control samples for sediment sampling activities should be prepared consistent with the requirements discussed below and at the frequency indicated unless different frequency requirements are listed in the FSP and QAPP.

The following table lists the quality control sample types and suggested frequencies for sediment sampling programs. Because sediment quality control sampling may require assessment of site cross-contamination, additional blanks may be required. A detailed explanation of each quality control sample type with the required preparation follows.

Table 1. Field Quality Control Sample Requirements

Quality Control Sample Name	Abbreviation	Preparation		Frequency[a]
		Location	Method	
Duplicate	DUP	Sampling site	Additional natural sample	One per 20 samples. May not be applicable if REP is being collected.
Replicate	REP	Sampling site	Additional natural sample	One replicate per 20 samples. May not be applicable if DUP is being collected.
Matrix spike/matrix spike duplicate	MS/MSD	Sampling site	Additional sample bottles filled for laboratory quality control requirements	One per 20 samples.
Equipment rinsate blank	ER	Sampling site	Deionized water collected after pouring through and over decontaminated equipment	Minimum of one per sampling event per type of sampling equipment used and then 1 per 20 thereafter.
Bottle blank	BB	Field	Unopened bottle	One per sample episode or one per bottle type.
Trip blank	TB	Laboratory	Deionized water with preservative	One pair per each VOC sample cooler shipment.
Temperature blank	TMB	Laboratory	Deionized water	One per sample cooler.
Environmental blank	EB	Field	Bottle filled at sample site with deionized water	One per 20 samples.
Standard reference material	SRM	Field laboratory or sampling site	SRM ampules or other containers for each analyte group	One set per 50 samples or one per episode.

[a] Frequencies provided here are general recommendations; specific frequencies should be provided in the project-specific FSP or QAPP.

FIELD DUPLICATE SAMPLES

Field duplicate (or split) samples are collected to assess the homogeneity of the samples collected in the field and the precision of the sampling process. Field duplicates will be prepared by collecting two aliquots for the sample and submitting them for analysis as separate samples. Field duplicates will be collected at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. The actual number of field duplicate samples collected during a sampling event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of field duplicate collection may vary by EPA region or state).

FIELD REPLICATE SAMPLES

Field replicate samples are co-located samples collected in an identical manner over a minimum period of time to provide a measure of the field and laboratory variance, including variance resulting from sample heterogeneity. Field replicates will be prepared by collecting two completely separate samples from the same station and submitting them for analysis as separate samples. Field replicates will be collected at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. If field duplicate samples are collected, then it is unlikely that field replicate samples will also be collected during a sampling event. The actual number of field replicate samples collected during a sampling event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of field duplicate collection may vary by EPA region or state).

MATRIX SPIKE/MATRIX SPIKE DUPLICATES

The matrix spike/matrix spike duplicate (MS/MSD) analyses provide information about the effect of the sample matrix on the design and measurement methodology used by the laboratory. To account for the additional volume needed by the laboratory to perform the analyses, extra sample volumes may be required to be collected from designated sediment stations. MS/MSDs may be collected at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. The actual number of extra bottles collected during a sampling event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements may vary by analyte group).

EQUIPMENT RINSATE BLANKS

Equipment rinsate blanks will be used to help identify possible contamination from the sampling environment and/or from decontaminated sampling equipment. Equipment rinsate blanks will be prepared by pouring laboratory distilled/deionized water through, over, and into the decontaminated sample collection equipment, and then transferring the water to the appropriate sample containers and adding any necessary preservatives. Equipment rinsate blanks will be prepared for all inorganic, organic, and conventional analytes at least once per sampling event per the type of sampling equipment used. The actual number of equipment rinsate blanks prepared during an event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of equipment rinsate blank collection may vary by EPA region or state).

BOTTLE BLANKS

The bottle blank is an unopened sample bottle. Bottle blanks are submitted along with sediment samples to ensure that contaminants are not originating from the bottles themselves because of improper preparation, handling, or cleaning techniques. If required, one bottle blank per lot of prepared bottles will be submitted for analysis. If more than one type of bottle will be used in the sampling (e.g., high-density polyethylene or glass), then a bottle blank should be submitted for each type of bottle and preservative. The actual number of bottle blanks analyzed during a project will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP as the requirements on frequency of bottle blank analysis may vary by EPA region or state).

To prepare a bottle blank in the field, set aside one unopened sample bottle from each bottle lot sent from the testing laboratory. Label the bottle as "Bottle Blank" on the sample label (and in the "Remarks" column on the COC/SAR form), and send the empty bottle to the laboratory with the field samples.

TRIP BLANKS

Trip blanks will be used to help identify whether contaminants may have been introduced during the shipment of the sediment samples from the field to the laboratory for VOC analyses only. Trip blanks are prepared at the testing laboratory by pouring distilled/ deionized water into two 40-mL VOC vials and tightly closing the lids. Each vial will be inverted and tapped lightly to ensure no air bubbles exist.

The trip blanks will be transported unopened to and from the field in the cooler with the VOC samples. A trip blank is labeled and placed inside the cooler that contains newly collected VOC samples and it remains in the cooler at all times. A trip blank must accompany samples

at all times in the field. One trip blank (consisting of a pair of VOC vials) will be sent with each cooler of samples shipped to the testing laboratory for VOC analysis.

TEMPERATURE BLANKS

Temperature blanks will be used by the laboratory to verify the temperature of the samples upon receipt at the testing laboratory. Temperature blanks will be prepared at the testing laboratory by pouring distilled/deionized water into a vial and tightly closing the lid. The blanks will be transported unopened to and from the field in the cooler with the sample containers. A temperature blank shall be included with each sample cooler shipped to the testing laboratory.

FIELD BLANKS

The field blank is prepared in the field to evaluate potential background concentrations present in the air and in the distilled/deionized water used for the final decontamination rinse. If unpreserved bottles are to be used, then the appropriate preservative (i.e., for metals samples use a 10 percent nitric acid solution to bring sample pH to 2 or less) must be added, as may be required. Field blanks should be collected at a minimum frequency of 1 in 20 samples. The actual number of field blanks analyzed during a project will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of field blank analysis may vary by EPA region or state).

To prepare a field blank in the field, open the laboratory-prepared sample bottle while at a sample collection site, fill the sample bottle with distilled/deionized water, and then seal it. Assign the field blank a unique sample number, label the bottle, and then send the bottle to the laboratory with the field samples.

REFERENCE MATERIALS

SRMs are samples containing known analytes at known concentrations that have been prepared by and obtained from EPA-approved sources. The SRMs have undergone multi-laboratory analyses using a standard method that provides certified concentrations. When available for a specific analyte, SRMs provide a measure of analytical performance and/or analytical method bias (i.e., accuracy) of the laboratory. Several SRMs may be required to cover all analytical parameters. For all analytes where available, one SRM will be analyzed at a frequency of one per 50 samples. The actual number of SRMs analyzed during a project will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of SRM analysis may vary by EPA region or state).

STANDARD OPERATING PROCEDURE (SOP) SD-08

SUBSURFACE SEDIMENT CORE COLLECTION USING A VIBRACORER

SCOPE AND APPLICATION

This SOP describes the procedure for collecting and processing sediment core samples using a vibracore system, which collects continuous and relatively undisturbed sediment cores. This method of sediment coring is performed from a boat and uses high-frequency low-amplitude vibration to break down the frictional resistance of the sediment and allow the core tube to penetrate into the sediment with minimal distortion. It is best used for sampling coarse, consolidated sediment and very cohesive sediment, where static weight (e.g., piston-type or conventional gravity corers) will not produce adequate penetration into the sediment. In addition, the vibracorer offers a high rate of production, superior retention of shallow samples, and a greater sample volume compared to conventional drilling equipment.

Vibracorers generally consist of a metal corer barrel (usually a 4-in.-outside-diameter, aluminum core barrel) with a location-dedicated polycarbonate or Lexan®-lined core tube, and a vibrator mechanism attached to the top of the barrel. The vibration is created either by an electric motor, a hydraulic system, or a pneumatic piston attached to the top of the barrel. Therefore, a generator or air compressor is needed on board to power the corer. The pneumatic piston does not have the same function as a piston in a piston corer. Because vibracorers generally do not have a piston in the corer, some compaction and/or bypass will occur, and recovery will be less than 100 percent.

A continuous sediment sample is retained within the tubing with the aid of a stainless-steel core cutter/catcher or nosecone attached to the bottom of each aluminum tube.

It is always best to keep the core in a vertical position to prevent the top layers of sediment (i.e., the top 5 to 15 cm) from slumping. However, in many cases, it is not feasible to process the core in a vertical position because the tripod needs to be at least twice the height of corer, and sectioning and logging the sample would have to be performed from a ladder. For studies that specify sectioning the sample into coarse intervals (>20 cm), processing the core in a horizontal position will generally not significantly disrupt the stratigraphy. For studies that specify shorter intervals (<5–10 cm), processing the core in a horizontal position is likely to disrupt stratigraphy. In this case, the top layers of sediment that have high water content should be sectioned while the core is in a vertical position, and when the sediment becomes thicker, the corer can be laid horizontally.

PROCEDURES

Decontamination

To prevent potential cross-contamination of samples, all reusable sediment sampling equipment must be decontaminated prior to use at each station and between field replicates.

Before each station is sampled, decontaminate the inner surfaces of the corer or core tube liner and all stainless-steel sample compositing equipment. Prior to sampling, all core liners will be washed in sequence with a standard detergent (e.g., Alconox®), rinsed with site water, and then air-dried. During storage and transport, decontaminated core liners will be capped at both ends to prevent contamination. Details on correct decontamination procedures can be found in SOP SD-01, *Decontamination of Equipment—Sediment*. The project-specific field sampling plan (FSP) should also be consulted to determine any project-specific decontamination procedures. The personnel performing the decontamination procedures will wear protective clothing as specified in the site-specific health and safety plan.

All solvent rinsates (if used) will be collected into a bucket or tub and allowed to evaporate over the course of the day. Any rinsate that has not evaporated by the end of the sampling event will be containerized and disposed of in accordance with applicable regulations.

Vibracorer Deployment and Retrieval

The following procedures are based on using the vibracorer aboard a boat equipped with a tripod or A-frame of sufficient height to allow recovery of the core (see project-specific FSP for information on target coring depth), and a power winch. On pontoon boats, the tripod is centered over a hole in the floor, whereas on other boats, the corer may be lowered over the side or stern. To obtain cores of high quality, the boat must be anchored with at least three anchors so the boat will not drift during the coring process.

1. Maneuver the sampling vessel to the targeted sampling location using the positioning procedures and minimum water depth restrictions.
2. Deploy 3- or 4-point anchor system to maintain position; record and monitor position throughout core acquisition.
3. Once on location, measure the water depth (depth to top of sediment) using the onboard depth sounder (fathometer) or lead line and record measured depth in the field logbook. If the water level is affected by tides, obtain tide level measurements and calculate tidal height in feet above mean low water. The date, time, weather, and water conditions (e.g., high wave activity, strong currents, turbidity, tidal flux) should also be recorded in the field logbook.

4. Assemble the decontaminated core tube, liner, core catcher, and cutter heads (or nose cone depending on the model of vibracorer used), using care to not contact decontaminated surfaces. Attach assembled vibracorer to winch cable. Note that several decontaminated catchers and cutterheads will be on hand, in case of loss. Core catchers and cutter heads can be decontaminated and reused for subsequent core collection.
5. Attach a tape measure to the vibracorer or mark the winch cable in 1/2-ft increments to measure penetration depth.
6. Inspect connections of winch cable and electrical or pneumatic lines to confirm they are secure.
7. Signal the winch operator to slowly raise the vibracorer into a vertical position and guide the vibracorer (with core liner, valve, core catcher, and cutterhead in place) overboard until it is clear of the vessel.
8. Using the winch, slowly lower the vibracorer through the water column at a speed of about 1 ft/s to avoid creating a bow wake or overturning of the vibracore. Stop lowering the corer a few feet above the sediment and confirm that the boat has not drifted.
9. Continue lowering the vibracorer until the tip of the core is resting on the sediment or to the depth recorded by the fathometer, depending on the consistency of the sediment. Record the vibracorer depth as derived from the attached tape measure or marked winch cable. Measurements will serve as a basis for determining penetration depth.
10. Resume lowering the corer at about 1 ft/s. When the nosecone or core catcher contacts the sediment, turn on the vibracorer motor. The vibracorer is then allowed to slowly penetrate the sediments. Initially, light tension should be maintained on the cable to keep the corer from tipping over.
11. Lower the vibracorer to the target penetration depth as measured by the attached tape measure or marked winch cable. If the targeted penetration depth is met, proceed to the next step; if refusal is met, retrieve the vibracorer, perform gross decontamination (i.e., rinse with river water and brush off visible sediment from the outside of the core barrel) and re-attempt at new location offset at least 3–5 ft from original location.
12. When the target penetration depth is reached, or refusal occurs, turn off the vibracorer and record the time, penetration depth, angle of the cable relative to the boat, and any other observations.
13. The vibracorer is slowly withdrawn from the sediments at a constant rate, to keep it upright and not dislodge any sediment from within the core barrel, and raised to the surface.

14. With the corer hanging in a vertical position, clean the vibracorer assembly by hosing down the equipment with site water prior to being brought on board. If the corer is not plugged, care should be taken not to direct water into the open end of the core barrel.
15. After collection of the core sample, the vibracorer is slowly guided onboard the vessel; use care to avoid jostling that might disturb the integrity of the core. Care must be taken to keep the top end of core elevated to prevent sediment from "pouring" out. Use a sawhorse or equivalent to elevate the top of the core. If necessary, as soon as the nosecone clears the water surface, the bottom of the corer may be plugged with a rubber stopper to prevent loss of sediment.
16. Before the polycarbonate or Lexan®-lined core tube is removed from the vibracorer, the nosecone or core cutter/catcher is visually inspected to ensure that proper penetration has been attained and that there is no obvious loss of sediment from the tube. Any presence of noticeable odors, the core penetration depth, and physical characteristics (e.g., color, texture, odor) of the sediment sample as observed at the ends of the tube will be recorded in the field logbook or on the Field Sediment Core form by an experienced geologist. In addition, any sheen in the water will also be noted in the field logbook.
17. If the core will be processed horizontally, slowly lay the corer down. Unscrew the cutter head (or nosecone) and carefully remove the core catcher, while retaining as much sediment as possible.
18. While removing the core catcher (or nosecone), be ready to immediately seal the end of the core liner by placing a plastic cap over the open end.
19. Carefully remove the core liner that contains the sample by lifting the lower end from the deck as needed to provide clearance. Affix core cap, wrap with tape, label core liner and end of core, remove valve from top of core liner, stand core upright, and place in a processing rack or tray to allow the sediment at the top of the core to settle. Avoid sudden movements to the core that would disrupt the sediment interface.
20. While waiting for sediment to settle, prepare the Field Sediment Core form. Identify any debris and note its depth in the core and what the debris is, if possible.
21. Once resuspended sediment has settled, measure the length of the recovered core, calculate percent recovery (100 x recovered length/penetration depth), and record in the logbook or on the Field Sediment Core form.
22. Check the core for acceptability. The following acceptability criteria should be satisfied:
    - The core tube is not overfilled with sample so that the sediment surface presses against the bottom of the vibracorer head.
    - Overlying water is present (indicates minimal leakage).

- The overlying water is not excessively turbid (indicates minimal disturbance).
- The desired penetration depth (see project-specific FSP for required penetration depth) or refusal has been reached.

Depending on requirements of the project-specific FSP, a core may be rejected based on percent recovery. Commonly, a core is deemed unacceptable if recovery is less than 80 percent. If recovery is less than 80 percent, the core sample will be retained for possible processing, while additional sampling attempts are made to collect a core with greater than 80 percent recovery. If subsequent attempts result in recoveries of less than 80 percent, then the sample with the highest percent recovery may be used for analysis. The number of attempts to collect an acceptable sample will be specified in the project-specific FSP. If recovery is less than 80 percent, the core may be acceptable if the penetration depth is deeper than the target core length. In this case, the recovered length should be equal to the target length.

23. Once sufficient time has been allowed for the sediment to settle (i.e., no sediment is suspended in the overlying water), use a decontaminated saw to cut a drain-slit or a decontaminated drill bit to drill in the side of the core liner approximately 1 to 2 in. above the sediment–water interface; allow excess water to drain. Cut excess polycarbonate liner with decontaminated blade and use a siphon to decant off the overlying water. Ensure that the saw blade, drill bit, or siphon does not contact the sediments and that fine-grained suspended sediment is not removed.
24. Cut cores into manageable sections (3–4 ft) aboard the vessel immediately after their retrieval. Cap each section with aluminum foil and plastic caps, and seal with duct tape. Mark the core with permanent marker using a unique number or alphanumeric code identifying sampling location, core number, core section, and segment orientation (i.e., which end is up). Following sectioning, store the cores in an upright position onboard the vessel in a core box and have them transported periodically throughout each field day by small boat to a field processing area where they are to be stored upright under custody on ice or refrigerated at 4°C to await processing.
25. In preparation for next core, thoroughly rinse the interior of the core barrel until all loose sediment has been washed off. Repeat process at next sampling location. Continue coring until requirements are met.

In situations where there is significant surface water depth and/or water current that could cause the vibracorer setup to lean at an unacceptable angle, a buoyant frame or rigid frame configuration should be used.

With the buoyant frame, the vibracorer is maintained in proper vertical position by two guidelines held taut between a float package and a weight stand. The larger weight stand is provided with ballast boxes so that easy-to-find ballasting material such as lead bags or scrap

metal can be used in the field. For deployment, the vibracorer is lowered with the weight stand hanging on its guidelines from the vibrahead. The float package is hooked up to the guidelines when the vibrahead reaches the deck level.

After coring and pull-up, the system is retrieved in the reverse manner. In case of limited deck space or overhead clearance, or to further accelerate the procedure on the water, the weight stand can be left in as overboard cradle.

Sample Handling, Storage, and Processing

Cores should be processed concurrently with core collection, and every effort should be made to ensure cores are processed within 24 hours of collection. Cores awaiting processing will be sealed tightly at both ends and stored upright in ice or in a refrigerator. If core collection outpaces processing such that significant delays in core processing appear likely, core collection will be suspended to allow the core processing to catch up.

As mentioned above, once coring has been completed at a given location, the cores will be transported in an upright position on ice to a designated field processing area, where they will be logged and processed. The field processing area will be equipped with a core-cutting table, core-processing tables, a decontamination area, and a storage area with appropriate refrigeration. Appropriate lighting will be installed in the field processing area so that consistent, high quality photographs can be taken of the opened cores. Care should be taken to create a field processing area that minimizes the potential for outside contamination.

Sample processing includes removing the sample from the liner, recording observations of sample characteristics, mixing subsamples, and distributing the sample to containers for shipping to the testing laboratory. Vibracore processing most often consists of the following steps:

1. Cut each core tube along the long axis using decontaminated hook blade. Rotate the tube 180° and cut again.
2. After each core is cut, move the entire core tube to an aluminum foil-covered table and open it so that it can be systematically logged, described, and photographed.

However, depending on the project-specific FSP, the core may be extruded from the liner and cut into the specified intervals as it emerges or the core liner may be cut into sections, sealed, and shipped intact to the testing laboratory.

Core Observations

1. Verify that the length of the core, water depth, and all required position data have been recorded in the field logbook together with all pertinent observations and communications with the field team leader.

2. After each core is cut open, describe the sediment on a Field Sediment Core form in the field processing area notebook. When recording the information for each core, follow the guidelines below:
   - Physical sediment description (i.e., sediment type [e.g., silt, sand], density/consistency, color)
   - Odor (e.g., hydrogen sulfide, petroleum, creosote)
   - Visual stratification, laminations, and lenses
   - Presence/location/thickness of the redox potential discontinuity layer (a visual indication of black is often adequate for documenting anoxia)
   - Approximate percentage of moisture
   - Vegetation
   - Approximate percentage of vegetation
   - Debris
   - Approximate percentage of debris
   - Presence of biological structures (e.g., detritus, shells, tubes, bioturbation, live or dead organisms, chironomids)
   - Approximate percentage of biological structures
   - Presence of a sheen
   - Other distinguishing characteristics or features.

   The visual observations of sediment lithology (dominant grain sizes) will be the primary criteria for determining sample intervals (i.e., lithologic units) in the cores. For consistency, core descriptions and terms used will follow the criteria below, which are modified from methods presented in ASTM D 2488-00 (ASTM 2000):

3. Record visual estimates of the grain-size percentages of sediment units within each core on the Field Sediment Core form so that the total sum will add up to 100 percent. Make estimates of gravel, sand, and fines (silt and clay) content generally to the nearest quartiles:
   - 0 to 25 percent
   - >25 to 50 percent
   - >50 to 75 percent
   - >75 to 100 percent.

   If appropriate, describe the sediment narratively on the log based on the estimated grain-size percentages. Use the dominant constituent grain size as the primary unit

descriptor, and describe the abundance of other grain sizes present using the following terms:

- The grain-size adjective (e.g., gravelly, sandy, silty, or clayey), if estimated to constitute more than 25 percent of the sediment
- *With,* for example, sand with silt, silt with sand, etc., if estimated to constitute less than 25 percent of the sediment
- *Trace,* if estimated at less than 5 percent of the sediment (and not included in the total 100 percent).

For other features observed, such as organic matter or debris, use the following additional descriptive terms as appropriate:

- *Mostly,* if estimated to constitute 50 percent or more of the unit
- *Some,* if estimated to constitute more than 25 to 50 percent of the unit
- *Little,* if estimated to be 25 percent of the unit or less
- *Trace,* if estimated at less than 5 percent (and not included in the total 100 percent).

4. Describe density using the following terms:
   - *Loose,* if easily penetrated with a sampling spoon
   - *Dense,* if penetration is more difficult.
5. Describe consistency using the following terms:
   - *Very soft,* if present as an ooze that holds no shape
   - *Soft,* if saggy
   - *Stiff,* if it holds a shape
   - *Very stiff,* if penetration with a spoon is low
   - *Hard,* if no penetration with a spoon is possible.
6. Use other observations (e.g., obvious anthropogenic material, dramatic color changes) to define or help define sample intervals (check project-specific FSP for sample interval definition; depending upon the project-specific requirements the sample interval could be based on lithology or it could be set to a specific interval [e.g., 1 ft]).
7. Determine the boundaries of lithologic units primarily by changes in the top two dominant grain sizes estimated visually (e.g., a change from a silty sand to a gravelly sand or to a sandy silt).

8. Photograph the cores after they have been described and before any sediment is removed for processing. It is important for each core section to be photographed with adequate lighting from a standard measured distance from the core. Digital photographs will be used later in the production of digital core logs.

Mixing and Sample Preparation

1. After the sample is characterized and the core observation logged on the Field Sediment Core form, remove the specified sample interval using a stainless-steel spatula or spoon (see project-specific FSP for correct sampling interval). Exercise care to not include sediment that is in direct contact with the core tube. With the approval of the field team leader, and using a decontaminated stainless-steel instrument, carefully remove unrepresentative material (e.g., large shells, stones). Exercise care not to touch the sediment during this process. Note any unrepresentative material removed from the sample in the field processing area notebook.
2. Remove subsamples for analysis of unstable constituents (e.g., volatile organic compounds, acid- volatile sulfides), and place them directly into sample containers without homogenization. Completely fill the sample container so that there is no headspace or entrapped bubbles.
3. Transfer the remainder of the sample interval to a decontaminated stainless-steel bowl for homogenization. If additional sediment volume is required to fill all sample bottles (see project-specific FSP) and multiple cores need to be collected at a given station, cover the compositing bowl covered with aluminum foil (dull side down) to prevent sample contamination (e.g., from precipitation, engine exhaust, splashing water) and place in a cool dark place until the next core from that location is processed.
4. After all the sediment is transferred to the compositing bowl, homogenize the contents of the bowl using stainless-steel spoons until the texture and color of the sediment appears to be uniform.
5. Distribute subsamples to the various containers specified in the project-specific FSP and preserve the samples as specified in the project-specific FSP. Briefly stir the sediment in the compositing bowl between each spoon transfer to the sample containers.
6. After all subsamples have been placed in the sample containers, if it is suspected that there is clay in a sample, perform a "ribbon test." Perform a separate ribbon test for each interval within the core where clay is suspected to be present. To perform this test, remove a small piece of sediment from the sampled interval using a decontaminated stainless-steel spoon and roll it between the fingers while wearing protective gloves. If the piece easily rolls into a ribbon it is clay; if it breaks apart, it is silt. Note this information in the field processing area notebook.
7. Subsequent intervals should be processed in the same way.

Field Quality Control Samples

If additional volumes of sediment are required to perform all analyses including quality control analyses, an additional core may need to be collected from the same location and subsampled and homogenized accordingly. Details on collection of field quality control samples (e.g., field duplicates) will be specified in the project-specific FSP. Details on collection of field quality control samples and preparation of the certified reference materials can be found in SOP SD-02, *Preparation of Field Quality Control Samples—Sediment,* and SOP SD-03, *Preparation of Reference Materials—Sediment.* Not all of the field quality control samples discussed in this SOP may be required for a given project. The specific field quality control samples will be described in the project-specific FSP and quality assurance project plan.

Field Measurements

A water depth measurement must be collected at every sampling location. Depending on the specific project objectives, it may be necessary to perform field measurements of the *in situ* environment. Possible field measurements include temperature and pH of the sediment at the sediment-water interface and concentration of dissolved oxygen, salinity, or conductivity in the overlying water. Details on collection of field measurements can be found in SOP SD-11, *Field Analyses for Sediment.* The specific field measurements, if any, will be specified in the project-specific FSP.

Station Location Coordinates

Station locations for all field sampling will be determined using a differential global positioning system (DGPS) or by surveying. The accuracy to which the latitude and longitude of a station location is determined will be specified in the FSP. At a minimum, a DGPS capable of providing latitude and longitude coordinates with an accuracy of approximately 3 m is recommended. The DGPS consists of two satellite receivers linked to each other by a VHF telemetry radio system. The receiver will be on the sampling vessel. Details on collection of very accurate station coordinates can be found in SOP AP-06, *Navigation.*

Sample Custody and Shipping

Sample custody will be maintained in accordance with procedures outlined in SOP AP-03, *Sample Custody.* All samples will be packaged and shipped with other samples in accordance with procedures outlined in SOP AP-01, *Sample Packaging and Shipping.*

Troubleshooting

Insufficient Sample

The corer may not collect enough sediment because of 1) inadequate penetration, 2) adequate penetration but poor recovery due to compaction, 3) adequate penetration but poor recovery as a result of bypass, or 4) adequate penetration but loss of sample during retrieval. Compaction and bypass are two different artifacts that are difficult to distinguish and quantify. Following is an approach to identifying the causes and remedies of insufficient sample length. Keep in mind that a combination of these causes may occur:

- **Inadequate Penetration**—Allow more vibration time at the refusal depth, or increase the vibrator frequency.
- **Poor Recovery Due to Compaction**—Compaction is the process of rearranging the sediment particles, so that less volume is occupied by pore water, which results in a shorter column of sediment in the corer than *in situ*. Compaction occurs only in clean coarse silt, sand, and gravel sediments that have a high hydraulic conductivity and are not terminally compacted *in situ*. Fine-grained cohesive sediment (i.e., low hydraulic conductivity) does not compact. The key feature of compaction is that all of the solids ahead of the nosecone are collected as the corer penetrates. So, although the calculated recovery is less than 10 percent, 100 percent of the sediment solids were recovered. Therefore, if the sample has poor recovery, is composed of clean coarse-grained materials, and there is no evidence of sediment falling out the bottom, then the sample is likely to have been compacted. Depending on the project-specific FSP, the specified sample intervals may be shortened proportional to recovery. Because compaction of the solids displaces pore water, minimal compaction is needed for cores that are intended for porewater studies, or cores that will be analyzed for substances that have low K_d values. Vibration in vibracorers is known to rearrange particles, which leads to compaction, so another type of corer may be appropriate if compaction is a problem.
- **Poor Recovery as a Result of Bypass**—Bypass is the process of pushing sediment out of the path of the nosecone/corer as it penetrates the sediment. This is caused by the friction of sediment inside the core liner making it difficult for more sediment to enter the tube. This is most pronounced in fine-grained sediments that have low hydraulic conductivity, or layers of hard and soft sediment, or long cores. The low hydraulic conductivity prevents porewater from being displaced, so compaction cannot occur. Fine-grained sediments in this context are those in which particles cannot be felt between the thumb and forefinger of an ungloved hand. These are generally "sticky" or cohesive sediments. Therefore, if a sample has poor recovery, is fine grained and cohesive, and there is no evidence of sediment falling out the bottom, then some of the sediment column has likely been bypassed.
- **Poor Recovery Because of Loss of Sample during Retrieval**—This is often diagnosed by observing some of the core falling out the bottom as the corer approaches the water surface during retrieval, or a core liner that is empty near the bottom. Sample slipping

out the bottom of the corer can be caused by a loss of suction or noncohesive sediment that does not stick to the liner wall. Depending on the specific design of the vibracorer, there are several places at which suction can be lost. These may include the valve seat, the valve assembly, the nose piece, and couplings between the barrel and extensions. To prevent loss of suction, Teflon® plumber's tape should be used on all the threaded connections, and the valve assembly should be clean. For coarse-grained sediment (e.g., clean coarse sand and gravel, and shells) that is non-cohesive and falls out the bottom of the corer, it is sometimes possible to penetrate to a lower layer that is finer grained and will effectively plug the bottom of the core. As mentioned above, core catchers may are used to retain sediment in a vibracorer, although they should not be used if the surface sediments have high water contents and are to be sectioned at less than about 2 inch intervals.

Because recovery can be an important indicator of corer performance, sediment characteristics, and sample quality, some simple tests can be performed as a diagnostic tool. Penetration of the corer can be measured by putting Velcro® tape on the outside of the corer. Velcro® tape can also be used on the inside of the liner during testing to see how far up inside the liner the sediment interface moves, how much sample slips out the bottom, and how much compaction or bypass occurs.

Notes

1. For long cores that require more than one piece of liner, squarely cut the ends of both pieces with a plastic pipe cutter, butt the ends of the two pieces of liner squarely together and tape them securely so no leaks occur. Do not use too many layers of tape or the liner will not fit into the barrel. Do not use duct tape for this process. Use a high quality tape (i.e., 3M 3750) and dry the tubes before applying.
2. Sometimes tripods are not tall enough to lift the corer so that the barrel will clear the top edge of the liner when removing the liner. To remove the liner in this case, upon unscrewing the cutter head (or nose piece), lower the cutter head (or nose piece) and liner into a pail that has a rope securely tied to the handle. While the corer is raised by the winch, lower the pail through the hole in the deck and into the water (if necessary) until the top edge of the liner clears the bottom edge of the barrel. Then lift it back onto the deck.
3. If the vibracorer does not penetrate significantly or if the cable is let out too quickly, the vibracorer will contact the bottom, tip over, and fall sideways. When this happens, the line will initially go slack, then quickly snap to the side and take up the slack. In this case, reject the core and begin again.

4. A good measure of whether the vibracorer collected the sediment-water interface is to inspect the interface for a thin layer (about 1 mm) of olive green benthic or detrital algae. Also, if the core liner is rotated back and forth gently, the top centimeter will appear to have a gelatinous response.
5. It is sometimes impossible to collect an intact interface because gas bubbles are commonly released from sediment when the corer contacts the sediment. The released gas bubbles entrain surface sediment and cause the overlying water to become turbid. If this is the case, gas bubbles in the sediment can likely be observed through the liner wall.

REFERENCES

ASTM. 2000. Standard practice for description and identification of soils (visual-manual procedure). ASTM Standard Method No. D 2488-00. In: ASTM Book of Standards, Volume 04.08. American Society for Testing and Materials, West Conshohocken, PA.

STANDARD OPERATING PROCEDURE (SOP) SD-15

SEDIMENT CORING PROCEDURES USING AN ELECTRIC IMPACT HAND CORER

SCOPE AND APPLICATION

This SOP outlines the procedure for collecting core samples from exposed sediment (i.e., beaches) with an electric impact hand corer using a new, clean dedicated core tube at each sampling station.

SUMMARY OF METHODS

Undisturbed, continuous sediment cores will be collected using an electric impact hand corer system. The electric impact hand corer uses a rotary hammer equipped with a special adapter to accommodate 3-in. diameter Lexan®-lined core tubes. The rotary hammer uses rotation-stop and impact-stop motion to release the frictional resistance of the sediment and allow the core tube to penetrate into the sediment with minimal distortion. A continuous sediment sample is retained within the tubing with the aid of a Lexan® core catcher attached to the bottom of each tube. This method is well suited for collecting continuous, undisturbed cores from exposed beach sediment.

PROCEDURES

The procedures for the electric impact hand corer operation are as follows.

1. Obtain all necessary ancillary equipment (i.e., gas tank, power generator, extension cord, Lexan®-lined cores, and core catchers).
2. Following positioning at a given sampling station, the electric impact corer is connected to a power generator via a 100-ft extension cord. Ensure that the power generator gas tank is full and that the operating procedures for the power generator have been read. The power generator must be placed away from (e.g., 100 ft) and downwind of the sampling location.

3. Affix the decontaminated core tube and catcher to the electric impact hand corer using care not to contact decontaminated surfaces.
4. Connect the extension cord to the generator and to the electric impact hand corer. Make sure impact corer is switched off.
5. Start power generator.
6. Attach a piece of tape or use a marker to the desired length to be inserted into the sediment.
7. Make sure no one is near when sampling. Operator should be wearing work gloves, safety goggles, ear protection, steel-toed boots and if necessary, a hard hat.
8. Slowly raise the impact corer to a vertical position, ensuring that the operator can comfortably and safely operate the unit while standing on firm ground. If the operator needs to extend arms above head, a platform should be provided to offset the operating height (see figure below).

9. Turn on the electric impact hand corer and slowly insert core tube to appropriate depth. Record the core depth in the field logbook or on the Field Sediment Core form as derived from the attached tape or mark.
10. If the targeted penetration depth is met (see project-specific FSP), proceed to the next step; if refusal is met, retrieve the core, remove contents, and perform gross decontamination (i.e., rinse with river water or tap water and brush off visible sediment from the outside of the core barrel, and re-attempt at new location offset at least 0.5 to 1 ft from original location.

11. Turn off the impact hand corer and record the time, penetration distance, and any other observations in the field logbook or on the Field Sediment Core form.
12. Retrieve the corer with a back and forth motion and raise it slowly at a constant rate to keep the instrument upright and not dislodge any sediment from within the core barrel.
13. Before the Lexan®-lined core tube is removed from the electric impact corer; the core catcher is visually inspected to ensure that proper penetration has been attained and that there is no obvious loss of sediment from the tube.
14. Place the core on the side of the sampling area; use care to avoid jostling the core, which might disturb the integrity of the core. Care must be taken to keep the top end of core elevated to prevent sediment from "pouring" out if overlying water is present. Use a sawhorse or equivalent to elevate the top of the core. If necessary, as soon as the core clears the surface, the bottom of the corer may be plugged with a rubber stopper to prevent loss of sediment.
15. Detach impact hand corer from core.
16. Check the core for acceptability. The following acceptability criteria should be satisfied:
    - The core tube is not overfilled with sample so that the sediment surface presses against the bottom of the impact corer head.
    - The desired penetration depth (see project-specific FSP for required penetration depth) or refusal has been reached.

    Depending on requirements of the project-specific FSP, a core may be rejected based on percent recovery. Commonly, a core is deemed unacceptable if recovery is less than 80 percent. If recovery is less than 80 percent, the core sample will be retained for possible processing, while additional sampling attempts are made to collect a core with greater than 80 percent recovery. If subsequent attempts result in recoveries of less than 80 percent, then the sample with the highest percent recovery may be used for analysis. The number of attempts to collect an acceptable sample will be specified in the project-specific FSP. If recovery is less than 80 percent, the core may be acceptable if the penetration depth is deeper than the target core length. In this case, the recovered length should be equal to the target length.
17. Affix core cap on both ends, wrap with tape, and label core liner and end of core.
18. Prepare the Field Sediment Core form (see SOP SL-06). Identify any debris and note its depth in the core and what the debris is, if possible.
19. If overlying water is present, then allow the resuspended sediment to settle, measure the length of the recovered core, calculate percent recovery (100 x recovered length/penetration depth), and record in the logbook or on the Field Sediment Core form.

20. If overlying water is present in the core, then allow sufficient time for the sediment to settle (i.e., no sediment is suspended in the overlying water), use a decontaminated saw to cut a drain-slit or a decontaminated drill bit to drill in the side of the core liner approximately 1 to 2 in. above the sediment–water interface; allow excess water to drain. Cut excess polycarbonate liner with decontaminated blade and use a siphon to decant off the overlying water. Ensure that the saw blade, drill bit, or siphon does not contact the sediments and that fine-grained suspended sediment is not removed.
21. In preparation for next core, thoroughly rinse the interior of the core barrel until all loose sediment has been washed off. Repeat process at next sampling location. Continue coring until requirements are met.

Sample Handling and Storage

Cores should be processed concurrently with core collection, and every effort should be made to ensure cores are processed within 24 hours of collection. Cores awaiting processing will be sealed tightly at both ends and stored upright in ice or in a refrigerator. If core collection outpaces processing such that significant delays in core processing appear likely, core collection will be suspended to allow the core processing to catch up.

Once coring has been completed at a given location, the cores will be transported in an upright position to a designated field processing area, where they will be logged and processed. The field processing area will be equipped with a core-cutting table, core-processing tables, a decontamination area, and a storage area with appropriate refrigeration (see figure below).

Appropriate lighting will be installed in the field processing area so that consistent, high quality photographs can be taken of the opened cores. Care should be taken to create a field processing area that minimizes the potential for outside contamination.

Sample processing includes removing the sample from the liner, recording observations of sample characteristics, mixing subsamples, and distributing the sample to containers for shipping to the testing laboratory. Electric impact core processing consists of the following steps:

1. Carefully remove the core catcher, while retaining as much sediment as possible.
2. Place core on processing table and remove contents using a plastic plunger covered in foil.
3. Extrude sediment onto clean foil at the sample processing table.

Core Observations

1. Verify that the length of the core and all required position data have been recorded in the field processing area notebook together with all pertinent observations and communications with the field team leader.
2. After each core is cut open, describe the sediment on a Field Sediment Core form in the field processing area notebook. When recording the information for each core, follow the guidelines below:
   - Physical sediment description (i.e., sediment type [e.g., silt, sand], density/consistency, color)
   - Odor (e.g., hydrogen sulfide, petroleum, creosote)
   - Visual stratification, laminations, and lenses
   - Presence/location/thickness of the redox potential discontinuity layer (a visual indication of black is often adequate for documenting anoxia)
   - Approximate percentage of moisture
   - Vegetation
   - Approximate percentage of vegetation
   - Debris
   - Approximate percentage of debris
   - Presence of biological structures (e.g., detritus, shells, tubes, bioturbation, live or dead organisms, chironomids)
   - Approximate percentage of biological structures
   - Presence of a sheen

- Other distinguishing characteristics or features.

The visual observations of sediment lithology (dominant grain sizes) will be the primary criteria for determining sample intervals (i.e., lithologic units) in the cores. For consistency, core descriptions and terms used will follow the criteria below, which are modified from methods presented in ASTM D 2488-00 (ASTM 2000):

3. Record visual estimates of the grain-size percentages of sediment units within each core on the Field Sediment Core form so that the total sum will add up to 100 percent. Make estimates of gravel, sand, and fines (silt and clay) content generally to the nearest quartiles:

   - 0 to 25 percent
   - >25 to 50 percent
   - >50 to 75 percent
   - >75 to 100 percent.

   If appropriate, describe the sediment narratively on the log based on the estimated grain-size percentages. Use the dominant constituent grain size as the primary unit descriptor, and describe the abundance of other grain sizes present using the following terms:

   - The grain-size adjective (e.g., gravelly, sandy, silty, or clayey), if estimated to constitute more than 25 percent of the sediment
   - *With*, for example, sand with silt, silt with sand, etc., if estimated to constitute less than 25 percent of the sediment
   - *Trace*, if estimated at less than 5 percent of the sediment (and not included in the total 100 percent).

   For other features observed, such as organic matter or debris, use the following additional descriptive terms as appropriate:

   - *Mostly*, if estimated to constitute 50 percent or more of the unit
   - *Some*, if estimated to constitute more than 25 to 50 percent of the unit
   - *Little*, if estimated to be 25 percent of the unit or less
   - *Trace*, if estimated at less than 5 percent (and not included in the total 100 percent).

4. Describe density using the following terms:

   - *Loose*, if easily penetrated with a sampling spoon
   - *Dense*, if penetration is more difficult.

5. Describe consistency using the following terms:

- *Very soft*, if present as an ooze that holds no shape
- *Soft*, if saggy
- *Stiff*, if it holds a shape
- *Very stiff*, if penetration with a spoon is low
- *Hard*, if no penetration with a spoon is possible.

6. Use other observations (e.g., obvious anthropogenic material, dramatic color changes) to define or help define sample intervals (check project-specific FSP for sample interval definition; depending upon the project-specific requirements the sample interval could be based on lithology or it could be set to a specific interval [e.g., 1 ft]).
7. Determine the boundaries of lithologic units primarily by changes in the top two dominant grain sizes estimated visually (e.g., a change from a silty sand to a gravelly sand or to a sandy silt).
8. Photograph the cores after they have been described and before any sediment is removed for processing. It is important for each core section to be photographed with adequate lighting from a standard measured distance from the core. Digital photographs will be used later in the production of digital core logs. Photographs should include a measuring tape and a label with sample ID, depth section, date, and time (see figure below)

Mixing and Sample Preparation

1. After the sample is characterized and the core observation logged on the Field Sediment Core form, remove the specified sample interval using a stainless-steel spatula or spoon (see project-specific FSP for correct sampling interval). Exercise care to not include sediment that is in direct contact with the core tube. With the approval of the field team leader, and using a decontaminated stainless steel instrument, carefully remove unrepresentative material (e.g., large shells, stones). Exercise care not to touch the sediment during this process. Note any unrepresentative material removed from the sample in the field processing area notebook.
2. Remove subsamples for analysis of unstable constituents (e.g., volatile organic compounds, acid- volatile sulfides), and place them directly into sample containers without homogenization. Completely fill the sample container so that there is no headspace or entrapped bubbles.
3. Transfer the remainder of the sample interval to a decontaminated stainless-steel bowl for homogenization. If additional sediment volume is required to fill all sample bottles (see project-specific FSP) and multiple cores need to be collected at a given station, cover the compositing bowl covered with aluminum foil (dull side down) to prevent sample contamination (e.g., from precipitation, engine exhaust) and place in a cool dark place until the next core from that location is processed.
4. After all the sediment is transferred to the compositing bowl, homogenize the contents of the bowl using stainless-steel spoons until the texture and color of the sediment appears to be uniform.
5. Distribute subsamples to the various containers specified in the project-specific FSP and preserve the samples as specified in the project-specific FSP. Briefly stir the sediment in the compositing bowl between each spoon transfer to the sample containers.
6. After all subsamples have been placed in the sample containers, if it is suspected that there is clay in a sample, perform a "ribbon test." Perform a separate ribbon test for each interval within the core where clay is suspected to be present. To perform this test, remove a small piece of sediment from the sampled interval using a decontaminated stainless-steel spoon and roll it between the fingers while wearing protective gloves. If the piece easily rolls into a ribbon it is clay; if it breaks apart, it is silt. Note this information in the field processing area notebook.
7. Subsequent intervals should be processed in the same way.

Field Quality Control Samples

If additional volumes of sediment are required to perform all analyses including quality control analyses, an additional core may need to be collected from the same location and

subsampled and homogenized accordingly. Details on collection of field quality control samples (e.g., field duplicates) will be specified in the project-specific FSP. Details on collection of field quality control samples and preparation of the certified reference materials can be found in SOP SD-02, *Preparation of Field Quality Control Samples—Sediment*. Not all of the field quality control samples discussed in this SOP may be required for a given project. The specific field quality control samples will be described in the project-specific FSP and quality assurance project plan.

Station Location Coordinates

Station locations for all field sampling will be determined using a differential global positioning system (DGPS) or by surveying. The accuracy to which the latitude and longitude of a station location is determined will be specified in the FSP. At a minimum, a DGPS capable of providing latitude and longitude coordinates with an accuracy of approximately 3 m is recommended. The DGPS consists of two satellite receivers linked to each other by a VHF telemetry radio system. The receiver will be placed over the sampling location. Details on collection of very accurate station coordinates can be found in SOP AP-06, *Navigation*.

Sample Custody and Shipping

Sample custody will be maintained in accordance with procedures outlined in SOP AP-03, *Sample Custody*. All samples will be packaged and shipped with other samples in accordance with procedures outlined in SOP AP-01, *Sample Packaging and Shipping*.

REFERENCES

ASTM. 2000. Standard practice for description and identification of soils (visual-manual procedure). ASTM Standard Method No. D 2488-00. In: ASTM Book of Standards, Volume 04.08. American Society for Testing and Materials, West Conshohocken, PA.

STANDARD OPERATING PROCEDURE (SOP) SD-16

SEDIMENT CORING USING A HAND CORER

SCOPE AND APPLICATION

This SOP defines and standardizes the collection of surface sediment samples at a fixed sampling interval (e.g., 0 to 4 in. or 0 to 6 in.) and 4-in. in diameter. This hand corer has a welded handle bar and teeth at the lower end. A stainless-steel plate is used to extract the core.

Sediment samples should be collected from areas having lower levels of constituents of interest first, followed by stations with higher expected levels of constituents of interest.

The procedures listed below may be modified in the field upon the agreement of the lead site sampler and field personnel, based on field and site conditions, after appropriate annotations have been made in the field logbook. If specialized sampling methods (e.g., ENCORE®) are to be used, refer to the manufacturer's recommended procedures. Record all pertinent information on Integral's surface sediment sampling field data form or field logbook.

EQUIPMENT AND SUPPLIES REQUIRED

- Decontaminated sampling tool (i.e., stainless-steel hand corer)
- Large stainless-steel mixing bowl and spoon
- Laboratory-supplied sample containers, insulated coolers, and ice

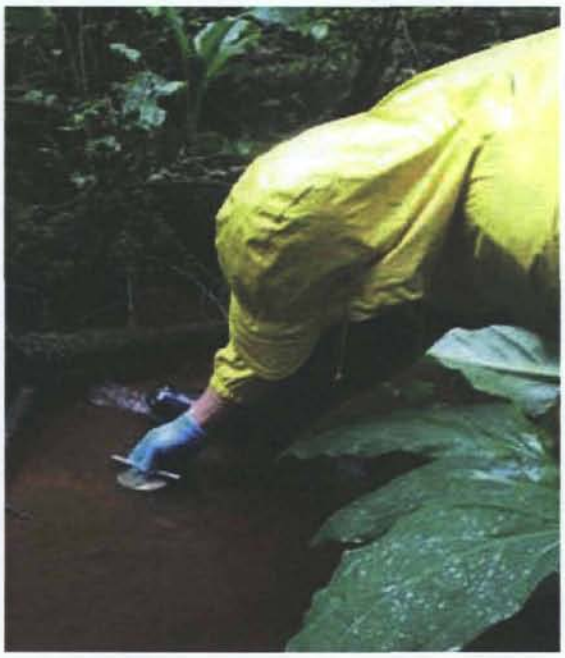
- Chain-of-custody forms, custody seals, sample labels
- Ziploc® bags
- Camera
- Stainless-steel ruler
- Field logbook, surface sediment field collection form, and pens
- Project-specific field sampling plan (FSP) and health and safety plan (HSP)
- Personal protective equipment (safety glasses, steel-toed boots, nitrile gloves, and any other items required by the project-specific HSP)
- Decontamination equipment.

PROCEDURES

Collection

1. Locate the sample station as directed in the project-specific FSP. Label containers with sample tags prior to filling in accordance with Integral's SOP on sample labeling (SOP-AP04). If analytical testing will be performed for volatile organic compounds (VOCs), collect the VOC sample first (with a minimum of disturbance) by placing the sample into the container with a minimum amount of headspace and sealed tightly.
2. Don a new pair of nitrile gloves at each sampling station.
3. At exposed sediment stations (e.g., beach sediment), clear an approximately 1 ft^2 area at the sampling site of any rocks or organic material greater than approximately 3 in. in size. Note any material removed from the sampling site in the field logbook.
4. Using a decontaminated stainless-steel hand corer, excavate the sediment to the depth specified in the project-specific FSP (either 4 in. [if side slot is used] or 6 in.).

5. At submerged sediment stations, insert the stainless-steel corer all the way until the sediment surface can be seen through the small peephole. To remove the core, insert the stainless-steel plate either on the side slot (4-in.; 10-cm) core or insert the stainless-steel plate flush with the opening end (6 in.;15-cm) as shown above. Swivel the latch on top of the core over the peephole before extracting the core. This will prevent water from flushing the sample back out when retrieving it. Once the sediment core has been retrieved, any overlying water in the core will be siphoned off the sediment surface. After the overlying water has been removed, pour sediment content into a decontaminated stainless-steel bowl. An example is shown below where sediments are collected from a shallow creek.



6. The types and number of field quality control samples for subsurface sediment samples will be specified in the project-specific FSP. If additional volumes of sediment are required to perform all analyses in addition to quality control analyses, additional cores may need to be collected from the same location and subsampled and homogenized accordingly.

Processing

1. If required for analysis, first collect VOC samples (prior to any homogenization) from a discrete location, placing the samples in the appropriate containers. Label sample containers before filling in accordance with Integral's SOP on sample labeling (SOP AP-04).

2. Describe the sediment and enter the description in the field logbook or surface sediment field collection form (see below for additional detail on preparing core descriptions).
3. Place subsampled sediment into a decontaminated stainless-steel bowl. Collect adequate volumes of sediment for all required analyses.
4. If required for analysis (consult project-specific FSP), first collect samples for grain-size tests before any large rocks are removed from the homogenized sediment.
5. Identify any rocks that are greater than 0.5 in. in diameter. Determine their percentage contribution to the homogenized sediment volume, note it on the surface sediment field collection form or in the field logbook, and then discard the rocks.
6. Mix sediment from each subsample individually in the decontaminated, stainless-steel bowl to a uniform color and texture using a decontaminated, stainless-steel spoon. Stir the sediment periodically while individual samples are taken to ensure that the mixture remains homogeneous. Exercise care to not include sediment that is in direct contact with the core tube. Fill pre-labeled jars for chemical testing with the homogenized sediment.
7. Remove samples of the homogenized sediment from the compositing bowl with the decontaminated stainless-steel spoon and place in the appropriate size sample container. Do not touch the sample with your gloves. Fill the sample container with sediment to about ½-in. below the container lip (to allow space for porewater seepage from the sediment), and seal the container tightly. Label sample containers before filling in accordance with Integral's SOP on sample labeling.
8. Complete all pertinent field QA/QC documentation, logbooks, sample labels, and field data sheets. Record any deviations from the specified sampling procedures or any obstacles encountered.
9. If appropriate, photograph sample location and document it in the logbook.
10. Decontaminate all sampling equipment according to Integral's SOP on decontaminating equipment for sediment sampling (SOP SD-01) and in accordance with the project-specific FSP.

Core Observations

1. After each sample is extruded from the hand corer, describe the sediment either in the field logbook or on the field data form. When recording the information for each core, follow the guidelines below:
   - Physical sediment description (i.e., sediment type, density/consistency, color)
   - Odor (e.g., hydrogen sulfide, petroleum)

- Visual stratification and lenses
- Vegetation
- Debris
- Evidence of biological activity (e.g., detritus, shells, tubes, bioturbation, live or dead organisms)
- Presence of oil sheen
- Other distinguishing characteristics or features.

The visual observations of sediment lithology (dominant grain sizes) may be the primary criteria for determining sample intervals (i.e., lithologic units) in the cores (consult the project-specific FSP for sample interval selection). For consistency, core descriptions and terms used will follow the criteria below, which are modified from methods presented in ASTM D 2488-00 (ASTM 2000).

2. Record visual estimates of the grain-size percentages of sediment units within each core on the core logs so that the total sum will add up to 100 percent. Make estimates of gravel, sand, and fines (silt and clay) content generally to the nearest quartiles:
   - 0 to 25 percent
   - >25 to 50 percent
   - >50 to 75 percent
   - >75 to 100 percent.
3. If appropriate, describe the sediment narratively in the field logbook or field data form based on the estimated grain-size percentages. Use the dominant constituent grain size as the primary unit descriptor, and describe the abundance of other grain sizes present using the following terms:
   - The grain-size adjective (e.g., gravelly, sandy, silty, or clayey), if estimated to constitute more than 25 percent of the sediment
   - *With*, for example, sand with silt, silt with sand, etc., if estimated to constitute less than 25 percent of the sediment
   - *Trace*, if estimated at less than 5 percent of the sediment (and not included in the total 100 percent).

   For other features observed, such as organics or debris, use the following additional descriptive terms as appropriate:
   - *Mostly*, if estimated to constitute 50 percent or more of the unit
   - *Some*, if estimated to constitute more than 25 to 50 percent of the unit
   - *Little*, if estimated to be 25 percent of the unit or less

- *Trace,* if estimated at less than 5 percent (and not included in the total 100 percent).

4. Describe density using the following terms:
   - *Loose,* if easily penetrated with a sampling spoon
   - *Dense,* if penetration is more difficult.
5. Describe consistency using the following terms:
   - *Very soft,* if present as an ooze that holds no shape
   - *Soft,* if saggy
   - *Stiff,* if it holds a shape
   - *Very stiff,* if penetration with a spoon is low
   - *Hard,* if no penetration with a spoon is possible.
6. Use other observations (e.g., obvious anthropogenic material, dramatic color changes) to define or help define sample intervals.
7. Determine the boundaries of lithologic units primarily by changes in the top two dominant grain sizes estimated visually (e.g., a change from a silty sand to a gravelly sand or to a sandy silt).

The cores should be photographed after being described and before any sediment is removed for processing. It is important for each core section to be photographed with adequate lighting from a standard measured distance from the core. Digital photographs will be used later in the production of digital core logs.

REFERENCES

ASTM. 2000. Standard practice for description and identification of soils (visual-manual procedure). ASTM Standard Method No. D 2488-00. In: ASTM Book of Standards, Volume 04.08. American Society for Testing and Materials, West Conshohocken, PA.

STANDARD OPERATING PROCEDURE (SOP) SL-01

DECONTAMINATION OF SOIL SAMPLING EQUIPMENT

SCOPE AND APPLICATION

This SOP describes procedures for decontaminating sampling and processing equipment contaminated by either organic or inorganic materials. To prevent potential cross contamination of samples, all reusable soil sampling and processing equipment is decontaminated before each use. At the sample collection site, a decontamination area is established in a clean location that is upwind of actual sampling locations, if possible. All soil sampling and processing equipment is cleaned in this location. Decontaminated equipment is stored away from areas that may cause recontamination. When handling decontamination chemicals, field personnel must follow all relevant procedures and wear protective clothing as stipulated in the site-specific health and safety plan (HSP).

Sampling equipment may be used to collect samples that will 1) undergo a full-suite analysis (organics, metals, and conventional parameters) or 2) be analyzed for metals and conventional parameters only. Decontamination of sampling equipment (e.g., hand auger, split-spoon sampler) used for both analyte groups should follow the order of a detergent wash, site water rinse, organic solvent rinses, and final site water rinse. Sample processing equipment (e.g., bowls, spoons) is rinsed with distilled/deionized water instead of with site water.

EQUIPMENT AND REAGENTS REQUIRED

Equipment required for decontamination includes the following:

- Steam cleaner and collection basin (if required)
- 55-gal, Department of Transportation (DOT)-approved drums (if required)
- Polyethylene or polypropylene tub (to collect solvent rinsate)
- Plastic bucket(s) (e.g., 5-gal bucket)
- Tap water or site water (i.e., potable water)
- Carboy, distilled/deionized water (analyte-free; received from testing laboratory or other reliable source)
- Properly labeled squirt bottles

- Funnels
- Alconox®, Liquinox®, or equivalent industrial nonphosphate detergent
- Pesticide-grade ethanol and hexane (consult project-specific field sampling plan [FSP], as the solvents may vary by U.S. Environmental Protection Agency [EPA] region or state)
- 10 percent diluted nitric acid or hydrochloric acid (reagent grade) for inorganic contaminants (if required; see project-specific FSP)
- Baking soda (if required)
- Long handled, hard-bristle brushes
- Plastic sheeting, garbage bags, and aluminum foil
- Personal protective equipment as specified in the HSP.

PROCEDURES

Decontamination Procedures for Full Suite Analysis (Organic, Metal, or Conventional Parameters)

Two organic solvents are used in this procedure. The first is miscible with water (e.g., ethanol) and is intended to scavenge water from the surface of the sampling equipment and allow the equipment to dry quickly. This allows the second solvent to fully contact the surface of the sampler. Make sure that the solvent ordered is anhydrous or has a very low water content (i.e., <1 percent). If ethanol is used, make sure that the denaturing agent in the alcohol is not one of the sample analytes. The second organic solvent is hydrophobic (e.g., hexane) and is intended to dissolve any organic chemicals that are on the surface of the equipment.

The exact solvents used for a given project may vary by EPA region or state (see project-specific FSP). Integral uses ethanol and hexane as preferred solvents for equipment decontamination. If specified in the project-specific FSP, isopropanol or acetone can be substituted for ethanol, and methanol can be substituted for hexane in the decontamination sequence. The choice of solvents is also dependent on the kind of material from which the equipment is made (e.g., acetone cannot be used on polycarbonate), and the ambient temperature (e.g., hexane is too volatile in hot climates). In addition, although methanol is slightly more effective than other solvents, its use is discouraged because of its potential toxicity to sampling personnel. Always follow the procedures listed in the site-specific HSP when decontaminating sampling equipment (e.g., always stand upwind when using volatile solvents, wear appropriate gloves and safety glasses or goggles). Containerize all decontamination fluids for proper disposal, following procedures listed in the FSP.

The specific procedures for decontaminating soil sampling equipment and soil compositing equipment are as follows:

1. Rinse the equipment thoroughly with tap or site water to remove visible soil. This step should be performed onsite for all equipment. After removing visible solids, set aside sampling equipment that does not need to be used again that day and see that it is thoroughly cleaned in the field laboratory at the end of the day.
2. Pour a small amount of concentrated laboratory detergent into a bucket (i.e., about 1 to 2 tablespoons per 5-gal bucket) and fill it halfway with tap or site water. If the detergent is in crystal form, make sure all crystals are completely dissolved prior to use.
3. Scrub the equipment in the detergent solution using a long-handled brush with rigid bristles, using a back-and-forth motion. Be sure to clean the outside of the compositing bowls and other pieces that may be covered with soil.
4. Double rinse the equipment with tap or site water and set upright on a stable surface to drain. The more completely the equipment drains, the less solvent will be needed in the next step. Do not allow any surface that will come in contact with the sample to touch any contaminated surface. If acid and solvent rinses are not required by the FSP, skip to step 8.
5. If an acid rinse is required by the FSP, rinse the equipment using a squirt bottle using a 10 percent acid solution. Double-rinse equipment with tap or site water and set right-side-up on a stable surface to drain. If solvent rinses are not required by the FSP, skip to step 8.
6. Carefully rinse the equipment with ethanol from a squirt bottle, and let the excess solvent drain into a waste container (which may need to be equipped with a funnel). These solvents act primarily as a drying agent by scavenging water from the equipment surface and carrying it away, but they also work as a solvent for some organic contamination. Hand-augers must be held over the waste container and turned slowly so the stream of solvent contacts the entire surface. The sample apparatus may be turned on its side, and if applicable, opened to be washed more effectively. Set the equipment in a clean location and allow it to air dry. Use only enough solvent to scavenge all of the water and flow off the surface of the equipment (i.e., establish sheet flow) into the waste container. Allow equipment to drain as much as possible. Ideally, the equipment will be dry. The more thoroughly it drains, the less solvent will be needed in the next step.
7. Carefully rinse the drained or air-dried equipment with hexane from a squirt bottle, and let the excess solvent drain into the waste container, which may need to be equipped with a funnel. Hexane acts as the primary solvent of organic chemicals. Ethanol is soluble in hexane but water is not. If water beading occurs, it means that the

equipment was not thoroughly rinsed with ethanol or that the ethanol that was purchased was not free of water. When the equipment has been rinsed with hexane, set it in a clean location and allow the hexane to evaporate before using the equipment for sampling. Use only enough solvent to scavenge all of the ethanol and flow off the surface of the equipment (i.e., establish sheet flow) into the waste container.

8. Do a final rinse with site water for the sampling equipment (i.e., hand-auger) and distilled/deionized water for the processing equipment (i.e., stainless-steel bowls and spoons). Equipment does not need to be dried before use.
9. If the decontaminated sampling equipment is not to be used immediately, wrap small stainless-steel items in aluminum foil (dull side facing the cleaned area).

   If the sample collection or processing equipment is precleaned at the field laboratory and transported to the site, then the decontaminated equipment will be wrapped in aluminum foil (dull side facing the cleaned area) and stored and transported in a clean plastic bag (e.g., a trash bag) until ready for use, unless the project-specific FSP lists special handling procedures.
10. After decontaminating all of the sampling equipment, dispose of the disposable gloves and used foil per the procedures listed in the project-specific FSP. When not in use, keep the waste solvent container closed and store in a secure area. The waste should be transferred to empty solvent bottles for disposal at a licensed facility per the procedures listed in the project-specific FSP. When not in use, keep the waste acid container closed and store in a secure area. The acid waste should be neutralized with baking soda or containerized and disposed of per the procedures listed in the project-specific FSP.

Decontamination Procedures for Metals and Conventional Parameters Only

The specific procedures for decontaminating soil sampling equipment and soil processing equipment are as follows:

1. Rinse the equipment thoroughly with tap or site water to remove the visible soil. Perform this step onsite for all equipment. Set aside any pieces that do not need to be used again that day see that they are thoroughly cleaned in the field laboratory at the end of the day.
2. Pour a small amount of concentrated laboratory detergent into a bucket (i.e., about 1 to 2 tablespoons per 5-gal bucket) and fill it halfway with tap or site water. If the detergent is in crystal form, make sure all crystals are completely dissolved prior to use.

3. Scrub the equipment in the detergent solution using a long-handled brush with rigid bristles. Be sure to clean the outside of the compositing bowls and other pieces that may be covered with soil.
4. Double-rinse the equipment with tap or site water (if an acid rinse is required) or with distilled/deionized water (if no acid rinse) and set right-side-up on a stable surface to drain. Do not allow any surface that will come in contact with the sample to touch any contaminated surface.
5. If an acid rinse is required by the FSP, rinse the equipment using a squirt bottle containing a 10 percent acid solution. Double-rinse equipment with distilled/deionized water and set right-side-up on a stable surface to drain.
6. If the decontaminated sampling equipment is not to be used immediately, wrap small stainless-steel items in aluminum foil (dull side facing the cleaned area).

   If the sample collecting or processing equipment is cleaned at the field laboratory and transported to the site, then the decontaminated equipment will be wrapped in aluminum foil (dull side facing the cleaned area) and stored and transported in a clean plastic bag until ready for use, unless the project-specific FSP lists special handling procedures.
7. After decontaminating all of the sampling equipment, place the disposable gloves and used foil in garbage bags for disposal in a solid waste landfill. When not in use, keep the waste acid container closed and store in a secure area. The acid waste should be neutralized with baking soda and disposed of per the procedures listed in the project-specific FSP.

Decontamination Procedures for Drill Rig or Test Pit Sampling Equipment

1. Decontaminate sampling equipment before use, between samples and stations, and upon completion of sampling operations.
2. Equipment used during drilling/test pit operations should be decontaminated in the Exclusion Zone prior to transport to the Support Zone (refer to site-specific HSP).
3. If the steam-cleaning location is in an area outside of the Exclusion Zone, remove loose soil on the drill rig, augers, drill pipe, and rods, and other large equipment at the drill site, then move the equipment directly to the steam-cleaning decontamination area for more thorough cleaning.
4. To decontaminate a drill rig or backhoe, pressure wash with a steam cleaner using potable water rinse upon mobilization, between drilling locations, and upon demobilization. Cleaning water can generally be allowed to drain directly on the ground near the station (refer to the FSP).

5. To decontaminate auger, drill rods, and other down-hole tools, pressure wash with a steam cleaner and potable water rinse upon mobilization, between drilling locations, and upon demobilization. All decontamination fluids are to be containerized for proper disposal.
6. To decontaminate split-spoon and hand-auger samplers, follow the decontamination procedures listed above (the selected decontamination procedures is dependent upon analyte list provided in the project-specific FSP). To the extent possible, allow to air dry prior to sampling. If the split-spoon is not used immediately, wrap it in aluminum foil. All decontamination fluids are to be containerized for proper disposal.

STANDARD OPERATING PROCEDURE (SOP) SL-02

PREPARATION OF FIELD QUALITY CONTROL SAMPLES FOR SOILS

SCOPE AND APPLICATION

This SOP describes the purpose, preparation, and collection frequency of field duplicate samples, field replicate samples, matrix spike/matrix spike duplicates (MS/MSDs), equipment rinsate blanks, bottle blanks, trip blanks, temperature blanks, environmental blanks, and reference materials (i.e., a standard reference material, a certified reference material, or other reference material) for soil samples. Not all of the field quality control samples discussed in this SOP may be required for a given project. The specific field quality control samples will be identified in the project-specific field sampling plan (FSP) and quality assurance project plan (QAPP). For most projects, Integral's recommended field quality control samples include an equipment rinsate blank, a field duplicate, and trip blanks if volatile organic compounds (VOCs) are to be analyzed. Definitions of all potential quality control samples are described below.

As part of the quality assurance and quality control (QA/QC) program, all field quality control samples will be sent to the laboratories blind. To accomplish this, field quality control samples will be prepared and labeled in the same manner as regular samples, with each quality control sample being assigned a unique sample number that is consistent with the numbering for regular samples. All of the containers that are required to complete the field quality control sample for the applicable analyte list must be labeled with the same sample number. The sample ID for field quality control samples should allow data management and data validation staff to identify them as such and should only be recorded in the field logbook or field sampling forms. Under no circumstances should the laboratory be allowed to use reference materials, rinsate blanks, or trip blanks for laboratory quality control analysis (i.e., duplicates, matrix spike, and matrix spike duplicates). To prevent this from happening, select and mark regular samples on the chain-of-custody/sampling analysis request (COC) form or instruct the laboratory to contact the project QA/QC coordinator to select appropriate samples for each sample group.

Prepare field quality control samples at least once per sampling event, and prepare certain types more often at predetermined frequencies. If the number of samples taken does not equal an integer multiple of the intervals specified in this SOP, the number of field quality control samples is specified by the next higher multiple. For example, if a frequency of 1 quality

control sample per 20 is indicated and 28 samples are collected, prepare 2 quality control samples. The method of preparation and frequency of field quality control samples required for soil sampling activities are described below. These protocols must be followed, unless different frequency requirements are listed in the FSP and QAPP.

For most projects, Integral's recommended field quality control samples include an equipment rinsate blank, a field duplicate, and trip blanks if VOCs are to be analyzed. The following table lists the possible quality control sample types and suggested frequencies for soil sampling programs (not all types of quality control samples will always be collected; see project-specific FSP and QAPP for actual quality control samples that need to be collected for a particular sampling event). A detailed explanation of each type of quality control sample with the required preparation follows.

Field Quality Control Sample Requirements

Quality Control Sample Name	Abbreviation	Preparation		Frequency[a]
		Location	Method	
Duplicate	DUP	Sampling site	Additional natural sample	One per 20 samples. May not be applicable if REP is being collected.
Replicate	REP	Sampling site	Additional natural sample	One replicate per 20 samples. May not be applicable if DUP is being collected.
Matrix spike/matrix spike duplicate	MS/MSD	Sampling site	Additional sample bottles filled for laboratory quality control requirements	One per 20 samples
Equipment rinsate blank	ER	Sampling site	Deionized water collected after pouring through and over decontaminated equipment	Minimum of one per sampling event per type of sampling equipment used and then 1:20 thereafter
Bottle blank	BB	Field	Unopened bottle	One per sample episode or one per bottle type
Trip blank	TB	Laboratory	Deionized water with preservative	One pair per each VOC sample cooler shipment
Temperature blank	TMB	Laboratory	Deionized water	One per sample cooler
Environmental (transfer) blank	EB	Field	Bottle filled at sample site with deionized water	One per 20 samples
Standard reference material	SRM	Field laboratory or sampling site	SRM ampules or other containers for each analyte group	One set per 50 samples or one per episode

[a] Frequencies provided here are general recommendations; specific frequencies should be provided in the project-specific FSP or QAPP.

FIELD DUPLICATE SAMPLES

Collect field duplicate (or split) samples to assess the homogeneity of the samples collected in the field and the precision of the sampling process. Prepare field duplicates by collecting two aliquots for the sample and submitting them for analysis as separate samples. Collect field duplicates at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. The project QA/QC coordinator will determine the actual number of field duplicate samples collected during a sampling event on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of field duplicate collection may vary by EPA region or state).

FIELD REPLICATE SAMPLES

Field replicate samples are co-located samples collected in an identical manner over a minimum period of time to provide a measure of the field and laboratory variance, including variance resulting from sample heterogeneity. Prepare field replicates by collecting two completely separate samples from the same station and submitting them for analysis as separate samples. Collect field replicates at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. If field duplicate samples are collected, then it is unlikely that field replicate samples will also be collected during a sampling event. The project QA/QC coordinator will determine the actual number of field replicate samples collected during a sampling event on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of field duplicate collection may vary by EPA region or state).

MATRIX SPIKE/MATRIX SPIKE DUPLICATES

The MS/MSD analyses provide information about the effect of the sample matrix on the design and measurement methodology used by the laboratory. To account for the additional volume that may be needed by the laboratory to perform the analyses, extra sample volumes may be required to be collected from designated soil stations. MS/MSDs may be collected at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. The project QA/QC coordinator will determine the actual number of extra bottles collected during a sampling event on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements may vary by analyte group).

EQUIPMENT RINSATE BLANKS

Use equipment rinsate blanks to help identify possible contamination from the sampling environment and/or from decontaminated sampling equipment. Prepare equipment rinsate

blanks by pouring laboratory distilled/deionized water through, over, and into the decontaminated sample collection equipment, then transferring the water to the appropriate sample containers and adding any necessary preservatives. Prepare equipment rinsate blanks for all inorganic, organic, and sometimes conventional analytes at least once per sampling event per the type of sampling equipment used. The project QA/QC coordinator will determine the actual number of equipment rinsate blanks prepared during an event on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of equipment rinsate blank collection may vary by EPA region or state).

BOTTLE BLANKS

The bottle blank is an unopened sample bottle. Submit bottle blanks along with soil samples to ensure that contaminants are not originating from the bottles themselves because of improper preparation, handling, or cleaning techniques. If required, submit one bottle blank per lot of prepared bottles for analysis. If more than one type of bottle will be used in the sampling (e.g., HDPE or glass), then submit a bottle blank for each type of bottle and preservative. The project QA/QC coordinator will determine the actual number of bottle blanks analyzed during a project on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of bottle blank analysis may vary by EPA region or state).

To prepare a bottle blank in the field, set aside one unopened sample bottle from each bottle lot sent from the testing laboratory. Label the bottle as "Bottle Blank" on the sample label (and in the "Remarks" column on the COC form), and send the empty bottle to the laboratory with the field samples.

TRIP BLANKS

Use trip blanks to help identify whether contaminants may have been introduced during shipment of the soil samples from the field to the laboratory for VOC analyses only. Trip blanks are prepared at the testing laboratory by pouring distilled/deionized water into two 40 mL VOC vials and tightly closing the lids. Invert each vial and tap lightly to determine if air bubbles exist. There should be no air bubbles in the VOC trip blank vials. If air bubbles are present, then note this information in the field logbook.

Transport the trip blanks unopened to and from the field in the cooler with the VOC samples. Label the trip blank and place it inside the cooler that contains newly collected VOC samples; it must remain in the cooler at all times. A trip blank must accompany samples at all times in the field. Send one trip blank (consisting of a pair of VOC vials) with each cooler of samples shipped to the testing laboratory for VOC analysis.

TEMPERATURE BLANKS

The laboratory will use temperature blanks to verify the temperature of the samples upon receipt at the testing laboratory. The testing laboratory will prepare temperature blanks by pouring distilled/deionized water into a vial and tightly closing the lid. The blanks will be transported unopened to and from the field in the cooler with the sample containers. A temperature blank must be included with each sample cooler shipped to the testing laboratory.

ENVIRONMENTAL BLANKS

Prepare the environmental (i.e., transfer) blank in the field to evaluate potential background concentrations present in the air and in the distilled/deionized water used for the final decontamination rinse. If you use unpreserved bottles, then you must add the appropriate preservative (e.g., for metals samples, use a 10 percent nitric acid solution to bring sample pH to 2 or less), if required. Collect environmental blanks at a minimum frequency of 1 in 20 samples. The project QA/QC coordinator will determine the actual number of environmental blanks analyzed during a project on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of environmental blank analysis may vary by EPA region or state).

To prepare an environmental blank in the field, open the laboratory-prepared sample bottle while at a sample collection site, fill the sample bottle with distilled/deionized water and then seal. Note the location from which the environmental blank was collected along with atmospheric conditions at the time of its collection in the field logbook. Assign the environmental blank a unique sample number, label the bottle, and then send the bottle to the laboratory with the field samples.

REFERENCE MATERIALS

Reference materials (i.e., a standard reference material, a certified reference material, or other reference material are samples containing known analytes at known concentrations that have been prepared by and obtained from EPA-approved sources. Reference materials have undergone multilaboratory analyses using a standard method which provides certified concentrations. When available for a specific analyte, Reference material samples provide a measure of analytical performance and/or analytical method bias (i.e., accuracy) of the laboratory. Several reference materials may be required to cover all analytical parameters. For all analytes where available, one reference material will be analyzed at a frequency of one per 50 samples. The project QA/QC coordinator will determine the actual number of reference materials analyzed during a project on a case-by-case basis (consult the project-specific FSP

and QAPP, as the requirements on frequency of reference material analysis may vary by EPA region or state).

STANDARD OPERATING PROCEDURE (SOP) SL-04

FIELD CLASSIFICATION OF SOIL

SCOPE AND APPLICATION

This SOP establishes the minimum information that must be recorded in the field to adequately document surface soil sampling and soil borehole advancement activities performed during field exploration. The surface soil sampling or borehole log form must be filled out completely for each station.

This SOP presents the field classification of soils to be used by Integral field staff. In general, Integral has adopted the procedures provided in American Society for Testing and Materials (ASTM) Method D-2488-00, Standard Practice for Description and Identification of Soils (attached). ASTM D-2488-00 uses the Unified Soil Classification (USC) system for naming soils. Field personnel are encouraged to study these procedures prior to initiation of fieldwork.

Soil descriptions should be precise and comprehensive without being verbose. The overall impression of the soil should not be distorted by excessive emphasis on minor constituents. In general, the similarities of consecutive soil samples should be emphasized and minor differences de-emphasized. These descriptions will be used to interpret potential contaminant transport properties, rather than interpret the exact mineralogy or tectonic environment. We are primarily interested in engineering and geochemical properties of the soil.

Soil descriptions should be provided on the surface soil field collection form or in the soil description column of the Integral's soil boring log for each sample collected. If there is no difference between consecutive soil samples, subsequent descriptions can be noted as "same as above" or minor changes such as "increasing sand" or "becomes dark brown" can be added.

The format and order of soil descriptions should be as follows:

- Group symbol (in the Unified Symbol column)
- USC name (should be identical to the ASTM D-2488-00 Group Name with the appropriate modifiers)
- Minor components
- Color
- Moisture
- Additional descriptions.

EQUIPMENT AND REAGENTS REQUIRED

- Surface soil field collection form or borehole log form (see SOP SL-06, *Logging of Soil Boreholes*)
- Munsell® soil color chart.

PROCEDURES

The USC is an engineering properties system that uses grain size to classify soils. The first major distinction is between fine-grained soils (more than 50 percent passing the No. 200 sieve [75 μm/0.0029 in.]) and coarse-grained soils (more than 50 percent retained by the No. 200 sieve). Small No. 200 sieves are necessary to classify soils near the cutoff size.

1. Fine-grained soils are classified as either silts or clays. Field determinations of silts and clays are based on observations of dry strength, dilatancy, toughness, and plasticity. Field procedures for these tests are included in ASTM D-2488-00. If these tests are used, include the results in the soil description. If these materials are encountered, perform at least one complete round of field tests for a site, preferably at the beginning of the field investigation. The modifiers "fat" and "lean" are used by ASTM to describe soils of high and low plasticity. The soil group symbols (e.g., CL, MH) already indicate plasticity characteristics, and these modifiers are not necessary in the description. Soils with high plasticity can be emphasized by describing them as "silty CLAY with high plasticity." Plasticity, for example, is an important descriptor because it is often used to interpret whether an ML soil is acting as either a leaky or a competent aquitard. For example, an ML soil can be dilatant/nonplastic and serve as a transport pathway, or it can be highly plastic and very impervious.
2. Coarse-grained soils are classified as either predominantly gravel or sand, with the No. 4 sieve (4.75 mm/0.19 in.) being the division. Use modifiers to describe the relative amounts of fine-grained soil, as noted below:

Description	Percent Fines	Group Symbol
Gravel (sand)	<5 percent	GW, GP (SW, SP)
Gravel (sand) with silt (clay)	5–15 percent	Hyphenated names
Silty (clayey) gravel (sand)	>15 percent	GM, GC (SM, SC)

The gradation of a coarse-grained soil is included in the specific soil name (e.g., fine to medium SAND with silt). Estimating the percent of size ranges following the group name is encouraged for mixtures of silt sand and gravel. Use of the modifiers "poorly graded" or "well graded" is not necessary, as they are indicated by the group symbol.

Show a borderline classification with a slash (e.g., GM/SM). Use this symbol when the soil cannot be distinctly placed in either soil group. Also use a borderline symbol when describing interbedded soils of two or more soil group names when the thickness of the beds are approximately equal, such as "interbedded lenses and layers of fine sand and silt." Do not use a borderline symbol indiscriminately. Make every effort to place the soil into a single group. (One very helpful addition to the soil log form description is the percentage of silt/sand/gravel. Even if the geologist did not have sufficient time to properly define the soil, this percentage breakdown allows classification at a later date).

3. Precede minor components, such as cobbles, roots, and construction debris with the appropriate adjective reflecting relative percentages: trace (0–5 percent), few (5–10 percent), little (15–25 percent), and some (30–45 percent). Use the word "occasional" to describe random particles of a larger size than the general soil matrix (i.e., occasional cobbles, occasional brick fragments). The term "with" indicates definite characteristics regarding the percentage of secondary particle size in the soil name. It is not to be used to describe minor components. If a nonsoil component exceeds 50 percent of an interval, state it in place of the group name.
4. Give the basic color of a soil, such as brown, gray, or red. Modify the color term with adjectives such as light, dark, or mottled, as appropriate. Especially note staining or mottling. This information, for example, may be useful to establish water table fluctuations or contamination in boreholes. The Munsell® soil color chart designation is the Integral color standard. These charts are readily available and offer a high degree of consistency in descriptions between geologists.
5. Define the degree of moisture present in the soil as dry, moist, or wet. Moisture content can be estimated from the criteria listed in Table 3 of ASTM D-2488-00.
6. If observed, note such features as discontinuities, inclusions, joints, fissures, slickensides, bedding, laminations, root holes, and major mineralogical components. Note anything unusual. Additional soil descriptions may be made at the discretion of the project manager or as the field conditions warrant. The surface soil field collection and soil boring log forms list some optional descriptions, as does Table 13 of the ASTM standard. The reader is referred to the ASTM standard for procedures of these descriptions.

The contact between two soil types must be clearly marked on the surface soil field collection or soil boring log forms. If the contact is obvious and sharp, draw it in with a straight line. If

it is gradational, use a slanted line over the interval. In the case where it is unclear, use a dashed line over the most likely interval.

For drilling activities, the field geologist, who has the advantage of watching the drilling rate and cuttings removal and can talk with the driller in real time, has a much better chance of interpreting the interval than someone in the office.

ATTACHMENT 1. ASTM D 2488 – 00, STANDARD PRACTICE FOR DESCRIPTION AND IDENTIFICATION OF SOILS (VISUAL-MANUAL PROCEDURE)

Designation: D 2488 – 00

Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)[1]

This standard is issued under the fixed designation D 2488; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope *

1.1 This practice covers procedures for the description of soils for engineering purposes.

1.2 This practice also describes a procedure for identifying soils, at the option of the user, based on the classification system described in Test Method D 2487. The identification is based on visual examination and manual tests. It must be clearly stated in reporting an identification that it is based on visual-manual procedures.

1.2.1 When precise classification of soils for engineering purposes is required, the procedures prescribed in Test Method D 2487 shall be used.

1.2.2 In this practice, the identification portion assigning a group symbol and name is limited to soil particles smaller than 3 in. (75 mm).

1.2.3 The identification portion of this practice is limited to naturally occurring soils (disturbed and undisturbed).

NOTE 1—This practice may be used as a descriptive system applied to such materials as shale, claystone, shells, crushed rock, etc. (see Appendix X2).

1.3 The descriptive information in this practice may be used with other soil classification systems or for materials other than naturally occurring soils.

1.4 The values stated in inch-pound units are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements see Section 8.

1.6 *This practice offers a set of instructions for performing one or more specific operations. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this practice may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 653 Terminology Relating to Soil, Rock, and Contained Fluids[2]

D 1452 Practice for Soil Investigation and Sampling by Auger Borings[2]

D 1586 Test Method for Penetration Test and Split-Barrel Sampling of Soils[2]

D 1587 Practice for Thin-Walled Tube Sampling of Soils[2]

D 2113 Practice for Diamond Core Drilling for Site Investigation[2]

D 2487 Classification of Soils for Engineering Purposes (Unified Soil Classification System)[2]

D 3740 Practice for Minimum Requirements for Agencies Engaged in the Testing and/or Inspection of Soil and rock as Used in Engineering Design and Construction[3]

D 4083 Practice for Description of Frozen Soils (Visual-Manual Procedure)[2]

3. Terminology

3.1 *Definitions*—Except as listed below, all definitions are in accordance with Terminology D 653.

NOTE 2—For particles retained on a 3-in. (75-mm) US standard sieve, the following definitions are suggested:
Cobbles—particles of rock that will pass a 12-in. (300-mm) square opening and be retained on a 3-in. (75-mm) sieve, and
Boulders—particles of rock that will not pass a 12-in. (300-mm) square opening.

3.1.1 *clay*—soil passing a No. 200 (75-μm) sieve that can be made to exhibit plasticity (putty-like properties) within a range of water contents, and that exhibits considerable strength when air-dry. For classification, a clay is a fine-grained soil, or the

[1] This practice is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.07 on Identification and Classification of Soils.

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[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 04.09.

***A Summary of Changes section appears at the end of this standard.**

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fine-grained portion of a soil, with a plasticity index equal to or greater than 4, and the plot of plasticity index versus liquid limit falls on or above the "A" line (see Fig. 3 of Test Method D 2487).

3.1.2 *gravel*—particles of rock that will pass a 3-in. (75-mm) sieve and be retained on a No. 4 (4.75-mm) sieve with the following subdivisions:

coarse—passes a 3-in. (75-mm) sieve and is retained on a ¾-in. (19-mm) sieve.

fine—passes a ¾-in. (19-mm) sieve and is retained on a No. 4 (4.75-mm) sieve.

3.1.3 *organic clay*—a clay with sufficient organic content to influence the soil properties. For classification, an organic clay is a soil that would be classified as a clay, except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.4 *organic silt*—a silt with sufficient organic content to influence the soil properties. For classification, an organic silt is a soil that would be classified as a silt except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.5 *peat*—a soil composed primarily of vegetable tissue in various stages of decomposition usually with an organic odor, a dark brown to black color, a spongy consistency, and a texture ranging from fibrous to amorphous.

3.1.6 *sand*—particles of rock that will pass a No. 4 (4.75-mm) sieve and be retained on a No. 200 (75-µm) sieve with the following subdivisions:

coarse—passes a No. 4 (4.75-mm) sieve and is retained on a No. 10 (2.00-mm) sieve.

medium—passes a No. 10 (2.00-mm) sieve and is retained on a No. 40 (425-µm) sieve.

fine—passes a No. 40 (425-µm) sieve and is retained on a No. 200 (75-µm) sieve.

3.1.7 *silt*—soil passing a No. 200 (75-µm) sieve that is nonplastic or very slightly plastic and that exhibits little or no strength when air dry. For classification, a silt is a fine-grained soil, or the fine-grained portion of a soil, with a plasticity index less than 4, or the plot of plasticity index versus liquid limit falls below the "A" line (see Fig. 3 of Test Method D 2487).

4. Summary of Practice

4.1 Using visual examination and simple manual tests, this practice gives standardized criteria and procedures for describing and identifying soils.

4.2 The soil can be given an identification by assigning a group symbol(s) and name. The flow charts, Fig. 1a and Fig. 1b for fine-grained soils, and Fig. 2, for coarse-grained soils, can be used to assign the appropriate group symbol(s) and name. If the soil has properties which do not distinctly place it into a specific group, borderline symbols may be used, see Appendix X3.

NOTE 3—It is suggested that a distinction be made between *dual symbols* and *borderline symbols*.

Dual Symbol—A dual symbol is two symbols separated by a hyphen, for example, GP-GM, SW-SC, CL-ML used to indicate that the soil has been identified as having the properties of a classification in accordance with Test Method D 2487 where two symbols are required. Two symbols are required when the soil has between 5 and 12 % fines or when the liquid limit and plasticity index values plot in the CL-ML area of the plasticity chart.

Borderline Symbol—A borderline symbol is two symbols separated by a slash, for example, CL/CH, GM/SM, CL/ML. A borderline symbol should be used to indicate that the soil has been identified as having properties that do not distinctly place the soil into a specific group (see Appendix X3).

5. Significance and Use

5.1 The descriptive information required in this practice can be used to describe a soil to aid in the evaluation of its significant properties for engineering use.

5.2 The descriptive information required in this practice should be used to supplement the classification of a soil as determined by Test Method D 2487.

5.3 This practice may be used in identifying soils using the classification group symbols and names as prescribed in Test Method D 2487. Since the names and symbols used in this practice to identify the soils are the same as those used in Test Method D 2487, it shall be clearly stated in reports and all other appropriate documents, that the classification symbol and name are based on visual-manual procedures.

5.4 This practice is to be used not only for identification of soils in the field, but also in the office, laboratory, or wherever soil samples are inspected and described.

5.5 This practice has particular value in grouping similar soil samples so that only a minimum number of laboratory tests need be run for positive soil classification.

NOTE 4—The ability to describe and identify soils correctly is learned more readily under the guidance of experienced personnel, but it may also be acquired systematically by comparing numerical laboratory test results for typical soils of each type with their visual and manual characteristics.

5.6 When describing and identifying soil samples from a given boring, test pit, or group of borings or pits, it is not necessary to follow all of the procedures in this practice for every sample. Soils which appear to be similar can be grouped together; one sample completely described and identified with the others referred to as similar based on performing only a few of the descriptive and identification procedures described in this practice.

5.7 This practice may be used in combination with Practice D 4083 when working with frozen soils.

NOTE 5—Notwithstanding the statements on precision and bias contained in this standard: The precision of this test method is dependent on the competence of the personnel performing it and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D 3740 are generally considered capable of competent and objective testing. Users of this test method are cautioned that compliance with Practice D 3740 does not in itself assure reliable testing. Reliable testing depends on several factors; Practice D 3740 provides a means for evaluating some of those factors.

6. Apparatus

6.1 *Required Apparatus*:

6.1.1 *Pocket Knife or Small Spatula.*

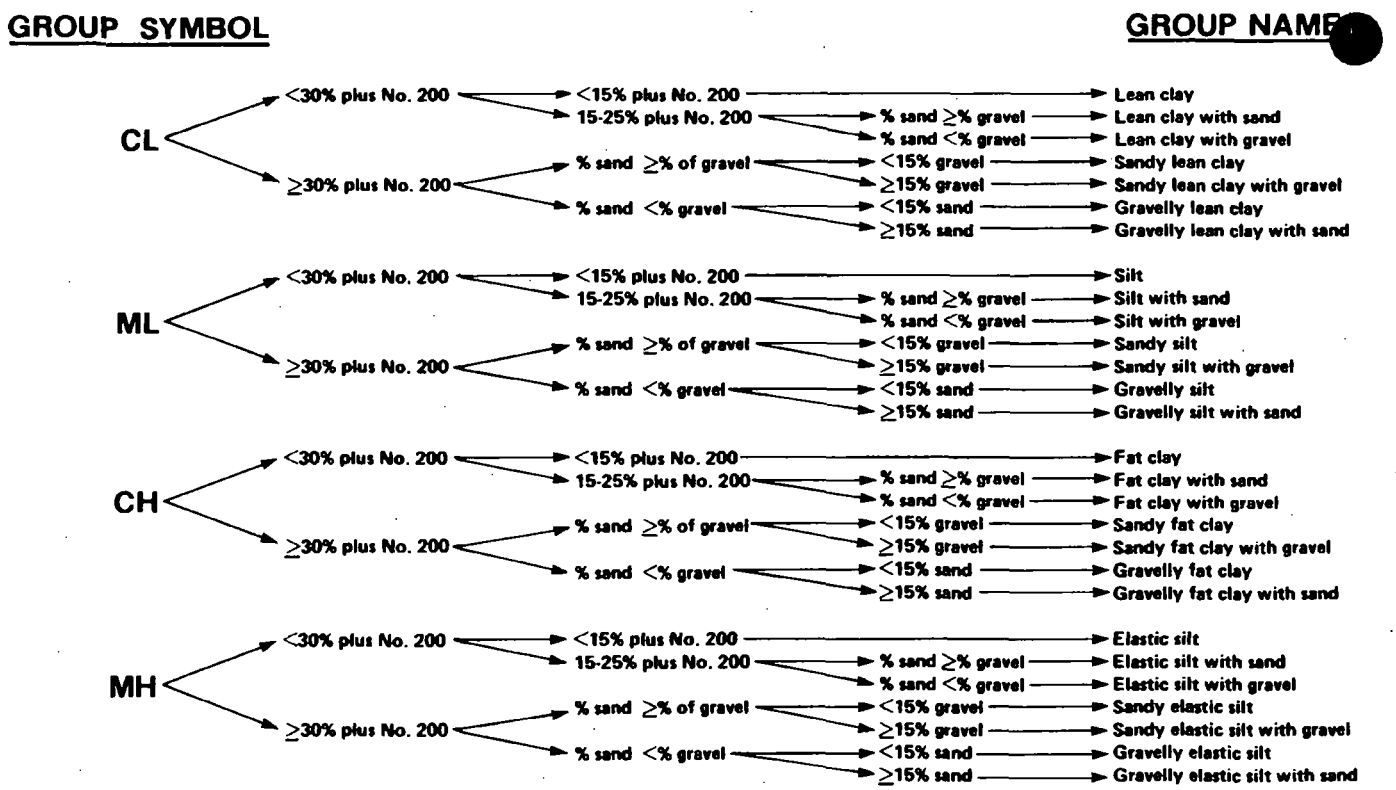
6.2 *Useful Auxiliary Apparatus*:

6.2.1 *Small Test Tube and Stopper* (or jar with a lid).

6.2.2 *Small Hand Lens.*

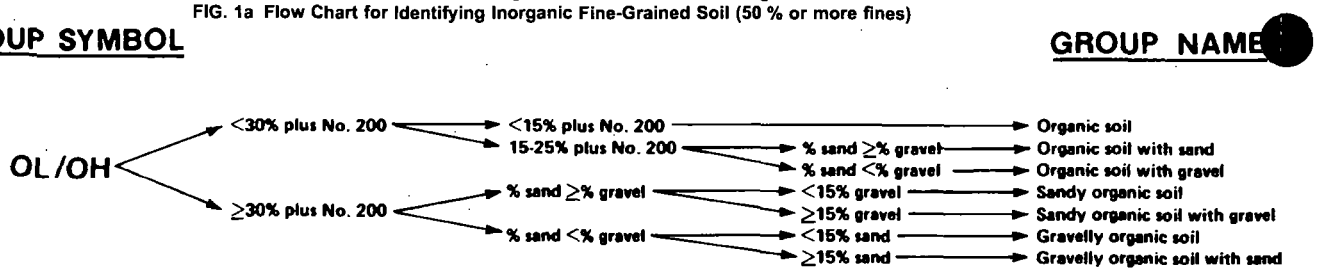
7. Reagents

7.1 *Purity of Water*—Unless otherwise indicated, references



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1a Flow Chart for Identifying Inorganic Fine-Grained Soil (50 % or more fines)



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1.b Flow Chart for Identifying Organic Fine-Grained Soil (50 % or more fines)

to water shall be understood to mean water from a city water supply or natural source, including non-potable water.

7.2 *Hydrochloric Acid*—A small bottle of dilute hydrochloric acid, HCl, one part HCl (10 *N*) to three parts water (This reagent is optional for use with this practice). See Section 8.

8. Safety Precautions

8.1 When preparing the dilute HCl solution of one part concentrated hydrochloric acid (10 *N*) to three parts of distilled water, slowly add acid into water following necessary safety precautions. Handle with caution and store safely. If solution comes into contact with the skin, rinse thoroughly with water.

8.2 **Caution**—Do not add water to acid.

9. Sampling

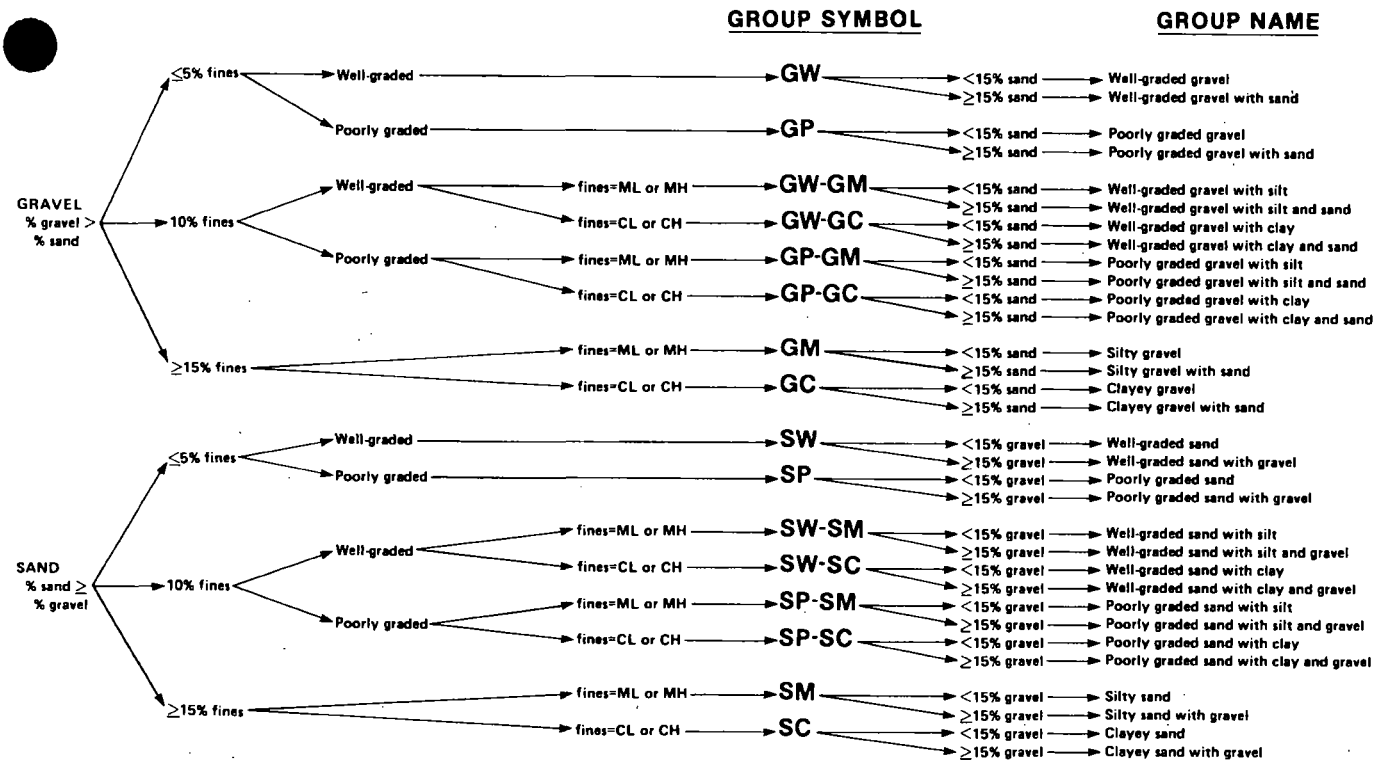
9.1 The sample shall be considered to be representative of the stratum from which it was obtained by an appropriate, accepted, or standard procedure.

NOTE 6—Preferably, the sampling procedure should be identified as having been conducted in accordance with Practices D 1452, D 1587, or D 2113, or Test Method D 1586.

9.2 The sample shall be carefully identified as to origin.

NOTE 7—Remarks as to the origin may take the form of a boring number and sample number in conjunction with a job number, a geologic stratum, a pedologic horizon or a location description with respect to a permanent monument, a grid system or a station number and offset with respect to a stated centerline and a depth or elevation.

9.3 For accurate description and identification, the minimum amount of the specimen to be examined shall be in accordance with the following schedule:



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 2 Flow Chart for Identifying Coarse-Grained Soils (less than 50 % fines)

Maximum Particle Size, Sieve Opening	Minimum Specimen Size, Dry Weight
4.75 mm (No. 4)	100 g (0.25 lb)
9.5 mm (⅜ in.)	200 g (0.5 lb)
19.0 mm (¾ in.)	1.0 kg (2.2 lb)
38.1 mm (1½ in.)	8.0 kg (18 lb)
75.0 mm (3 in.)	60.0 kg (132 lb)

NOTE 8—If random isolated particles are encountered that are significantly larger than the particles in the soil matrix, the soil matrix can be accurately described and identified in accordance with the preceeding schedule.

9.4 If the field sample or specimen being examined is smaller than the minimum recommended amount, the report shall include an appropriate remark.

10. Descriptive Information for Soils

10.1 *Angularity*—Describe the angularity of the sand (coarse sizes only), gravel, cobbles, and boulders, as angular, subangular, subrounded, or rounded in accordance with the criteria in Table 1 and Fig. 3. A range of angularity may be stated, such as: subrounded to rounded.

10.2 *Shape*—Describe the shape of the gravel, cobbles, and boulders as flat, elongated, or flat and elongated if they meet the criteria in Table 2 and Fig. 4. Otherwise, do not mention the shape. Indicate the fraction of the particles that have the shape, such as: one-third of the gravel particles are flat.

10.3 *Color*—Describe the color. Color is an important property in identifying organic soils, and within a given locality it may also be useful in identifying materials of similar geologic origin. If the sample contains layers or patches of varying colors, this shall be noted and all representative colors shall be described. The color shall be described for moist samples. If the color represents a dry condition, this shall be stated in the report.

TABLE 1 Criteria for Describing Angularity of Coarse-Grained Particles (see Fig. 3)

Description	Criteria
Angular	Particles have sharp edges and relatively plane sides with unpolished surfaces
Subangular	Particles are similar to angular description but have rounded edges
Subrounded	Particles have nearly plane sides but have well-rounded corners and edges
Rounded	Particles have smoothly curved sides and no edges

10.4 *Odor*—Describe the odor if organic or unusual. Soils containing a significant amount of organic material usually have a distinctive odor of decaying vegetation. This is especially apparent in fresh samples, but if the samples are dried, the odor may often be revived by heating a moistened sample. If the odor is unusual (petroleum product, chemical, and the like), it shall be described.

10.5 *Moisture Condition*—Describe the moisture condition as dry, moist, or wet, in accordance with the criteria in Table 3.

10.6 *HCl Reaction*—Describe the reaction with HCl as none, weak, or strong, in accordance with the critera in Table 4. Since calcium carbonate is a common cementing agent, a report of its presence on the basis of the reaction with dilute hydrochloric acid is important.

10.7 *Consistency*—For intact fine-grained soil, describe the

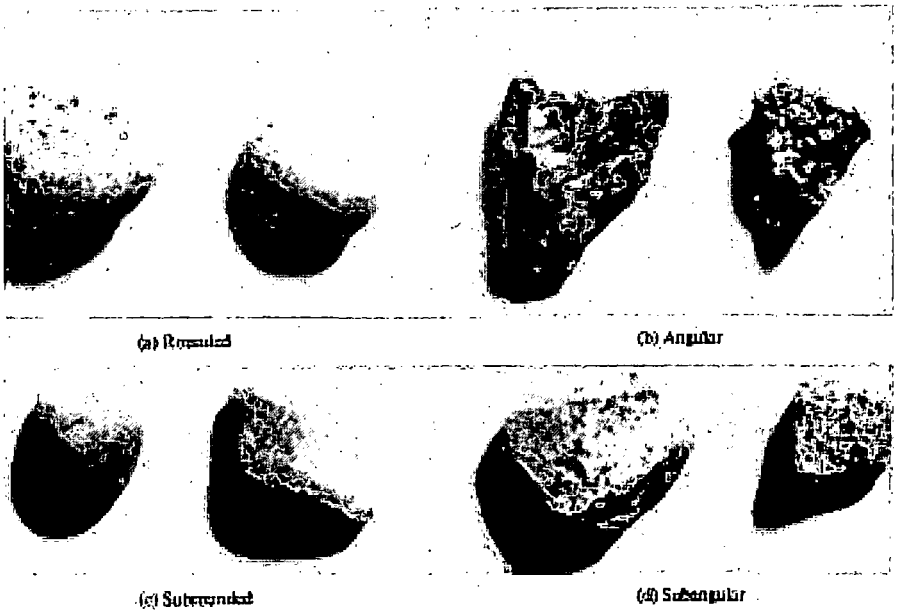


FIG. 3 Typical Angularity of Bulky Grains

TABLE 2 Criteria for Describing Particle Shape (see Fig. 4)

The particle shape shall be described as follows where length, width, and thickness refer to the greatest, intermediate, and least dimensions of a particle, respectively.	
Flat	Particles with width/thickness > 3
Elongated	Particles with length/width > 3
Flat and elongated	Particles meet criteria for both flat and elongated

consistency as very soft, soft, firm, hard, or very hard, in accordance with the criteria in Table 5. This observation is inappropriate for soils with significant amounts of gravel.

10.8 *Cementation*—Describe the cementation of intact coarse-grained soils as weak, moderate, or strong, in accordance with the criteria in Table 6.

10.9 *Structure*—Describe the structure of intact soils in accordance with the criteria in Table 7.

10.10 *Range of Particle Sizes*—For gravel and sand components, describe the range of particle sizes within each component as defined in 3.1.2 and 3.1.6. For example, about 20 % fine to coarse gravel, about 40 % fine to coarse sand.

10.11 *Maximum Particle Size*—Describe the maximum particle size found in the sample in accordance with the following information:

10.11.1 *Sand Size*—If the maximum particle size is a sand size, describe as fine, medium, or coarse as defined in 3.1.6. For example: maximum particle size, medium sand.

10.11.2 *Gravel Size*—If the maximum particle size is a gravel size, describe the maximum particle size as the smallest sieve opening that the particle will pass. For example, maximum particle size, 1½ in. (will pass a 1½-in. square opening but not a ¾-in. square opening).

10.11.3 *Cobble or Boulder Size*—If the maximum particle size is a cobble or boulder size, describe the maximum dimension of the largest particle. For example: maximum dimension, 18 in. (450 mm).

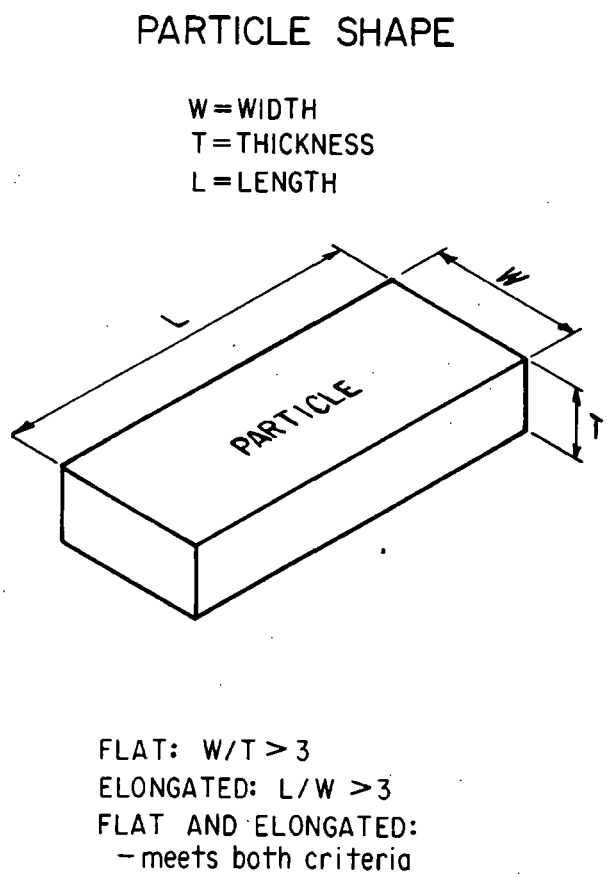


FIG. 4 Criteria for Particle Shape

10.12 *Hardness*—Describe the hardness of coarse sand and larger particles as hard, or state what happens when the

TABLE 3 Criteria for Describing Moisture Condition

Description	Criteria
Dry	Absence of moisture, dusty, dry to the touch
Moist	Damp but no visible water
Wet	Visible free water, usually soil is below water table

TABLE 4 Criteria for Describing the Reaction With HCl

Description	Criteria
None	No visible reaction
Weak	Some reaction, with bubbles forming slowly
Strong	Violent reaction, with bubbles forming immediately

TABLE 5 Criteria for Describing Consistency

Description	Criteria
Very soft	Thumb will penetrate soil more than 1 in. (25 mm)
Soft	Thumb will penetrate soil about 1 in. (25 mm)
Firm	Thumb will indent soil about ¼in. (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very hard	Thumbnail will not indent soil

TABLE 6 Criteria for Describing Cementation

Description	Criteria
Weak	Crumbles or breaks with handling or little finger pressure
Moderate	Crumbles or breaks with considerable finger pressure
Strong	Will not crumble or break with finger pressure

TABLE 7 Criteria for Describing Structure

Description	Criteria
Stratified	Alternating layers of varying material or color with layers at least 6 mm thick; note thickness
Laminated	Alternating layers of varying material or color with the layers less than 6 mm thick; note thickness
Fissured	Breaks along definite planes of fracture with little resistance to fracturing
Slickensided	Fracture planes appear polished or glossy, sometimes striated
Blocky	Cohesive soil that can be broken down into small angular lumps which resist further breakdown
Lensed	Inclusion of small pockets of different soils, such as small lenses of sand scattered through a mass of clay; note thickness
Homogeneous	Same color and appearance throughout

particles are hit by a hammer, for example, gravel-size particles fracture with considerable hammer blow, some gravel-size particles crumble with hammer blow. "Hard" means particles do not crack, fracture, or crumble under a hammer blow.

10.13 Additional comments shall be noted, such as the presence of roots or root holes, difficulty in drilling or augering hole, caving of trench or hole, or the presence of mica.

10.14 A local or commercial name or a geologic interpretation of the soil, or both, may be added if identified as such.

10.15 A classification or identification of the soil in accordance with other classification systems may be added if identified as such.

11. Identification of Peat

11.1 A sample composed primarily of vegetable tissue in various stages of decomposition that has a fibrous to amorphous texture, usually a dark brown to black color, and an organic odor, shall be designated as a highly organic soil and shall be identified as peat, PT, and not subjected to the identification procedures described hereafter.

12. Preparation for Identification

12.1 The soil identification portion of this practice is based on the portion of the soil sample that will pass a 3-in. (75-mm) sieve. The larger than 3-in. (75-mm) particles must be removed, manually, for a loose sample, or mentally, for an intact sample before classifying the soil.

12.2 Estimate and note the percentage of cobbles and the percentage of boulders. Performed visually, these estimates will be on the basis of volume percentage.

NOTE 9—Since the percentages of the particle-size distribution in Test Method D 2487 are by dry weight, and the estimates of percentages for gravel, sand, and fines in this practice are by dry weight, it is recommended that the report state that the percentages of cobbles and boulders are by volume.

12.3 Of the fraction of the soil smaller than 3 in. (75 mm), estimate and note the percentage, by dry weight, of the gravel, sand, and fines (see Appendix X4 for suggested procedures).

NOTE 10—Since the particle-size components appear visually on the basis of volume, considerable experience is required to estimate the percentages on the basis of dry weight. Frequent comparisons with laboratory particle-size analyses should be made.

12.3.1 The percentages shall be estimated to the closest 5 %. The percentages of gravel, sand, and fines must add up to 100 %.

12.3.2 If one of the components is present but not in sufficient quantity to be considered 5 % of the smaller than 3-in. (75-mm) portion, indicate its presence by the term *trace*, for example, trace of fines. A trace is not to be considered in the total of 100 % for the components.

13. Preliminary Identification

13.1 The soil is *fine grained* if it contains 50 % or more fines. Follow the procedures for identifying fine-grained soils of Section 14.

13.2 The soil is *coarse grained* if it contains less than 50 % fines. Follow the procedures for identifying coarse-grained soils of Section 15.

14. Procedure for Identifying Fine-Grained Soils

14.1 Select a representative sample of the material for examination. Remove particles larger than the No. 40 sieve (medium sand and larger) until a specimen equivalent to about a handful of material is available. Use this specimen for performing the dry strength, dilatancy, and toughness tests.

14.2 *Dry Strength*:

14.2.1 From the specimen, select enough material to mold into a ball about 1 in. (25 mm) in diameter. Mold the material until it has the consistency of putty, adding water if necessary.

14.2.2 From the molded material, make at least three test specimens. A test specimen shall be a ball of material about ½ in. (12 mm) in diameter. Allow the test specimens to dry in air, or sun, or by artificial means, as long as the temperature does not exceed 60°C.

14.2.3 If the test specimen contains natural dry lumps, those that are about ½ in. (12 mm) in diameter may be used in place of the molded balls.

NOTE 11—The process of molding and drying usually produces higher strengths than are found in natural dry lumps of soil.

14.2.4 Test the strength of the dry balls or lumps by crushing between the fingers. Note the strength as none, low, medium, high, or very high in accorance with the criteria in Table 8. If natural dry lumps are used, do not use the results of any of the lumps that are found to contain particles of coarse sand.

14.2.5 The presence of high-strength water-soluble cementing materials, such as calcium carbonate, may cause exceptionally high dry strengths. The presence of calcium carbonate can usually be detected from the intensity of the reaction with dilute hydrochloric acid (see 10.6).

14.3 *Dilatancy*:

14.3.1 From the specimen, select enough material to mold into a ball about ½ in. (12 mm) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.

14.3.2 Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally, striking the side of the hand vigorously against the other hand several times. Note the reaction of water appearing on the surface of the soil. Squeeze the sample by closing the hand or pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the criteria in Table 9. The reaction is the speed with which water appears while shaking, and disappears while squeezing.

14.4 *Toughness*:

14.4.1 Following the completion of the dilatancy test, the test specimen is shaped into an elongated pat and rolled by hand on a smooth surface or between the palms into a thread about ⅛ in. (3 mm) in diameter. (If the sample is too wet to roll easily, it should be spread into a thin layer and allowed to lose some water by evaporation.) Fold the sample threads and reroll repeatedly until the thread crumbles at a diameter of about ⅛ in. The thread will crumble at a diameter of ⅛ in. when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, the pieces should be lumped together and kneaded until the lump crumbles. Note the toughness of the material during kneading.

14.4.2 Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in Table 10.

TABLE 8 Criteria for Describing Dry Strength

Description	Criteria
None	The dry specimen crumbles into powder with mere pressure of handling
Low	The dry specimen crumbles into powder with some finger pressure
Medium	The dry specimen breaks into pieces or crumbles with considerable finger pressure
High	The dry specimen cannot be broken with finger pressure. Specimen will break into pieces between thumb and a hard surface
Very high	The dry specimen cannot be broken between the thumb and a hard surface

TABLE 9 Criteria for Describing Dilatancy

Description	Criteria
None	No visible change in the specimen
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing

14.5 *Plasticity*—On the basis of observations made during the toughness test, describe the plasticity of the material in accordance with the criteria given in Table 11.

14.6 Decide whether the soil is an *inorganic* or an *organic* fine-grained soil (see 14.8). If inorganic, follow the steps given in 14.7.

14.7 *Identification of Inorganic Fine-Grained Soils*:

14.7.1 Identify the soil as a *lean clay*, CL, if the soil has medium to high dry strength, no or slow dilatancy, and medium toughness and plasticity (see Table 12).

14.7.2 Identify the soil as a *fat clay*, CH, if the soil has high to very high dry strength, no dilatancy, and high toughness and plasticity (see Table 12).

14.7.3 Identify the soil as a *silt*, ML, if the soil has no to low dry strength, slow to rapid dilatancy, and low toughness and plasticity, or is nonplastic (see Table 12).

14.7.4 Identify the soil as an *elastic silt*, MH, if the soil has low to medium dry strength, no to slow dilatancy, and low to medium toughness and plasticity (see Table 12).

NOTE 12—These properties are similar to those for a lean clay. However, the silt will dry quickly on the hand and have a smooth, silky feel when dry. Some soils that would classify as MH in accordance with the criteria in Test Method D 2487 are visually difficult to distinguish from lean clays, CL. It may be necessary to perform laboratory testing for proper identification.

14.8 *Identification of Organic Fine-Grained Soils*:

14.8.1 Identify the soil as an *organic soil*, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soils usually have a dark brown to black color and may have an organic odor. Often, organic soils will change color, for example, black to brown, when exposed to the air. Some organic soils will lighten in color significantly when air dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

NOTE 13—In some cases, through practice and experience, it may be possible to further identify the organic soils as organic silts or organic clays, OL or OH. Correlations between the dilatancy, dry strength, toughness tests, and laboratory tests can be made to identify organic soils in certain deposits of similar materials of known geologic origin.

TABLE 10 Criteria for Describing Toughness

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

TABLE 11 Criteria for Describing Plasticity

Description	Criteria
Nonplastic	A ⅛-in. (3-mm) thread cannot be rolled at any water content
Low	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rerolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit

TABLE 12 Identification of Inorganic Fine-Grained Soils from Manual Tests

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

14.9 If the soil is estimated to have 15 to 25 % sand or gravel, or both, the words "with sand" or "with gravel" (whichever is more predominant) shall be added to the group name. For example: "lean clay with sand, CL" or "silt with gravel, ML" (see Fig. 1a and Fig. 1b). If the percentage of sand is equal to the percentage of gravel, use "with sand."

14.10 If the soil is estimated to have 30 % or more sand or gravel, or both, the words "sandy" or "gravelly" shall be added to the group name. Add the word "sandy" if there appears to be more sand than gravel. Add the word "gravelly" if there appears to be more gravel than sand. For example: "sandy lean clay, CL", "gravelly fat clay, CH", or "sandy silt, ML" (see Fig. 1a and Fig. 1b). If the percentage of sand is equal to the percent of gravel, use "sandy."

15. Procedure for Identifying Coarse-Grained Soils
(Contains less than 50 % fines)

15.1 The soil is a *gravel* if the percentage of gravel is estimated to be more than the percentage of sand.

15.2 The soil is a *sand* if the percentage of gravel is estimated to be equal to or less than the percentage of sand.

15.3 The soil is a *clean gravel* or *clean sand* if the percentage of fines is estimated to be 5 % or less.

15.3.1 Identify the soil as a *well-graded gravel*, GW, or as a *well-graded sand*, SW, if it has a wide range of particle sizes and substantial amounts of the intermediate particle sizes.

15.3.2 Identify the soil as a *poorly graded gravel*, GP, or as a *poorly graded sand*, SP, if it consists predominantly of one size (uniformly graded), or it has a wide range of sizes with some intermediate sizes obviously missing (gap or skip graded).

15.4 The soil is either a *gravel with fines* or a *sand with fines* if the percentage of fines is estimated to be 15 % or more.

15.4.1 Identify the soil as a *clayey gravel*, GC, or a *clayey sand*, SC, if the fines are clayey as determined by the procedures in Section 14.

15.4.2 Identify the soil as a *silty gravel*, GM, or a *silty sand*, SM, if the fines are silty as determined by the procedures in Section 14.

15.5 If the soil is estimated to contain 10 % fines, give the soil a dual identification using two group symbols.

15.5.1 The first group symbol shall correspond to a clean gravel or sand (GW, GP, SW, SP) and the second symbol shall correspond to a gravel or sand with fines (GC, GM, SC, SM).

15.5.2 The group name shall correspond to the first group symbol plus the words "with clay" or "with silt" to indicate the plasticity characteristics of the fines. For example: "well-graded gravel with clay, GW-GC" or "poorly graded sand with silt, SP-SM" (see Fig. 2).

15.6 If the specimen is predominantly sand or gravel but contains an estimated 15 % or more of the other coarse-grained constituent, the words "with gravel" or "with sand" shall be added to the group name. For example: "poorly graded gravel with sand, GP" or "clayey sand with gravel, SC" (see Fig. 2).

15.7 If the field sample contains any cobbles or boulders, or both, the words "with cobbles" or "with cobbles and boulders" shall be added to the group name. For example: "silty gravel with cobbles, GM."

16. Report

16.1 The report shall include the information as to origin, and the items indicated in Table 13.

NOTE 14—*Example: Clayey Gravel with Sand and Cobbles, GC*—About 50 % fine to coarse, subrounded to subangular gravel; about 30 % fine to coarse, subrounded sand; about 20 % fines with medium plasticity, high dry strength, no dilatancy, medium toughness; weak reaction with HCl; original field sample had about 5 % (by volume) subrounded cobbles, maximum dimension, 150 mm.

In-Place Conditions—Firm, homogeneous, dry, brown

Geologic Interpretation—Alluvial fan

TABLE 13 Checklist for Description of Soils

1. Group name
2. Group symbol
3. Percent of cobbles or boulders, or both (by volume)
4. Percent of gravel, sand, or fines, or all three (by dry weight)
5. Particle-size range:
   Gravel—fine, coarse
   Sand—fine, medium, coarse
6. Particle angularity: angular, subangular, subrounded, rounded
7. Particle shape: (if appropriate) flat, elongated, flat and elongated
8. Maximum particle size or dimension
9. Hardness of coarse sand and larger particles
10. Plasticity of fines: nonplastic, low, medium, high
11. Dry strength: none, low, medium, high, very high
12. Dilatancy: none, slow, rapid
13. Toughness: low, medium, high
14. Color (in moist condition)
15. Odor (mention only if organic or unusual)
16. Moisture: dry, moist, wet
17. Reaction with HCl: none, weak, strong

For intact samples:

18. Consistency (fine-grained soils only): very soft, soft, firm, hard, very hard
19. Structure: stratified, laminated, fissured, slickensided, lensed, homogeneous
20. Cementation: weak, moderate, strong
21. Local name
22. Geologic interpretation
23. Additional comments: presence of roots or root holes, presence of mica, gypsum, etc., surface coatings on coarse-grained particles, caving or sloughing of auger hole or trench sides, difficulty in augering or excavating, etc.

NOTE 15—Other examples of soil descriptions and identification are given in Appendix X1 and Appendix X2.

NOTE 16—If desired, the percentages of gravel, sand, and fines may be stated in terms indicating a range of percentages, as follows:

Trace—Particles are present but estimated to be less than 5 %
Few—5 to 10 %
Little—15 to 25 %
Some—30 to 45 %
Mostly—50 to 100 %

16.2 If, in the soil description, the soil is identified using a classification group symbol and name as described in Test Method D 2487, it must be distinctly and clearly stated in log forms, summary tables, reports, and the like, that the symbol and name are based on visual-manual procedures.

17. Precision and Bias

17.1 This practice provides qualitative information only, therefore, a precision and bias statement is not applicable.

18. Keywords

18.1 classification; clay; gravel; organic soils; sand; silt; soil classification; soil description; visual classification

APPENDIXES

(Nonmandatory Information)

X1. EXAMPLES OF VISUAL SOIL DESCRIPTIONS

X1.1 The following examples show how the information required in 16.1 can be reported. The information that is included in descriptions should be based on individual circumstances and need.

X1.1.1 *Well-Graded Gravel with Sand (GW)*—About 75 % fine to coarse, hard, subangular gravel; about 25 % fine to coarse, hard, subangular sand; trace of fines; maximum size, 75 mm, brown, dry; no reaction with HCl.

X1.1.2 *Silty Sand with Gravel (SM)*—About 60 % predominantly fine sand; about 25 % silty fines with low plasticity, low dry strength, rapid dilatancy, and low toughness; about 15 % fine, hard, subrounded gravel, a few gravel-size particles fractured with hammer blow; maximum size, 25 mm; no reaction with HCl (Note—Field sample size smaller than recommended).

In-Place Conditions—Firm, stratified and contains lenses of silt 1 to 2 in. (25 to 50 mm) thick, moist, brown to gray; in-place density 106 lb/ft^3; in-place moisture 9 %.

X1.1.3 *Organic Soil (OL/OH)*—About 100 % fines with low plasticity, slow dilatancy, low dry strength, and low toughness; wet, dark brown, organic odor; weak reaction with HCl.

X1.1.4 *Silty Sand with Organic Fines (SM)*—About 75 % fine to coarse, hard, subangular reddish sand; about 25 % organic and silty dark brown nonplastic fines with no dry strength and slow dilatancy; wet; maximum size, coarse sand; weak reaction with HCl.

X1.1.5 *Poorly Graded Gravel with Silt, Sand, Cobbles and Boulders (GP-GM)*—About 75 % fine to coarse, hard, subrounded to subangular gravel; about 15 % fine, hard, subrounded to subangular sand; about 10 % silty nonplastic fines; moist, brown; no reaction with HCl; original field sample had about 5 % (by volume) hard, subrounded cobbles and a trace of hard, subrounded boulders, with a maximum dimension of 18 in. (450 mm).

X2. USING THE IDENTIFICATION PROCEDURE AS A DESCRIPTIVE SYSTEM FOR SHALE, CLAYSTONE, SHELLS, SLAG, CRUSHED ROCK, AND THE LIKE

X2.1 The identification procedure may be used as a descriptive system applied to materials that exist in-situ as shale, claystone, sandstone, siltstone, mudstone, etc., but convert to soils after field or laboratory processing (crushing, slaking, and the like).

X2.2 Materials such as shells, crushed rock, slag, and the like, should be identified as such. However, the procedures used in this practice for describing the particle size and plasticity characteristics may be used in the description of the material. If desired, an identification using a group name and symbol according to this practice may be assigned to aid in describing the material.

X2.3 The group symbol(s) and group names should be placed in quotation marks or noted with some type of distinguishing symbol. See examples.

X2.4 Examples of how group names and symbols can be incororated into a descriptive system for materials that are not naturally occurring soils are as follows:

X2.4.1 *Shale Chunks*—Retrieved as 2 to 4-in. (50 to 100-mm) pieces of shale from power auger hole, dry, brown, no reaction with HCl. After slaking in water for 24 h, material identified as "Sandy Lean Clay (CL)"; about 60 % fines with medium plasticity, high dry strength, no dilatancy, and medium toughness; about 35 % fine to medium, hard sand; about 5 % gravel-size pieces of shale.

X2.4.2 *Crushed Sandstone*—Product of commercial crushing operation; "Poorly Graded Sand with Silt (SP-SM)"; about 90 % fine to medium sand; about 10 % nonplastic fines; dry, reddish-brown, strong reaction with HCl.

X2.4.3 *Broken Shells*—About 60 % gravel-size broken

shells; about 30 % sand and sand-size shell pieces; about 10 % fines; "Poorly Graded Gravel with Sand (GP)."

X2.4.4 *Crushed Rock*—Processed from gravel and cobbles in Pit No. 7; "Poorly Graded Gravel (GP)"; about 90 % fine, hard, angular gravel-size particles; about 10 % coarse, hard, angular sand-size particles; dry, tan; no reaction with HCl.

X3. SUGGESTED PROCEDURE FOR USING A BORDERLINE SYMBOL FOR SOILS WITH TWO POSSIBLE IDENTIFICATIONS.

X3.1 Since this practice is based on estimates of particle size distribution and plasticity characteristics, it may be difficult to clearly identify the soil as belonging to one category. To indicate that the soil may fall into one of two possible basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example: SC/CL or CL/CH.

X3.1.1 A borderline symbol may be used when the percentage of fines is estimated to be between 45 and 55 %. One symbol should be for a coarse-grained soil with fines and the other for a fine-grained soil. For example: GM/ML or CL/SC.

X3.1.2 A borderline symbol may be used when the percentage of sand and the percentage of gravel are estimated to be about the same. For example: GP/SP, SC/GC, GM/SM. It is practically impossible to have a soil that would have a borderline symbol of GW/SW.

X3.1.3 A borderline symbol may be used when the soil could be either well graded or poorly graded. For example: GW/GP, SW/SP.

X3.1.4 A borderline symbol may be used when the soil could either be a silt or a clay. For example: CL/ML, CH/MH, SC/SM.

X3.1.5 A borderline symbol may be used when a fine-grained soil has properties that indicate that it is at the boundary between a soil of low compressibility and a soil of high compressibility. For example: CL/CH, MH/ML.

X3.2 The order of the borderline symbols should reflect similarity to surrounding or adjacent soils. For example: soils in a borrow area have been identified as CH. One sample is considered to have a borderline symbol of CL and CH. To show similarity, the borderline symbol should be CH/CL.

X3.3 The group name for a soil with a borderline symbol should be the group name for the first symbol, except for:

CL/CH lean to fat clay
ML/CL clayey silt
CL/ML silty clay

X3.4 The use of a borderline symbol should not be used indiscriminately. Every effort shall be made to first place the soil into a single group.

X4. SUGGESTED PROCEDURES FOR ESTIMATING THE PERCENTAGES OF GRAVEL, SAND, AND FINES IN A SOIL SAMPLE

X4.1 *Jar Method*—The relative percentage of coarse- and fine-grained material may be estimated by thoroughly shaking a mixture of soil and water in a test tube or jar, and then allowing the mixture to settle. The coarse particles will fall to the bottom and successively finer particles will be deposited with increasing time; the sand sizes will fall out of suspension in 20 to 30 s. The relative proportions can be estimated from the relative volume of each size separate. This method should be correlated to particle-size laboratory determinations.

X4.2 *Visual Method*—Mentally visualize the gravel size particles placed in a sack (or other container) or sacks. Then, do the same with the sand size particles and the fines. Then, mentally compare the number of sacks to estimate the percentage of plus No. 4 sieve size and minus No. 4 sieve size present. The percentages of sand and fines in the minus sieve size No. 4 material can then be estimated from the wash test (X4.3).

X4.3 *Wash Test (for relative percentages of sand and fines)*—Select and moisten enough minus No. 4 sieve size material to form a 1-in (25-mm) cube of soil. Cut the cube in half, set one-half to the side, and place the other half in a small dish. Wash and decant the fines out of the material in the dish until the wash water is clear and then compare the two samples and estimate the percentage of sand and fines. Remember that the percentage is based on weight, not volume. However, the volume comparison will provide a reasonable indication of grain size percentages.

X4.3.1 While washing, it may be necessary to break down lumps of fines with the finger to get the correct percentages.

X5. ABBREVIATED SOIL CLASSIFICATION SYMBOLS

X5.1 In some cases, because of lack of space, an abbreviated system may be useful to indicate the soil classification symbol and name. Examples of such cases would be graphical logs, databases, tables, etc.

X5.2 This abbreviated system is not a substitute for the full name and descriptive information but can be used in supplementary presentations when the complete description is referenced.

X5.3 The abbreviated system should consist of the soil classification symbol based on this standard with appropriate lower case letter prefixes and suffixes as:

Prefix:	Suffix:
s = sandy	s = with sand
g = gravelly	g = with gravel
	c = with cobbles
	b = with boulders

X5.4 The soil classification symbol is to be enclosed in parenthesis. Some examples would be:

Group Symbol and Full Name	Abbreviated
CL, Sandy lean clay	s(CL)
SP-SM, Poorly graded sand with silt and gravel	(SP-SM)g
GP, poorly graded gravel with sand, cobbles, and boulders	(GP)scb
ML, gravelly silt with sand and cobbles	g(ML)sc

SUMMARY OF CHANGES

In accordance with Committee D18 policy, this section identifies the location of changes to this standard since the last edition (1993$^{\epsilon 1}$) that may impact the use of this standard.

(1) Added Practice D 3740 to Section 2.

(2) Added Note 5 under 5.7 and renumbered subsequent notes.

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STANDARD OPERATING PROCEDURE (SOP) SL-05

SURFACE SOIL SAMPLING

SCOPE AND APPLICATION

This SOP defines and standardizes the collection of surface soil samples (e.g., 0 to 12 in. below ground surface). Soil samples should be collected from areas having lower levels of constituents of interest first, followed by stations with higher expected levels of constituents of interest.

The procedures listed below may be modified in the field upon the agreement of the lead site sampler and field personnel, based on field and site conditions, after appropriate annotations have been made in the field logbook. If specialized sampling methods (e.g., ENCORE®) are to be used, refer to the manufacturer's recommended procedures. If methanol preservation is required, refer to Integral's SOP on methanol preservation of soil samples. Record all pertinent information on Integral's surface soil sampling field data form or field logbook.

EQUIPMENT AND SUPPLIES REQUIRED

- Decontaminated sampling tool (stainless-steel shovel, scoop, trowel, spoon, or hand corer)
- Large stainless steel mixing bowl and spoon
- Laboratory-supplied sample containers, insulated coolers, and ice
- Chain-of-custody forms, custody seals, sample labels
- Ziploc® bags
- Camera
- Tape measure
- Field logbook, surface soil field collection form, and pens
- Project-specific field sampling plan (FSP) and health and safety plan (HSP)
- Personal protective equipment (safety glasses, steel-toed boots, nitrile gloves, and any other items required by the project-specific HSP)
- Decontamination equipment.

PROCEDURES

Sample Collection

1. Locate the sample station as directed in the project-specific FSP. Label containers with sample labels prior to filling in accordance with Integral's SOP on sample labeling (SOP-AP04).
2. At each station, don a new pair of nitrile gloves and expose the soil surface by clearing an approximately 1 ft^2 area at the sampling site of any rocks or organic material greater than approximately 3 in. in size. Note any material removed from the sampling site in the field logbook.
3. If using a decontaminated stainless-steel sampling tool (i.e., shovel, scoop, trowel, spoon), excavate the soil to the depth specified in the project-specific FSP.
4. If using a decontaminated stainless-steel hand corer, then several different versions of a hand corer can be used to excavate the soil as shown below.

   a. Long-handled hand corer

      Several layers of soil can be removed at a time minimizing cross-contamination between layers. This method is ideal for moist soils where small soil clumps do not fall to top of lower sections. The core can be replaced by a newly decontaminated core by unscrewing the core from the long handle bar.

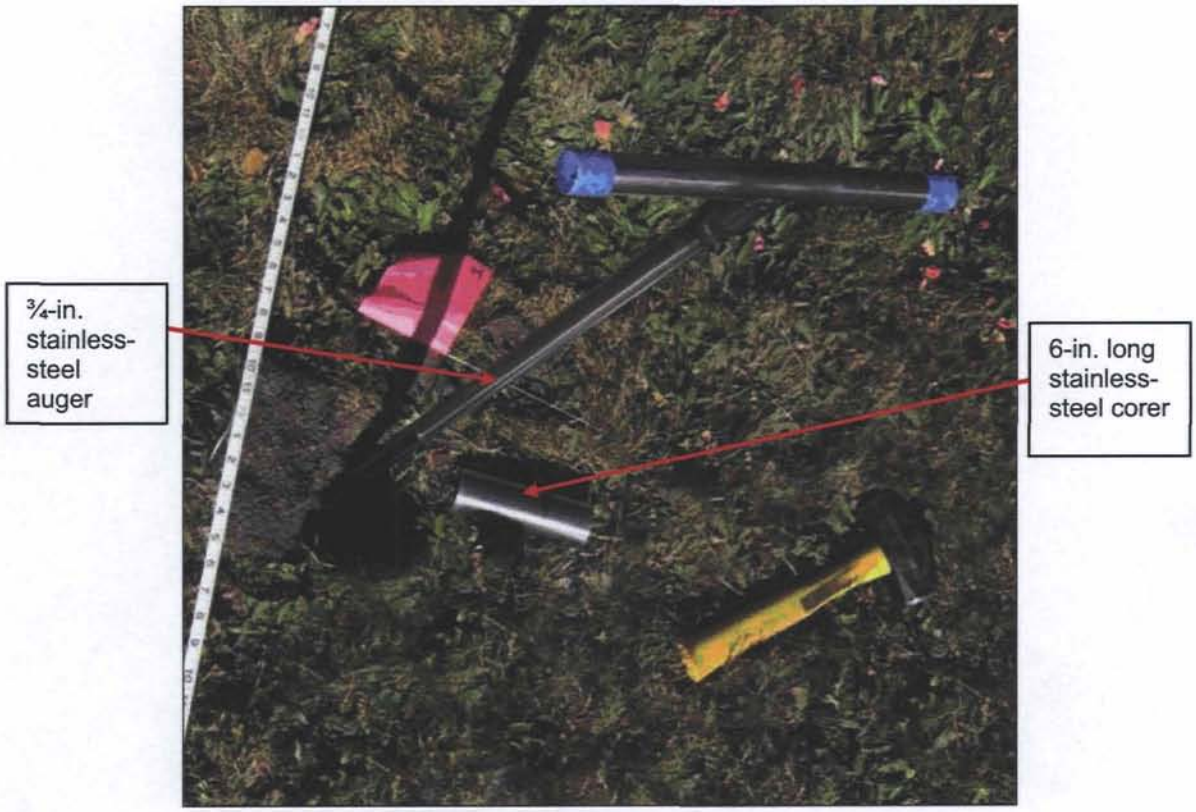
Note: If roots or rocks are present, making it difficult to use a long-handled hand corer, seek additional help.

b. Short-handled hand corer (used when a long handle is not necessary)

Note: The short handle can be unscrewed from the core tube to allow the handle to be used again on other decontaminated core tubes.

c. Stem auger

A decontaminated aluminum plate is placed over a 6-in. long and 2-in. diameter stainless-steel core tube and driven into the soil with a mallet. A ¾-in. stem auger is then used to advance the core tube to the next soil sampling interval (per the project-specific FSP).



Note: This method prevents higher concentrations of contaminants found in surface soil from being pushed down into deeper layers.

d. Predetermined sampling interval hand corer

Another type of stainless-steel hand corer is a fixed 6 in. long (4 in. long if side slot is used) and 4 in. in diameter. It has a welded handle bar and teeth at the lower end. A stainless-steel plate is used to extract the core.

To collect sediment using this corer, insert the corer all the way until the soil surface can be seen through the small peephole. To remove the core, insert the stainless-steel plate on the side slot for a 4-in. (10-cm) core or flush with the opening end for a 6-in. (15-cm) core as shown below.

Sample Processing

1. If required for analysis, first collect VOC samples with a minimum of disturbance (prior to any homogenization) from a discrete location, placing the samples in the appropriate containers with no headspace and sealing the container tightly. Label sample containers before filling in accordance with Integral's SOP on sample labeling (SOP AP-04).
2. If using a decontaminated stainless-steel sampling tool (i.e., shovel, scoop, trowel, spoon) to collect the soil sample, place sample material directly into a decontaminated plastic or stainless-steel mixing bowl, and cover the bowl with foil until an adequate sample volume (as stipulated in the project-specific FSP) has been collected for that station.
3. If using a decontaminated hand-corer to collect the soil sample, then once the corer is full, or has attained the penetration depth specified in the project-specific FSP, use a decontaminated stainless-steel spoon to transfer the soil into a decontaminated stainless-steel bowl, and cover the bowl with foil until an adequate sample volume (as stipulated in the project-specific FSP) has been collected for that station.

   If the soil is difficult to remove from the hand corer, then the contents can be loosened by carefully tapping on the outside of the corer with a separate implement from the one used to collect and composite the sample (i.e., the decontaminated spoon). Care will always be taken to ensure that any substance on the outside of the corer does not enter the sample collection bowl (see figure below).

4. Describe the soil in accordance with ASTM D2488-00 (see Integral's SOP on field classification of soils, SOP SL-04).
5. Thoroughly mix and homogenize the sample using disposable equipment or a decontaminated stainless-steel spoon until the color and texture are consistent throughout.
6. If required for analysis, first collect samples for grain-size tests before any large rocks are removed from the homogenized soil.
7. Identify any rocks that are greater than 0.5 in. in diameter. Determine their percentage contribution to the homogenized soil volume, note it on the surface soil field collection form or in the field logbook, and then discard the rocks.
8. Remove samples of the homogenized soil from the mixing bowl with the decontaminated stainless steel spoon and place in the appropriate size sample container. Do not touch the sample with your gloves. Fill the sample container with soil to just below the container lip, and seal the container tightly. Label sample containers before filling in accordance with Integral's SOP on sample labeling.
9. Mark the sampling site with a wire flag, wooden stake, metal rebar, or flagging, as appropriate.
10. Complete all pertinent field QA/QC documentation, logbooks, sample labels, and field data sheets. Record any deviations from the specified sampling procedures or any obstacles encountered.
11. Photograph the sample location and document it in the logbook.

12. Decontaminate all sampling equipment according to Integral's SOP on decontaminating equipment for soil sampling (SOP SL-01) and in accordance with the project-specific FSP.

STANDARD OPERATING PROCEDURE (SOP) SL-06

LOGGING OF SOIL BOREHOLES

SCOPE AND APPLICATION

This SOP describes how to complete a Soil Boring Log form, which must be completed for Integral projects where soil boring techniques are performed during field exploration. A correctly completed form contains all of the information that must be recorded in the field to adequately characterize soil boreholes.

These procedures are adapted from ASTM D-2488-00. Field staff are encouraged to examine ASTM D-2488-00 in its entirety. This SOP represents minor modifications to emphasize environmental investigations rather than geotechnical investigations, for which the standards were written. Because each environmental project is unique and because job requirements can vary widely, the minimum standards presented may need to be supplemented with additional technical descriptions or field test results. However, all soil boring field logs, regardless of special project circumstances, must include information addressed in this SOP to achieve the minimum acceptable standards required by Integral.

LOG FORM INFORMATION

Project Number—Use the standard contract number.

Client—Identify the name of the client and the project site location.

Location—If stations, coordinates, mileposts, or similar markers are applicable, use them to identify the location of the project. If this information is not available, identify the facility (e.g., 20 ft NE of Retort #1).

Drilling Method—Identify the bit size and type, drilling fluid (if used), and method of drilling (e.g., rotary, hollow-stem auger, cable tool) and the name of the drill rig (e.g., Mobil B 61, CME 55).

Diameter—Provide the diameter of the borehole. If the borehole has variable diameters, provide the depth interval for each diameter.

Sampling Method—Identify the type of sampler(s) used (e.g., standard split spoon, Dames & Moore sampler, grab).

Drilling Contractor—Provide the name of the drilling contractor.

Integral Staff—Enter the name(s) of Integral staff members performing logging and sampling activities.

Water Level Information—Provide the date, time, depth to static water, and casing depth. Generally, water levels should be taken each day before resuming drilling and at the completion of drilling. If water is not encountered in the boring, this information should be recorded.

Boring Number—Provide the boring number. A numbering system should be developed prior to drilling that does not conflict with other site information, such as previous drilling or other sampling activities.

Sheet—Number the sheets consecutively for each boring and continue the consecutive depth numbering.

Drilling Start and Finish—Provide the drilling start and finish dates and times.

For consecutive sheets, provide (at a minimum) the job number, boring number, and sheet number.

TECHNICAL DATA

Sampler Type—Provide the sampler type (e.g., SS = split spoon, G = grab).

Depth of Casing—Enter the depth of the casing below ground surface immediately prior to sampling.

Driven/Recovery—Provide the length that the sampler was driven and the length of sample recovered in the sampler. This column would not apply to grab samples.

Sample Number/Sample Depth—Provide the sample number. The sample numbering scheme should be established prior to drilling. One method is to use the boring number and consecutive alphabetical letters. For instance, the first sample obtained from boring MW-4 would be identified as 4A, the second would be identified as 4B, and so on. Another method for sample identification is naming the boring number with the depth. For example, the sample from Boring 1 at 10 ft would be labeled B1-10'. The depth of the sample is the depth of the casing plus the length to the middle of the recovered sample to the nearest 0.1 ft. Typically, split spoon samplers are 18 in. long. Samples should be obtained from the middle of the recovered sample. The depth of the sample with the casing at 10 ft would then be 10.7 ft.

Number of Blows—For standard split-spoon samplers, record the number of blows for each 6 in. of sampler penetration. A typical blow count of 6, 12, and 14 is recorded as 6/12/14. Refusal is a penetration of less than 6 in. with a blow count of 50. A partial penetration of 50 blows for 4 in. is recorded as 50/4". Total blows will be recorded for nonstandard split spoons (e.g., 5-ft tube used for continuous sampling).

Blank Columns—Two blank columns are provided. Use these columns for site-specific information, usually related to the chemicals of concern. Examples for a hydrocarbon site would be sheen and photoionization detector readings of the samples.

Depth—Use a depth scale that is appropriate for the complexity of the subsurface conditions. The boxes located to the right of the scale should be used to graphically indicate sample locations as shown in the example.

Surface Conditions—Describe the surface conditions (e.g., paved, 4-in. concrete slab, grass, natural vegetation and surface soil, oil-stained gravel).

Soil Description—Enter the soil classification and definition of soil contacts using the format described in SOP SL-04, *Field Classification of Soil.*

Comments—Include all pertinent observations. Drilling observations might include drilling chatter, rod-bounce (boulder), sudden differences in drilling speed, damaged samplers, and malfunctioning equipment. Information provided by the driller should be attributed to the driller. Information on possible contaminants might include odor, staining, color, and presence or absence of some indicator of contamination. Describe what it is that indicates contamination (e.g., fuel-like odor, oily sheen in drill cuttings, yellow water in drill cuttings).

ATTACHMENT 1. SOIL BORING LOG FORM

integral

319 SW Washington St., Suite 1150
Portland, OR 97204
(503) 284-5545

STATION NUMBER
PROJECT
LOCATION
PROJECT NUMBER
LOGGED BY

SAMPLE INFORMATION						STRATA	DESCRIPTION
Sample ID	Depth	Time	Tag No.	% Recov.	Depth (Feet)		USCS group name, color, grain size range, minor constituents, plasticity, odor, sheen, moisture content, texture, weathering, cementation, geologic interpretation, etc.
					2--		
					4--		
					6--		
					8--		
					10--		
					12--		
					14--		

DRILLING CONTRACTOR
DRILLING METHOD
SAMPLING EQUIPMENT
DRILLING STARTED
COORDINATES
SURFACE ELEVATION
DATUM

Location Sketch

ATTACHMENT 2. ASTM D 2488 – 00, STANDARD PRACTICE FOR DESCRIPTION AND IDENTIFICATION OF SOILS (VISUAL-MANUAL PROCEDURE)

Designation: D 2488 – 00

Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)[1]

This standard is issued under the fixed designation D 2488; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope *

1.1 This practice covers procedures for the description of soils for engineering purposes.

1.2 This practice also describes a procedure for identifying soils, at the option of the user, based on the classification system described in Test Method D 2487. The identification is based on visual examination and manual tests. It must be clearly stated in reporting an identification that it is based on visual-manual procedures.

1.2.1 When precise classification of soils for engineering purposes is required, the procedures prescribed in Test Method D 2487 shall be used.

1.2.2 In this practice, the identification portion assigning a group symbol and name is limited to soil particles smaller than 3 in. (75 mm).

1.2.3 The identification portion of this practice is limited to naturally occurring soils (disturbed and undisturbed).

NOTE 1—This practice may be used as a descriptive system applied to such materials as shale, claystone, shells, crushed rock, etc. (see Appendix X2).

1.3 The descriptive information in this practice may be used with other soil classification systems or for materials other than naturally occurring soils.

1.4 The values stated in inch-pound units are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements see Section 8.

1.6 *This practice offers a set of instructions for performing one or more specific operations. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this practice may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 653 Terminology Relating to Soil, Rock, and Contained Fluids[2]

D 1452 Practice for Soil Investigation and Sampling by Auger Borings[2]

D 1586 Test Method for Penetration Test and Split-Barrel Sampling of Soils[2]

D 1587 Practice for Thin-Walled Tube Sampling of Soils[2]

D 2113 Practice for Diamond Core Drilling for Site Investigation[2]

D 2487 Classification of Soils for Engineering Purposes (Unified Soil Classification System)[2]

D 3740 Practice for Minimum Requirements for Agencies Engaged in the Testing and/or Inspection of Soil and rock as Used in Engineering Design and Construction[3]

D 4083 Practice for Description of Frozen Soils (Visual-Manual Procedure)[2]

3. Terminology

3.1 *Definitions*—Except as listed below, all definitions are in accordance with Terminology D 653.

NOTE 2—For particles retained on a 3-in. (75-mm) US standard sieve, the following definitions are suggested:
Cobbles—particles of rock that will pass a 12-in. (300-mm) square opening and be retained on a 3-in. (75-mm) sieve, and
Boulders—particles of rock that will not pass a 12-in. (300-mm) square opening.

3.1.1 *clay*—soil passing a No. 200 (75-μm) sieve that can be made to exhibit plasticity (putty-like properties) within a range of water contents, and that exhibits considerable strength when air-dry. For classification, a clay is a fine-grained soil, or the

[1] This practice is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.07 on Identification and Classification of Soils.

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[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 04.09.

***A Summary of Changes section appears at the end of this standard.**

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fine-grained portion of a soil, with a plasticity index equal to or greater than 4, and the plot of plasticity index versus liquid limit falls on or above the "A" line (see Fig. 3 of Test Method D 2487).

3.1.2 *gravel*—particles of rock that will pass a 3-in. (75-mm) sieve and be retained on a No. 4 (4.75-mm) sieve with the following subdivisions:

coarse—passes a 3-in. (75-mm) sieve and is retained on a ¾-in. (19-mm) sieve.

fine—passes a ¾-in. (19-mm) sieve and is retained on a No. 4 (4.75-mm) sieve.

3.1.3 *organic clay*—a clay with sufficient organic content to influence the soil properties. For classification, an organic clay is a soil that would be classified as a clay, except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.4 *organic silt*—a silt with sufficient organic content to influence the soil properties. For classification, an organic silt is a soil that would be classified as a silt except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.5 *peat*—a soil composed primarily of vegetable tissue in various stages of decomposition usually with an organic odor, a dark brown to black color, a spongy consistency, and a texture ranging from fibrous to amorphous.

3.1.6 *sand*—particles of rock that will pass a No. 4 (4.75-mm) sieve and be retained on a No. 200 (75-µm) sieve with the following subdivisions:

coarse—passes a No. 4 (4.75-mm) sieve and is retained on a No. 10 (2.00-mm) sieve.

medium—passes a No. 10 (2.00-mm) sieve and is retained on a No. 40 (425-µm) sieve.

fine—passes a No. 40 (425-µm) sieve and is retained on a No. 200 (75-µm) sieve.

3.1.7 *silt*—soil passing a No. 200 (75-µm) sieve that is nonplastic or very slightly plastic and that exhibits little or no strength when air dry. For classification, a silt is a fine-grained soil, or the fine-grained portion of a soil, with a plasticity index less than 4, or the plot of plasticity index versus liquid limit falls below the "A" line (see Fig. 3 of Test Method D 2487).

4. Summary of Practice

4.1 Using visual examination and simple manual tests, this practice gives standardized criteria and procedures for describing and identifying soils.

4.2 The soil can be given an identification by assigning a group symbol(s) and name. The flow charts, Fig. 1a and Fig. 1b for fine-grained soils, and Fig. 2, for coarse-grained soils, can be used to assign the appropriate group symbol(s) and name. If the soil has properties which do not distinctly place it into a specific group, borderline symbols may be used, see Appendix X3.

NOTE 3—It is suggested that a distinction be made between *dual symbols* and *borderline symbols*.

Dual Symbol—A dual symbol is two symbols separated by a hyphen, for example, GP-GM, SW-SC, CL-ML used to indicate that the soil has been identified as having the properties of a classification in accordance with Test Method D 2487 where two symbols are required. Two symbols are required when the soil has between 5 and 12 % fines or when the liquid limit and plasticity index values plot in the CL-ML area of the plasticity chart.

Borderline Symbol—A borderline symbol is two symbols separated by a slash, for example, CL/CH, GM/SM, CL/ML. A borderline symbol should be used to indicate that the soil has been identified as having properties that do not distinctly place the soil into a specific group (see Appendix X3).

5. Significance and Use

5.1 The descriptive information required in this practice can be used to describe a soil to aid in the evaluation of its significant properties for engineering use.

5.2 The descriptive information required in this practice should be used to supplement the classification of a soil as determined by Test Method D 2487.

5.3 This practice may be used in identifying soils using the classification group symbols and names as prescribed in Test Method D 2487. Since the names and symbols used in this practice to identify the soils are the same as those used in Test Method D 2487, it shall be clearly stated in reports and all other appropriate documents, that the classification symbol and name are based on visual-manual procedures.

5.4 This practice is to be used not only for identification of soils in the field, but also in the office, laboratory, or wherever soil samples are inspected and described.

5.5 This practice has particular value in grouping similar soil samples so that only a minimum number of laboratory tests need be run for positive soil classification.

NOTE 4—The ability to describe and identify soils correctly is learned more readily under the guidance of experienced personnel, but it may also be acquired systematically by comparing numerical laboratory test results for typical soils of each type with their visual and manual characteristics.

5.6 When describing and identifying soil samples from a given boring, test pit, or group of borings or pits, it is not necessary to follow all of the procedures in this practice for every sample. Soils which appear to be similar can be grouped together; one sample completely described and identified with the others referred to as similar based on performing only a few of the descriptive and identification procedures described in this practice.

5.7 This practice may be used in combination with Practice D 4083 when working with frozen soils.

NOTE 5—Notwithstanding the statements on precision and bias contained in this standard: The precision of this test method is dependent on the competence of the personnel performing it and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D 3740 are generally considered capable of competent and objective testing. Users of this test method are cautioned that compliance with Practice D 3740 does not in itself assure reliable testing. Reliable testing depends on several factors; Practice D 3740 provides a means for evaluating some of those factors.

6. Apparatus

6.1 *Required Apparatus*:

6.1.1 *Pocket Knife or Small Spatula.*

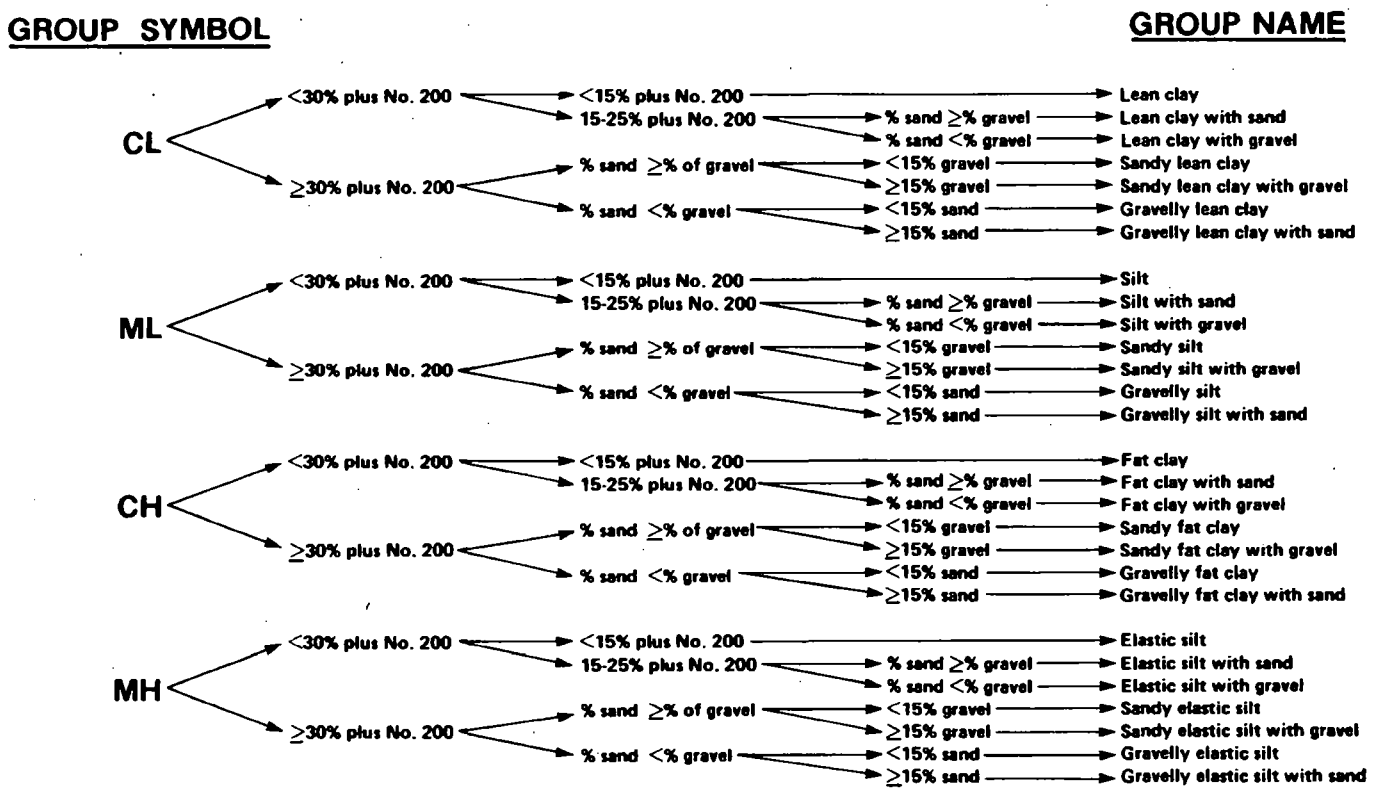
6.2 *Useful Auxiliary Apparatus*:

6.2.1 *Small Test Tube and Stopper* (or jar with a lid).

6.2.2 *Small Hand Lens.*

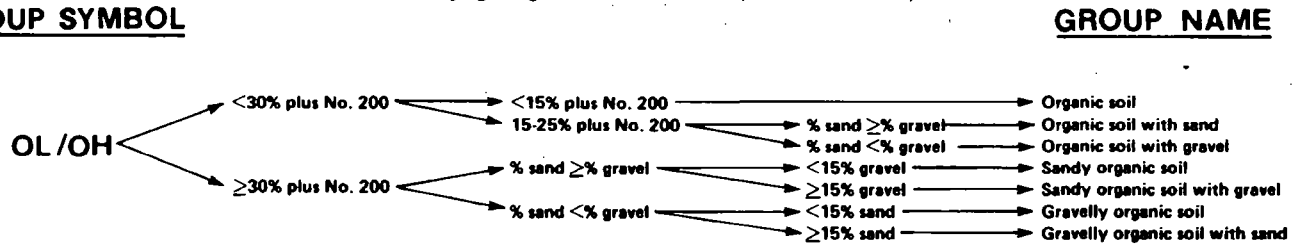
7. Reagents

7.1 *Purity of Water*—Unless otherwise indicated, references



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1a Flow Chart for Identifying Inorganic Fine-Grained Soil (50 % or more fines)



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1 b Flow Chart for Identifying Organic Fine-Grained Soil (50 % or more fines)

to water shall be understood to mean water from a city water supply or natural source, including non-potable water.

7.2 *Hydrochloric Acid*—A small bottle of dilute hydrochloric acid, HCl, one part HCl (10 *N*) to three parts water (This reagent is optional for use with this practice). See Section 8.

8. Safety Precautions

8.1 When preparing the dilute HCl solution of one part concentrated hydrochloric acid (10 *N*) to three parts of distilled water, slowly add acid into water following necessary safety precautions. Handle with caution and store safely. If solution comes into contact with the skin, rinse thoroughly with water.

8.2 **Caution**—Do not add water to acid.

9. Sampling

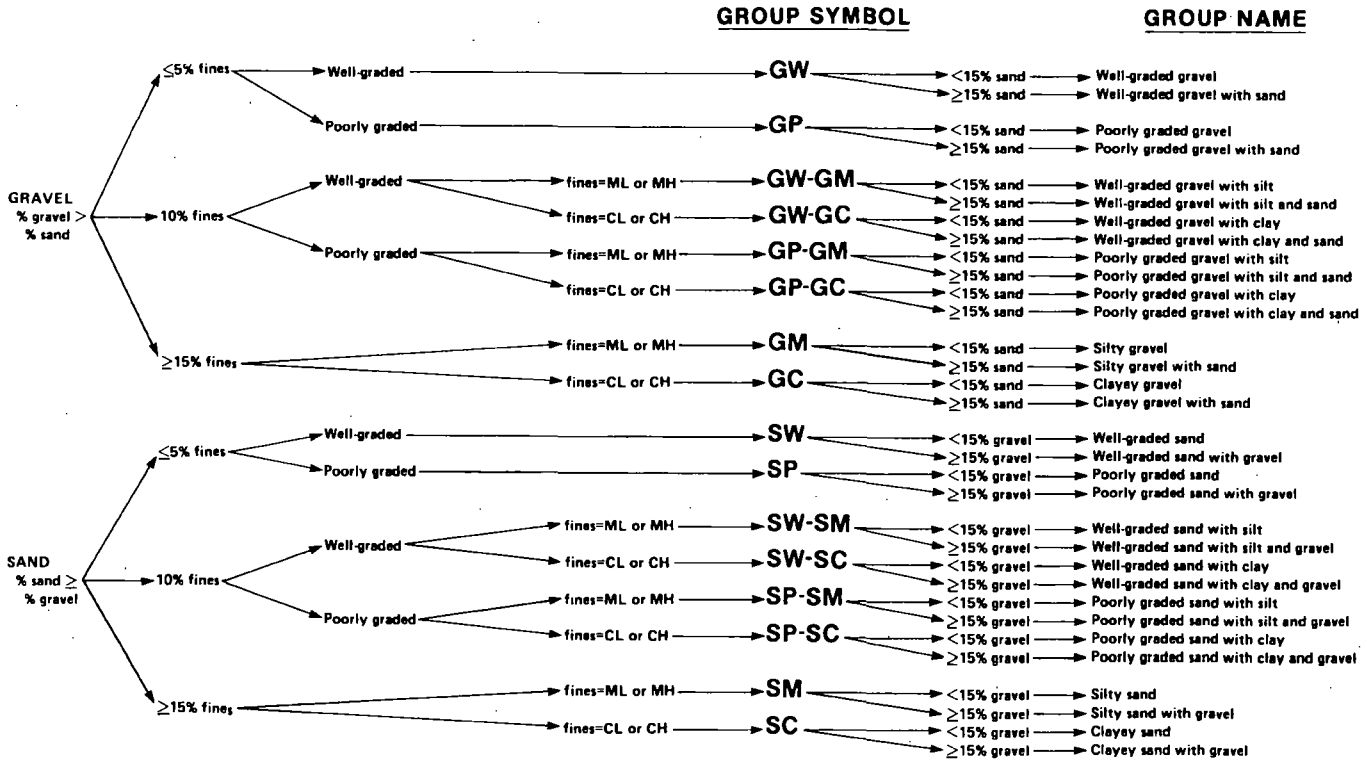
9.1 The sample shall be considered to be representative of the stratum from which it was obtained by an appropriate, accepted, or standard procedure.

NOTE 6—Preferably, the sampling procedure should be identified as having been conducted in accordance with Practices D 1452, D 1587, or D 2113, or Test Method D 1586.

9.2 The sample shall be carefully identified as to origin.

NOTE 7—Remarks as to the origin may take the form of a boring number and sample number in conjunction with a job number, a geologic stratum, a pedologic horizon or a location description with respect to a permanent monument, a grid system or a station number and offset with respect to a stated centerline and a depth or elevation.

9.3 For accurate description and identification, the minimum amount of the specimen to be examined shall be in accordance with the following schedule:



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 2 Flow Chart for Identifying Coarse-Grained Soils (less than 50 % fines)

Maximum Particle Size, Sieve Opening	Minimum Specimen Size, Dry Weight
4.75 mm (No. 4)	100 g (0.25 lb)
9.5 mm (⅜ in.)	200 g (0.5 lb)
19.0 mm (¾ in.)	1.0 kg (2.2 lb)
38.1 mm (1½ in.)	8.0 kg (18 lb)
75.0 mm (3 in.)	60.0 kg (132 lb)

NOTE 8—If random isolated particles are encountered that are significantly larger than the particles in the soil matrix, the soil matrix can be accurately described and identified in accordance with the preceeding schedule.

9.4 If the field sample or specimen being examined is smaller than the minimum recommended amount, the report shall include an appropriate remark.

10. Descriptive Information for Soils

10.1 *Angularity*—Describe the angularity of the sand (coarse sizes only), gravel, cobbles, and boulders, as angular, subangular, subrounded, or rounded in accordance with the criteria in Table 1 and Fig. 3. A range of angularity may be stated, such as: subrounded to rounded.

10.2 *Shape*—Describe the shape of the gravel, cobbles, and boulders as flat, elongated, or flat and elongated if they meet the criteria in Table 2 and Fig. 4. Otherwise, do not mention the shape. Indicate the fraction of the particles that have the shape, such as: one-third of the gravel particles are flat.

10.3 *Color*—Describe the color. Color is an important property in identifying organic soils, and within a given locality it may also be useful in identifying materials of similar geologic origin. If the sample contains layers or patches of varying colors, this shall be noted and all representative colors shall be described. The color shall be described for moist samples. If the color represents a dry condition, this shall be stated in the report.

TABLE 1 Criteria for Describing Angularity of Coarse-Grained Particles (see Fig. 3)

Description	Criteria
Angular	Particles have sharp edges and relatively plane sides with unpolished surfaces
Subangular	Particles are similar to angular description but have rounded edges
Subrounded	Particles have nearly plane sides but have well-rounded corners and edges
Rounded	Particles have smoothly curved sides and no edges

10.4 *Odor*—Describe the odor if organic or unusual. Soils containing a significant amount of organic material usually have a distinctive odor of decaying vegetation. This is especially apparent in fresh samples, but if the samples are dried, the odor may often be revived by heating a moistened sample. If the odor is unusual (petroleum product, chemical, and the like), it shall be described.

10.5 *Moisture Condition*—Describe the moisture condition as dry, moist, or wet, in accordance with the criteria in Table 3.

10.6 *HCl Reaction*—Describe the reaction with HCl as none, weak, or strong, in accordance with the critera in Table 4. Since calcium carbonate is a common cementing agent, a report of its presence on the basis of the reaction with dilute hydrochloric acid is important.

10.7 *Consistency*—For intact fine-grained soil, describe the

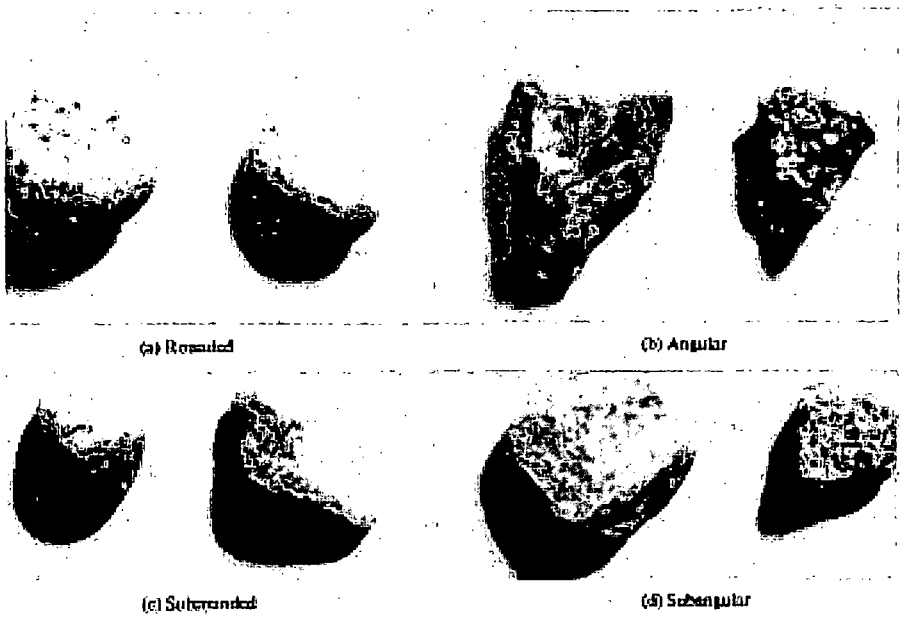


FIG. 3 Typical Angularity of Bulky Grains

TABLE 2 Criteria for Describing Particle Shape (see Fig. 4)

The particle shape shall be described as follows where length, width, and thickness refer to the greatest, intermediate, and least dimensions of a particle, respectively.

Flat	Particles with width/thickness > 3
Elongated	Particles with length/width > 3
Flat and elongated	Particles meet criteria for both flat and elongated

consistency as very soft, soft, firm, hard, or very hard, in accordance with the criteria in Table 5. This observation is inappropriate for soils with significant amounts of gravel.

10.8 *Cementation*—Describe the cementation of intact coarse-grained soils as weak, moderate, or strong, in accordance with the criteria in Table 6.

10.9 *Structure*—Describe the structure of intact soils in accordance with the criteria in Table 7.

10.10 *Range of Particle Sizes*—For gravel and sand components, describe the range of particle sizes within each component as defined in 3.1.2 and 3.1.6. For example, about 20 % fine to coarse gravel, about 40 % fine to coarse sand.

10.11 *Maximum Particle Size*—Describe the maximum particle size found in the sample in accordance with the following information:

10.11.1 *Sand Size*—If the maximum particle size is a sand size, describe as fine, medium, or coarse as defined in 3.1.6. For example: maximum particle size, medium sand.

10.11.2 *Gravel Size*—If the maximum particle size is a gravel size, describe the maximum particle size as the smallest sieve opening that the particle will pass. For example, maximum particle size, 1½ in. (will pass a 1½-in. square opening but not a ¾-in. square opening).

10.11.3 *Cobble or Boulder Size*—If the maximum particle size is a cobble or boulder size, describe the maximum dimension of the largest particle. For example: maximum dimension, 18 in. (450 mm).

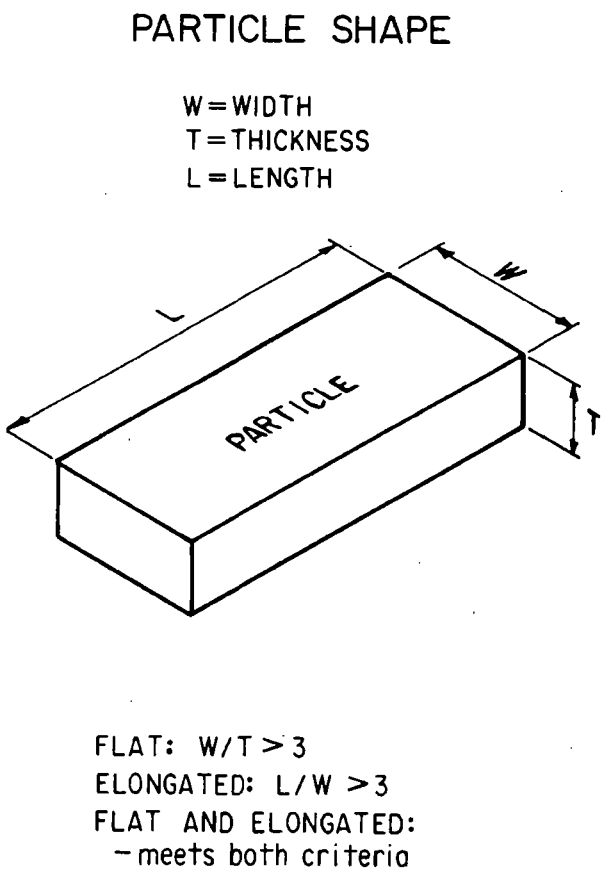


FIG. 4 Criteria for Particle Shape

10.12 *Hardness*—Describe the hardness of coarse sand and larger particles as hard, or state what happens when the

TABLE 3 Criteria for Describing Moisture Condition

Description	Criteria
Dry	Absence of moisture, dusty, dry to the touch
Moist	Damp but no visible water
Wet	Visible free water, usually soil is below water table

TABLE 4 Criteria for Describing the Reaction With HCl

Description	Criteria
None	No visible reaction
Weak	Some reaction, with bubbles forming slowly
Strong	Violent reaction, with bubbles forming immediately

TABLE 5 Criteria for Describing Consistency

Description	Criteria
Very soft	Thumb will penetrate soil more than 1 in. (25 mm)
Soft	Thumb will penetrate soil about 1 in. (25 mm)
Firm	Thumb will indent soil about ¼in. (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very hard	Thumbnail will not indent soil

TABLE 6 Criteria for Describing Cementation

Description	Criteria
Weak	Crumbles or breaks with handling or little finger pressure
Moderate	Crumbles or breaks with considerable finger pressure
Strong	Will not crumble or break with finger pressure

TABLE 7 Criteria for Describing Structure

Description	Criteria
Stratified	Alternating layers of varying material or color with layers at least 6 mm thick; note thickness
Laminated	Alternating layers of varying material or color with the layers less than 6 mm thick; note thickness
Fissured	Breaks along definite planes of fracture with little resistance to fracturing
Slickensided	Fracture planes appear polished or glossy, sometimes striated
Blocky	Cohesive soil that can be broken down into small angular lumps which resist further breakdown
Lensed	Inclusion of small pockets of different soils, such as small lenses of sand scattered through a mass of clay; note thickness
Homogeneous	Same color and appearance throughout

particles are hit by a hammer, for example, gravel-size particles fracture with considerable hammer blow, some gravel-size particles crumble with hammer blow. "Hard" means particles do not crack, fracture, or crumble under a hammer blow.

10.13 Additional comments shall be noted, such as the presence of roots or root holes, difficulty in drilling or augering hole, caving of trench or hole, or the presence of mica.

10.14 A local or commercial name or a geologic interpretation of the soil, or both, may be added if identified as such.

10.15 A classification or identification of the soil in accordance with other classification systems may be added if identified as such.

11. Identification of Peat

11.1 A sample composed primarily of vegetable tissue in various stages of decomposition that has a fibrous to amorphous texture, usually a dark brown to black color, and an organic odor, shall be designated as a highly organic soil and shall be identified as peat, PT, and not subjected to the identification procedures described hereafter.

12. Preparation for Identification

12.1 The soil identification portion of this practice is based on the portion of the soil sample that will pass a 3-in. (75-mm) sieve. The larger than 3-in. (75-mm) particles must be removed, manually, for a loose sample, or mentally, for an intact sample before classifying the soil.

12.2 Estimate and note the percentage of cobbles and the percentage of boulders. Performed visually, these estimates will be on the basis of volume percentage.

NOTE 9—Since the percentages of the particle-size distribution in Test Method D 2487 are by dry weight, and the estimates of percentages for gravel, sand, and fines in this practice are by dry weight, it is recommended that the report state that the percentages of cobbles and boulders are by volume.

12.3 Of the fraction of the soil smaller than 3 in. (75 mm), estimate and note the percentage, by dry weight, of the gravel, sand, and fines (see Appendix X4 for suggested procedures).

NOTE 10—Since the particle-size components appear visually on the basis of volume, considerable experience is required to estimate the percentages on the basis of dry weight. Frequent comparisons with laboratory particle-size analyses should be made.

12.3.1 The percentages shall be estimated to the closest 5 %. The percentages of gravel, sand, and fines must add up to 100 %.

12.3.2 If one of the components is present but not in sufficient quantity to be considered 5 % of the smaller than 3-in. (75-mm) portion, indicate its presence by the term *trace*, for example, trace of fines. A trace is not to be considered in the total of 100 % for the components.

13. Preliminary Identification

13.1 The soil is *fine grained* if it contains 50 % or more fines. Follow the procedures for identifying fine-grained soils of Section 14.

13.2 The soil is *coarse grained* if it contains less than 50 % fines. Follow the procedures for identifying coarse-grained soils of Section 15.

14. Procedure for Identifying Fine-Grained Soils

14.1 Select a representative sample of the material for examination. Remove particles larger than the No. 40 sieve (medium sand and larger) until a specimen equivalent to about a handful of material is available. Use this specimen for performing the dry strength, dilatancy, and toughness tests.

14.2 *Dry Strength*:

14.2.1 From the specimen, select enough material to mold into a ball about 1 in. (25 mm) in diameter. Mold the material until it has the consistency of putty, adding water if necessary.

14.2.2 From the molded material, make at least three test specimens. A test specimen shall be a ball of material about ½ in. (12 mm) in diameter. Allow the test specimens to dry in air, or sun, or by artificial means, as long as the temperature does not exceed 60°C.

14.2.3 If the test specimen contains natural dry lumps, those that are about ½ in. (12 mm) in diameter may be used in place of the molded balls.

NOTE 11—The process of molding and drying usually produces higher strengths than are found in natural dry lumps of soil.

14.2.4 Test the strength of the dry balls or lumps by crushing between the fingers. Note the strength as none, low, medium, high, or very high in accorance with the criteria in Table 8. If natural dry lumps are used, do not use the results of any of the lumps that are found to contain particles of coarse sand.

14.2.5 The presence of high-strength water-soluble cementing materials, such as calcium carbonate, may cause exceptionally high dry strengths. The presence of calcium carbonate can usually be detected from the intensity of the reaction with dilute hydrochloric acid (see 10.6).

14.3 *Dilatancy*:

14.3.1 From the specimen, select enough material to mold into a ball about ½ in. (12 mm) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.

14.3.2 Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally, striking the side of the hand vigorously against the other hand several times. Note the reaction of water appearing on the surface of the soil. Squeeze the sample by closing the hand or pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the criteria in Table 9. The reaction is the speed with which water appears while shaking, and disappears while squeezing.

14.4 *Toughness*:

14.4.1 Following the completion of the dilatancy test, the test specimen is shaped into an elongated pat and rolled by hand on a smooth surface or between the palms into a thread about ⅛ in. (3 mm) in diameter. (If the sample is too wet to roll easily, it should be spread into a thin layer and allowed to lose some water by evaporation.) Fold the sample threads and reroll repeatedly until the thread crumbles at a diameter of about ⅛ in. The thread will crumble at a diameter of ⅛ in. when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, the pieces should be lumped together and kneaded until the lump crumbles. Note the toughness of the material during kneading.

14.4.2 Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in Table 10.

TABLE 8 Criteria for Describing Dry Strength

Description	Criteria
None	The dry specimen crumbles into powder with mere pressure of handling
Low	The dry specimen crumbles into powder with some finger pressure
Medium	The dry specimen breaks into pieces or crumbles with considerable finger pressure
High	The dry specimen cannot be broken with finger pressure. Specimen will break into pieces between thumb and a hard surface
Very high	The dry specimen cannot be broken between the thumb and a hard surface

TABLE 9 Criteria for Describing Dilatancy

Description	Criteria
None	No visible change in the specimen
Slow	Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing
Rapid	Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing

14.5 *Plasticity*—On the basis of observations made during the toughness test, describe the plasticity of the material in accordance with the criteria given in Table 11.

14.6 Decide whether the soil is an *inorganic* or an *organic* fine-grained soil (see 14.8). If inorganic, follow the steps given in 14.7.

14.7 *Identification of Inorganic Fine-Grained Soils*:

14.7.1 Identify the soil as a *lean clay*, CL, if the soil has medium to high dry strength, no or slow dilatancy, and medium toughness and plasticity (see Table 12).

14.7.2 Identify the soil as a *fat clay*, CH, if the soil has high to very high dry strength, no dilatancy, and high toughness and plasticity (see Table 12).

14.7.3 Identify the soil as a *silt*, ML, if the soil has no to low dry strength, slow to rapid dilatancy, and low toughness and plasticity, or is nonplastic (see Table 12).

14.7.4 Identify the soil as an *elastic silt*, MH, if the soil has low to medium dry strength, no to slow dilatancy, and low to medium toughness and plasticity (see Table 12).

NOTE 12—These properties are similar to those for a lean clay. However, the silt will dry quickly on the hand and have a smooth, silky feel when dry. Some soils that would classify as MH in accordance with the criteria in Test Method D 2487 are visually difficult to distinguish from lean clays, CL. It may be necessary to perform laboratory testing for proper identification.

14.8 *Identification of Organic Fine-Grained Soils*:

14.8.1 Identify the soil as an *organic soil*, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soils usually have a dark brown to black color and may have an organic odor. Often, organic soils will change color, for example, black to brown, when exposed to the air. Some organic soils will lighten in color significantly when air dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

NOTE 13—In some cases, through practice and experience, it may be possible to further identify the organic soils as organic silts or organic clays, OL or OH. Correlations between the dilatancy, dry strength, toughness tests, and laboratory tests can be made to identify organic soils in certain deposits of similar materials of known geologic origin.

TABLE 10 Criteria for Describing Toughness

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

TABLE 11 Criteria for Describing Plasticity

Description	Criteria
Nonplastic	A ⅛-in. (3-mm) thread cannot be rolled at any water content
Low	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rerolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit

TABLE 12 Identification of Inorganic Fine-Grained Soils from Manual Tests

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

14.9 If the soil is estimated to have 15 to 25 % sand or gravel, or both, the words "with sand" or "with gravel" (whichever is more predominant) shall be added to the group name. For example: "lean clay with sand, CL" or "silt with gravel, ML" (see Fig. 1a and Fig. 1b). If the percentage of sand is equal to the percentage of gravel, use "with sand."

14.10 If the soil is estimated to have 30 % or more sand or gravel, or both, the words "sandy" or "gravelly" shall be added to the group name. Add the word "sandy" if there appears to be more sand than gravel. Add the word "gravelly" if there appears to be more gravel than sand. For example: "sandy lean clay, CL", "gravelly fat clay, CH", or "sandy silt, ML" (see Fig. 1a and Fig. 1b). If the percentage of sand is equal to the percent of gravel, use "sandy."

15. Procedure for Identifying Coarse-Grained Soils

(Contains less than 50 % fines)

15.1 The soil is a *gravel* if the percentage of gravel is estimated to be more than the percentage of sand.

15.2 The soil is a *sand* if the percentage of gravel is estimated to be equal to or less than the percentage of sand.

15.3 The soil is a *clean gravel* or *clean sand* if the percentage of fines is estimated to be 5 % or less.

15.3.1 Identify the soil as a *well-graded gravel*, GW, or as a *well-graded sand*, SW, if it has a wide range of particle sizes and substantial amounts of the intermediate particle sizes.

15.3.2 Identify the soil as a *poorly graded gravel*, GP, or as a *poorly graded sand*, SP, if it consists predominantly of one size (uniformly graded), or it has a wide range of sizes with some intermediate sizes obviously missing (gap or skip graded).

15.4 The soil is either a *gravel with fines* or a *sand with fines* if the percentage of fines is estimated to be 15 % or more.

15.4.1 Identify the soil as a *clayey gravel*, GC, or a *clayey sand*, SC, if the fines are clayey as determined by the procedures in Section 14.

15.4.2 Identify the soil as a *silty gravel*, GM, or a *silty sand*, SM, if the fines are silty as determined by the procedures in Section 14.

15.5 If the soil is estimated to contain 10 % fines, give the soil a dual identification using two group symbols.

15.5.1 The first group symbol shall correspond to a clean gravel or sand (GW, GP, SW, SP) and the second symbol shall correspond to a gravel or sand with fines (GC, GM, SC, SM).

15.5.2 The group name shall correspond to the first group symbol plus the words "with clay" or "with silt" to indicate the plasticity characteristics of the fines. For example: "well-graded gravel with clay, GW-GC" or "poorly graded sand with silt, SP-SM" (see Fig. 2).

15.6 If the specimen is predominantly sand or gravel but contains an estimated 15 % or more of the other coarse-grained constituent, the words "with gravel" or "with sand" shall be added to the group name. For example: "poorly graded gravel with sand, GP" or "clayey sand with gravel, SC" (see Fig. 2).

15.7 If the field sample contains any cobbles or boulders, or both, the words "with cobbles" or "with cobbles and boulders" shall be added to the group name. For example: "silty gravel with cobbles, GM."

16. Report

16.1 The report shall include the information as to origin, and the items indicated in Table 13.

NOTE 14—*Example: Clayey Gravel with Sand and Cobbles, GC—About 50 % fine to coarse, subrounded to subangular gravel; about 30 % fine to coarse, subrounded sand; about 20 % fines with medium plasticity, high dry strength, no dilatancy, medium toughness; weak reaction with HCl; original field sample had about 5 % (by volume) subrounded cobbles, maximum dimension, 150 mm.*

In-Place Conditions—Firm, homogeneous, dry, brown

Geologic Interpretation—Alluvial fan

TABLE 13 Checklist for Description of Soils

1. Group name
2. Group symbol
3. Percent of cobbles or boulders, or both (by volume)
4. Percent of gravel, sand, or fines, or all three (by dry weight)
5. Particle-size range:
   Gravel—fine, coarse
   Sand—fine, medium, coarse
6. Particle angularity: angular, subangular, subrounded, rounded
7. Particle shape: (if appropriate) flat, elongated, flat and elongated
8. Maximum particle size or dimension
9. Hardness of coarse sand and larger particles
10. Plasticity of fines: nonplastic, low, medium, high
11. Dry strength: none, low, medium, high, very high
12. Dilatancy: none, slow, rapid
13. Toughness: low, medium, high
14. Color (in moist condition)
15. Odor (mention only if organic or unusual)
16. Moisture: dry, moist, wet
17. Reaction with HCl: none, weak, strong

For intact samples:

18. Consistency (fine-grained soils only): very soft, soft, firm, hard, very hard
19. Structure: stratified, laminated, fissured, slickensided, lensed, homogeneous
20. Cementation: weak, moderate, strong
21. Local name
22. Geologic interpretation
23. Additional comments: presence of roots or root holes, presence of mica, gypsum, etc., surface coatings on coarse-grained particles, caving or sloughing of auger hole or trench sides, difficulty in augering or excavating, etc.

ATTACHMENT A3

FIELD FORMS

LIST OF FIELD FORMS

Surface Sediment/Soil Collection Form

Sediment Core Log

Chain of Custody/Laboratory Analysis Request Form

Field Change Request and Corrective Action Record

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SURFACE SEDIMENT/SOIL COLLECTION FORM

Project Name:_____ **Project No.** _____ **Page:**_____

Date:_____ **Crew:**_____

Weather:_____

Sampling Method:_____

Time:_____ **Station:** _____ **Replicate:**_____ **Acceptable grab:** ☐ Yes ☐ No

Bottom Depth:_____ **Penetration Depth:**_____ **RPD Depth:**_____

Analyses before homogenization: ☐ **VOC** ☐ **Sulfides** ☐ **Other**

Sample ID:_____

Type: ☐ cobble ☐ gravel ☐ sand C M F ☐ silt clay ☐ organic matter ☐ wood/shell fragments

Color: ☐ drab olive ☐ gray ☐ black ☐ brown ☐ brown surface

Odor: ☐ none ☐ slight ☐ moderate ☐ strong ☐ sulfidic ☐ petroleum ☐ other

Comments:

Time:_____ **Station:** _____ **Replicate:**_____ **Acceptable grab:** ☐ Yes ☐ No

Bottom Depth:_____ **Penetration Depth:**_____ **RPD Depth:**_____

Analyses before homogenization: ☐ **VOC** ☐ **Sulfides** ☐ **Other**

Sample ID:_____

Type: ☐ cobble ☐ gravel ☐ sand C M F ☐ silt clay ☐ organic matter ☐ wood/shell fragments

Color: ☐ drab olive ☐ gray ☐ black ☐ brown ☐ brown surface

Odor: ☐ none ☐ slight ☐ moderate ☐ strong ☐ sulfidic ☐ petroleum ☐ other

Comments:

Time:_____ **Station:** _____ **Replicate:**_____ **Acceptable grab:** ☐ Yes ☐ No

Bottom Depth:_____ **Penetration Depth:**_____ **RPD Depth:**_____

Analyses before homogenization: ☐ **VOC** ☐ **Sulfides** ☐ **Other**

Sample ID:_____

Type: ☐ cobble ☐ gravel ☐ sand C M F ☐ silt clay ☐ organic matter ☐ wood/shell fragments

Color: ☐ drab olive ☐ gray ☐ black ☐ brown ☐ brown surface

Odor: ☐ none ☐ slight ☐ moderate ☐ strong ☐ sulfidic ☐ petroleum ☐ other

Comments:

Time:_____ **Station:** _____ **Replicate:**_____ **Acceptable grab:** ☐ Yes ☐ No

Bottom Depth:_____ **Penetration Depth:**_____ **RPD Depth:**_____

Analyses before homogenization: ☐ **VOC** ☐ **Sulfides** ☐ **Other**

Sample ID:_____

Type: ☐ cobble ☐ gravel ☐ sand C M F ☐ silt clay ☐ organic matter ☐ wood/shell fragments

Color: ☐ drab olive ☐ gray ☐ black ☐ brown ☐ brown surface

Odor: ☐ none ☐ slight ☐ moderate ☐ strong ☐ sulfidic ☐ petroleum ☐ other

Comments:

integral consulting inc.

SEDIMENT CORE LOG

PROJECT: _____ **Core ID:** _____ pg ___of ____

Collected:		**Processed:**	
Date: / /2010	Drive Length: _____	Date: / /2010	
Time: _____	Tide Level (CRD): _____	Time: _____	
Recovery Length: _____	Mudline Depth: _____	Core Length: _____	
Recovery Efficiency: _____	Vessel: _____	Location: _____	
Crew: _____		Crew: _____	

Depth in Core (cm)	Lithologic Description: (Grainsize, color, density/consistency, odor, organics, debris)	Grain Size (%)			Photo ID	Sample ID
		G	S	Si/Cl		

Core segment breaks at (cm):

CHAIN OF CUSTODY/LABORATORY ANALYSIS REQUEST FORM

Page _____ of _____ Turn Around Requested: _____

Report to:	Phone:	Analyses Requested										Notes/Comments
Company:	Fax:											
Address:	Proj Name:											
City:	Proj Number:											
State:	Sampler:											
Zipcode:												

Sample ID	Sample Date	Sample Time	Sample Matrix	No. Containers											

Relinquished: (Signature)	Received by: (Signature)	Special Instructions/Notes
Printed name:	Printed name:	
Company:	Company:	Number of Coolers:
		Cooler Temp(s):
Date: Time:	Date: Time:	COC Seals Intact?
		Bottles Intact?

	FIELD CHANGE REQUEST	Project Number:

Field Change No.
Page _____ to

Project Number:
Project Name:

CHANGE REQUEST
Applicable Reference:
Description of Change:

Reason for Change:

Impact on Present and Completed Work:

Requested by:
Date: ____/____/

(Field Scientist)

Acknowledged by:
Date: ____/____/

(Field Task Leader)

FIELD OPERATIONS MANAGER RECOMMENDATION

Recommended Disposition:

Recommendation by:
Date: ____/____/

(Sampling and Analysis Coordinator)

PROJECT MANAGER APPROVAL

Final Deposition:

Approved/Disapproved by:
Date: ____/____/

(CERCLA Coordinator)

CORRECTIVE ACTION RECORD

Page ___ of

Audit Report No. :_____ Date:

Report Originator:

Person Responsible for Response:

DESCRIPTION OF PROBLEM:

Date and Time Problem Recognized: _____ By:

Date of Actual Occurrence: _____ By:

Analyte: _____ Analytical Method:

Cause of Problem:

CORRECTIVE ACTION PLANNED:

Person Responsible for Corrective Action:

Date of Corrective Action:

Corrective Action Plan Approval: _____ Date:

DESCRIPTION OF FOLLOW-UP ACTIVITIES:

Person Responsible for Follow-up Activities:

Date of Follow-up Activity:

Final Corrective Action Approval: _____ Date:

APPENDIX B

LABORATORY QUALITY ASSURANCE MANUALS (ON CD)

Superfund Records Center
SITE: Centredale
BREAK: _____
OTHER: _____

integral consulting inc.

Integral Consulting Inc.
45 Exchange Street
Suite 200
Portland, Maine 04101

telephone: 207.874.9000
facsimile: 207.874.7800
www.integral-corp.com

August 4, 2010

Project No. C656-0003

Via Electronic and Regular Mail
Ms. Anna Krasko
Project Manager
United States Environmental Protection Agency
5 Post Office Square, Suite 100
Mail Code OSRR07-1
Boston, MA 02109-3912

Subject: **Errata Sheet for the June 21, 2010 Sampling and Analysis Plan: Supplemental Investigation of the Lyman Mill Reach Sediment and Flood Plain Soils**

Dear Ms. Krasko:

The attached errata sheet provides written responses to the comments you emailed to me on July 1, 2010 regarding Integral's June 21, 2010 Sampling and Analysis Plan: Supplemental Investigation of the Lyman Mill Reach Sediment and Flood Plain Soils (SAP). Additionally, where necessary, the errata sheet identifies where in SAP changes are needed to comply with the comment. As we discussed, given the small number of changes required to the SAP, the submission of an errata sheet should suffice. Therefore, Integral does not intend on submitting a revised SAP.

Should you have any questions regarding the errata sheet, please contact me at (207) 874-9000 ext. 206.

Sincerely,

Patrick O. Gwinn
Senior Managing Scientist

cc: Louis Maccarone – RIDEM
Deirdre Dahlen – Battelle
Jeffrey Karp – Sullivan & Worcester, LLP
David Scotti – Loureiro Engineering Associates, Inc.

APPENDIX B

LABORATORY QUALITY ASSURANCE MANUALS

QUALITY ASSURANCE MANUAL

Columbia Analytical Services, Inc.

1317 South 13th Avenue
Kelso, Washington 98626
(360) 577-7222

Effective Date: October 30, 2009

Laboratory Director/Technical Director: _____
Jeff Christian

Quality Assurance Manager: _____
Julie Gish

Technical Director – Metals: _____
Jeff Coronado

Technical Director – Metals R & D: _____
Nicholas Bloom

Technical Director – Organics: _____
J eff Grindstaff

Technical Director – Organic Extractions: _____
Dr. Gregory Salata

Technical Director – Inorganics/Microbiology: _____
Harv ey Jacky

PROPRIETARY
CAS
CONFIDENTIAL
UNCONTROLLED
COPY

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DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

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3.0 INTRODUCTION AND COMPANY QUALITY ASSURANCE POLICY

Columbia Analytical Services, Inc. (CAS) is an employee-owned professional analytical services laboratory which performs chemical and microbiological analyses on a wide variety of sample matrices, including drinking water, groundwater, surface water, wastewater, soil, sludge, sediment, tissue, industrial and hazardous waste, and other material.

Quality Management Systems are established, implemented and maintained by management. Systems are designed so that there will be sufficient Quality Assurance (QA) activities conducted in the laboratory to ensure that all analytical data generated and processed will be scientifically sound, legally defensible, of known and documented quality, and will accurately reflect the material being tested. Quality Systems are applicable to all fields of testing in which the laboratory in involved.

This goal is achieved by ensuring that adequate Quality Control (QC) procedures are used throughout the monitoring process, and by establishing a means to assess performance of these Quality Control and other QA activities. Policies and procedures are established in order to meet the quality objectives of clients, accrediting authorities, and certifying organizations. Columbia Analytical Services, Inc. is committed to operate in accordance to: ISO/IEC 17025:2005 International Standards, The NELAC Institute (TNI) National Environmental Laboratory Accreditation Program (NELAP), and DoD Environmental Laboratory Accreditation Program. Quality Systems are established to meet the requirements of these standards.

Laboratory management is committed to continually improve the effectiveness of its quality systems and to ensure that all tests are carried out in accordance to customer requirements. Key elements of this commitment are set fourth in the *Columbia Analytical Services, Inc. Quality and Ethics Policy Statement March* 2009 and in this *Kelso Quality Assurance Manual* (QAM). We recognize that quality assurance requires a commitment to quality by everyone in the organization - individually, within each operating unit, and throughout the entire laboratory.

Columbia Analytical maintains control of analytical results by adhering to written standard operating procedures (SOPs) and by observing sample custody requirements. All analytical results are calculated and reported in units consistent with project specifications to allow comparability of data.

Columbia Analytical is a network of laboratories. In addition to the Kelso, WA facility, to which this manual is applicable, Columbia Analytical also operates laboratories in California, Florida, New York, Arizona, and Texas.

The information in this document has been organized according to the format described in *EPA Requirements for Quality Management Plans, EPA QA/R-2,* USEPA, 2001; *EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5,* USEPA, 2001, and *ISO17025 International Standard.*

4.0 PROGRAM DESCRIPTION

The purpose of the QA program at Columbia Analytical is to ensure that our clients are provided with analytical data that is scientifically sound, legally defensible, and of known and documented quality. The concept of Quality Assurance can be extended, and is expressed in the mission statement of Columbia Analytical:

> "The mission of Columbia Analytical Services, Inc. is to provide high quality, cost-effective, and timely professional testing services to our customers. We recognize that our success as a company is based on our ability to maintain customer satisfaction. To do this requires constant attention to customer needs, maintenance of state-of-the-art testing capabilities and successful management of our most important asset - our people - in a way that encourages professional growth, personal development and company commitment."

4.1 Quality Management Systems

In support of this mission, the Kelso laboratory has developed Quality Management Systems to ensure all products and services meet our client's needs. These systems incorporate the requirements of ISO17025 standards. Quality Management Systems Include:

- Standard Operating Procedures
- Sample Management
- Chain of Custody Procedures
- Statistical Control Charting
- Standards Traceability
- Core Ethics Training
- Document Control
- Corrective Action Program
- Management Reviews
- Demonstration of Capability

The effectiveness of the Quality Management System is assessed in several ways:

- Internal and External Audits covering all aspects of the organization
- Annual Management Reviews
- Analysis of Customer Complaints
- Internal and External Proficiency Testing

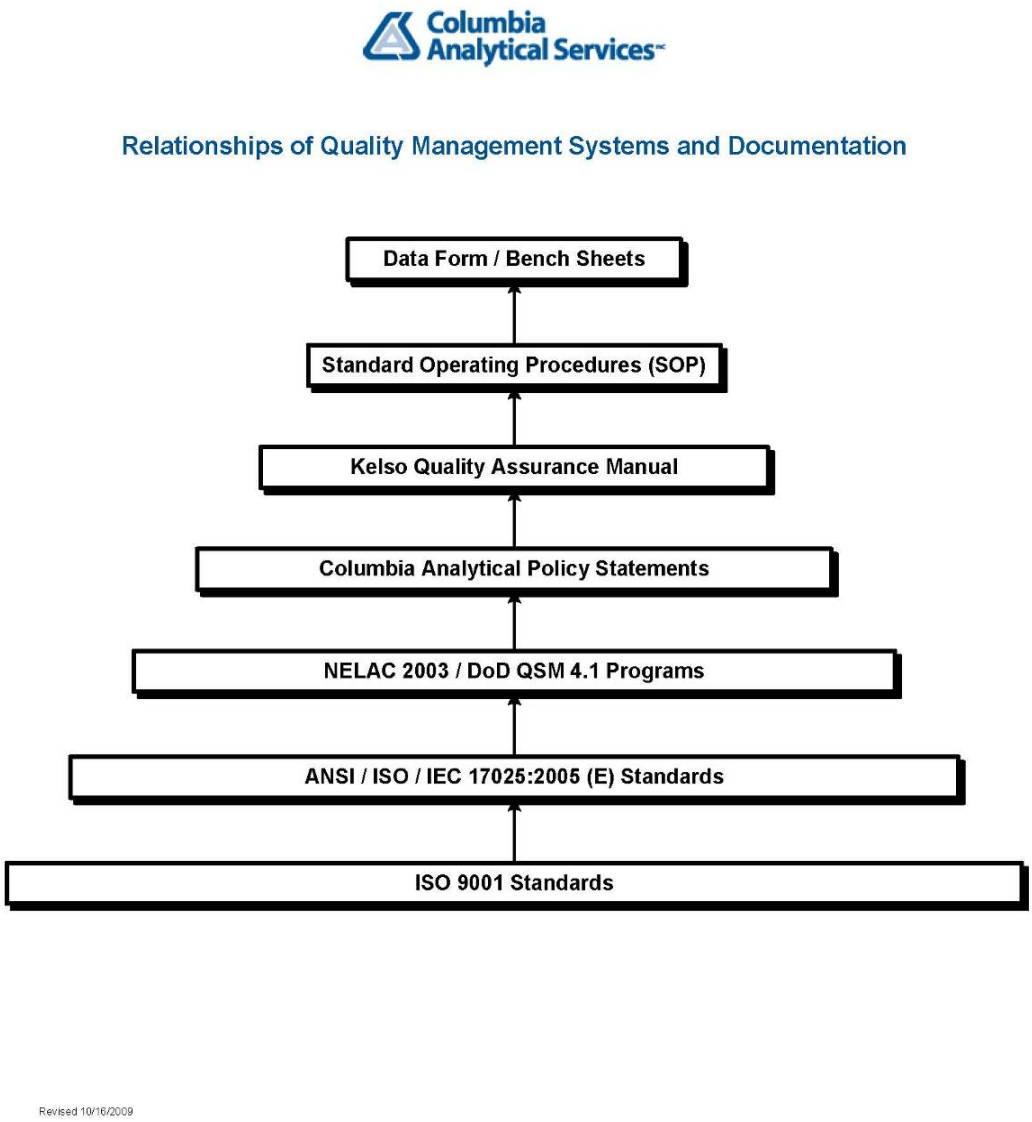


Figure 4-1

Kelso Quality Management Systems are based upon ISO 17025:2005 standards. Fundamental programs (NELAC 2003 and DoD QSM) are based upon these standards. Implementation and documentation against these standards are communicated in corporate policy statements, and Kelso's Quality Assurance Manual. Actual procedures, actions and documentation are defined in both administrative and technical SOP's.

4.2 Facilities and Equipment

Columbia Analytical features over 45,000 square feet of laboratory and administrative workspace. The laboratory has been designed and constructed to provide safeguards against cross-contamination of samples and is arranged according to work function, which enhances the efficiency of analytical operations. The ventilation system has been specially designed to meet the needs of the analyses performed in each work space. Also, Columbia Analytical minimizes laboratory contamination sources by employing janitorial and maintenance staff to ensure that good housekeeping and facilities maintenance are performed. In addition, the segregated laboratory areas are designed for safe and efficient handling of a variety of sample types. These specialized areas (and access restrictions) include:

- Shipping and Receiving/Purchasing
- Sample Management Office, including controlled-access sample storage areas
- Inorganic/Metals Sample Preparation Laboratories (2)
- Inorganic/Metals “clean room” sample preparation laboratory
- ICP-AES Laboratory
- ICP-MS Laboratory
- AA Laboratory
- Metals R&D Laboratory
- Water Chemistry & General Chemistry Laboratories (3)
- Semi-volatile Organics Sample Preparation Laboratory
- Gas Chromatography/High Performance Liquid Chromatography Laboratories
- Gas Chromatography/Mass Spectrometry Laboratory
- Petroleum Hydrocarbon Laboratory
- Semi-volatile Organics Drinking Water Laboratories (2)
- Volatile Organics Laboratory
    - Separate sample preparation laboratory
    - Access by semi-volatile sample preparation staff only after removing lab coat and solvent-contaminated gloves, etc.
- Microbiology Laboratory
- Laboratory Deionized Water Systems (2)
- Laboratory Management, Client Service, Report Generation and Administration
- Data Archival, Data Review and support functions areas
- Information Technology (IT) and LIMS

In addition, the designated areas for sample receiving, refrigerated sample storage, dedicated sample container preparation and shipping provide for the efficient and safe handling of a variety of sample types. Figure 4-1 shows the facility floor plan. The laboratory is equipped with state-of-the-art analytical and administrative support equipment. The equipment and instrumentation are appropriate for the procedures in use. Appendix C lists the major equipment, illustrating the laboratory's overall capabilities and depth.

4.3 Technical Elements of the Quality Assurance Program

The laboratory’s technical procedures are based upon procedures published by various agencies or organizations (See Section 18). The Quality Assurance Program provides to the laboratory organization, procedures, and policies by which the laboratory operates. The necessary certifications and approvals administe red by external agencies are maintained by the QA department. This includes method approvals and audit administration. In addition,

internal audits are performed to assess compliance with policies and procedures. Standard Operating Procedures (SOPs) are maintained for technical and administrative functions. A document control system is used for SOPs, as well as laboratory notebooks, and this QA Manual. A list of QA Program documents is provided in Appendix A.

Acceptable calibration procedures are defined in the SOP for each test procedure. Calibration procedures for other laboratory equipment (balances, thermometers, etc.) are also defined. Quality Control (QC) procedures are used to monitor the testing performed. Each analytical procedure has associated QC requirements to be achieved in order to demonstrate data quality. The use of method detection limit studies, control charting, technical training and preventative maintenance procedures further ensure the quality of data produced. Proficiency Testing (PT) samples are used as an external means of monitoring the quality and proficiency of the laboratory. PT samples are obtained from qualified vendors and are performed on a regular basis. In addition to method proficiency, documentation of analyst training is performed to ensure proficiency and competency of laboratory analysts and technicians. Sample handling and custody procedures are defined in SOPs. Procedures are also in place to monitor the sample storage areas. The technical elements of the QA program are discussed in further detail in later sections of this QA manual.

4.4 Operational Assessments

The laboratory uses a number of systems to assess its daily operations. In addition to the routine quality control (QC) measurements, the senior laboratory management examines a number of other indicators to assess the overall ability of the laboratory to successfully perform analyses for its clients including; On-time performance, customer complaints, training reports and non-conformity reports. A frequent, routine assessment must also be made of the laboratory's facilities and resources in anticipation of accepting an additional or increased workload.

Columbia Analytical utilizes a number of different methods to ensure that adequate resources are available in anticipation of the demand for service. Regularly scheduled senior staff meetings, tracking of outstanding proposals and an accurate, current synopsis of incoming work all assist the senior staff in properly allocating resources to achieve the required results. All Requests for Proposal (RFP) documents are reviewed by the Project Chemist and appropriate managerial staff to identify any project specific requirements that differ from the standard practices of the laboratory. Any requirements that cannot be met are noted and communicated to the client, as well as requesting the client to provide any project specific Quality Assurance Plans (QAPPs) if available. A weekly status meeting is also conducted with the laboratory staff by the Client Services Manager to inform the staff of the status of incoming work, future projects, or project requirements.

4.5 Document Control

Procedures for control and maintenance of documents are described in the *SOP for Document Control (ADM-DOC_CTRL).* The requirements of the SOP apply to all standards preparation logbooks, instrument maintenance logbooks, run logbooks, certificates of analysis, standard operating procedures (SOPs), quality assurance manuals (QAMs), quality assurance project plans (QAPPs), Environmental Health & Safety (EHS) manuals, and other controlled Columbia Analytical documents.

Each controlled copy of a controlled document will be released only after a document control number is assigned and the recipient is recorded on a document distribution list. Filing and distribution is performed by the Quality Assurance Manager, or designee, and ensure that only the most current version of the document is distributed and in use. A document control number is assigned to logbooks. Completed logbooks that are no longer in use are archived in a master logbook file.

Columbia Analytical maintains a records system that ensures all laboratory records (including raw data, reports, and supporting records) are retained and available. The archiving system is described in the *SOP for Data Archiving (ADM-ARCH).*

4.6 Subcontracting

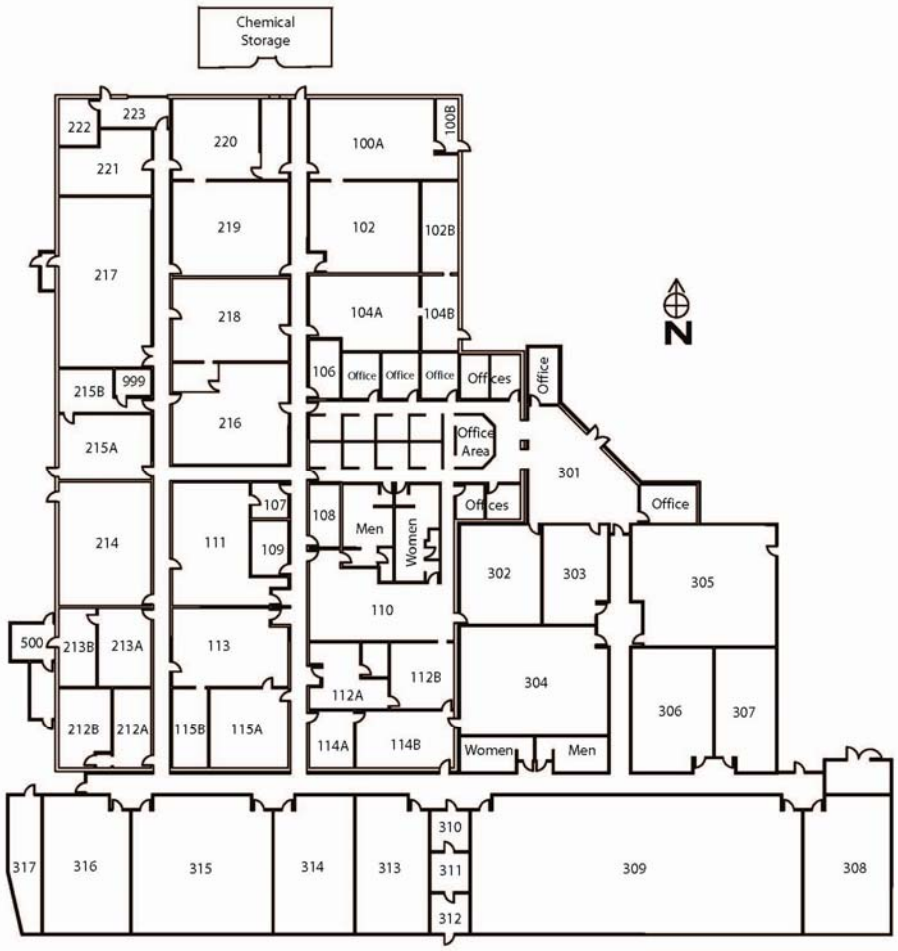
Analytical services are subcontracted when Columbia Analytical/Kelso needs to balance workload or when the requested analyses are not performed by Columbia Analytical/Kelso. Subcontracting is only done with the knowledge and approval of the client and to qualified laboratories. Subcontracting to another Columbia Analytical laboratory is preferred over external-laboratory subcontracting. Further, sub-contracting is done using capable and qualified laboratories. Established procedures are used to qualify external subcontract laboratories. These procedures are described in the *SOP for Qualification of Subcontract Laboratories (ADM-SUBLAB).* The Corporate Quality Assurance staff is responsible for qualifying and oversight of subcontract laboratories.

4.7 Procurement

The quality level of reagents and materials (grade, traceability, etc.) required is specified in analytical SOPs. Department supervisors ensure that the proper materials are purchased. Inspection and verification of material ordered is performed at the time of receipt by receiving personnel. The receiving staff labels the material with the date received. Expiration dates are assigned (by the laboratory user) as appropriate for the material. Storage conditions and expiration dates are specified in the analytical SOP. Supplies and services that are critical in maintaining the quality of laboratory testing are procured from pre-approved vendors. The policy and procedure for purchasing and procurement are described in the *SOP for Purchasing through CAS Purchasing Department in Kelso (SOP ADM-PUR).* Also, refer to section 10.4 for a discussion of reference materials.

Figure 4-2
Columbia Analytical/Kelso Laboratory Floor Plan

Room #	
100A	Semivolatile GC Lab
100B	Electrical Room
102	Semivolatile GC Lab
102B	LC Lab
104A	Petroleum Hydrocarbon Lab
104B	Petroleum Hydrocarbon Lab
106	Deionized Water System Room
107	Storage
108	Zero Headspace Extraction
109	Storage
110	Lunch Room
111	General Chemistry Lab
112A	Microbiology Lab
112B	Copy Center
113	Metals Lab
114A	Sample Preparation
114B	Sample Preparation
115A	Metals Lab
115B	Metals Lab
212A	Metals Lab
212B	Low Level Mercury Lab
213A	Sample Preparation
213B	Sample Preparation
214	General Chemistry Lab
215A	Drinking Water Lab
215B	IT
216	Data Review Area
217	Sample Storage
218	Volatiles Lab
219	Semivolatile GC/MS Lab
220	Semivolatile GC/MS Lab
221	Drinking Water Lab
222	Purchasing
223	General Receiving
301	Lobby
302	Lunch Room
303	Conference Room
304	Sample Storage
305	Sample Receiving
306	Metals R & D
307	Pharmaceutical B
308	Pharmaceutical A
309	Organics Extractions Lab
310	Communication Room
311	Electrical Room
312	Fire Room/Water System
313	Glass Room
314	Clean Room
315	Metals Digestion Room
316	Tissue Preparation
317	Mechanical
500	Janitorial Room
999	Computer Room



5.0 PROFESSIONAL CONDUCT AND ETHICAL PRACTICES

One of the most important aspects of the success of Columbia Analytical is the emphasis placed on the integrity of the data provided and services per formed. To promote product quality, employees are required to comply with certain standards of conduct and ethical practices. The following examples of Columbia Analytical policy are representative of these standards, and are not intended to be limiting or all-inclusive:

- Under no circumstances is the willful act of fraudulent manipulation of analytical data condoned. Such acts are to be reported immediately to senior management for appropriate corrective action. Unless specifically required in writing by a client, alteration, deviation or omission of written contractual requirements is not permitted. Such changes must be in writing and approved by senior management.
- Falsification of data in any form will not be tolerated. While much analytical data is subject to professional judgment and interpretation, outright falsification, whenever observed or discovered, will be documented, and appropriate remedies and punitive measures will be taken toward those individuals responsible. Employee discipline is progressive in its severity and each situation is handled individually in that the discipline is designed to fit the circumstances. Potential disciplinary actions may include a verbal warning, written warning, a second written notice (more severe and more strongly worded than a warning), suspension without pay, demotion, or termination.
- It is the responsibility of all Columbia Analyt ical employees to safeguard sensitive company and client information. The nature of our business and the well being of our company and of our clients is dependent upon protecting and maintaining proprietary company/client information. All information, data, and reports (except that in the public domain) collected or assembled on behalf of a client is treated as confidential. Information may not be given to third parties without the consent of the client. Unauthorized release of confidential information about the company or its clients is taken seriously and is subject to formal disciplinary action.

All employees are required to sign and adhere to the requirements set forth in the Columbia Analytical *Confidentiality and Conflicts of Interest Employee Agreement* and the Columbia Analytical *Commitment to Excellence in Data Quality Policy.* All employees receive in-house ethics training and are periodically reminded of their data quality and ethical conduct responsibilities.

Columbia Analytical makes every attempt to ensure that employees are free from any commercial, financial, or other undue pressures that might affect their quality of work. Related policies are described in the Columbia Analytical Employee Handbook. This includes the Columbia Analytical Ombudsman Program, the Columbia Analytical Open Door Policy, and the use of flexible work hours. Operational assessments are regularly made to ensure that project planning is performed and that adequate resources are available during anticipated periods of increased workloads (Section 4.3). Procedures for subcontracting work are established, and within the Columbia Analytical laboratory network additional capacity is typically available for subcontracting, if necessary.

6.0 ORGANIZATION AND RESPONSIBILITIES

The Columbia Analytical/Kelso staff, consisting of approximately 130 employees, includes chemists, technicians and support personnel. They represent diverse educational backgrounds and experience, and provide the comprehensive skills that the laboratory requires. During seasonal workload increases, additional temporary employees may be hired to perform specific tasks.

Columbia Analytical is committed to providing an environment that encourages excellence. Everyone within Columbia Analytical shares responsibility for maintaining and improving the quality of our analytical services. The responsibilities of key personnel within the laboratory are described below. Table 6-1 lists the Columbia Analytical/Kelso personnel assigned to these key positions. Managerial staff members are provided the authority and resources needed to perform their duties. An organizational chart of the laboratory, as well as the resumes of these key personnel, can be found in Appendix B.

- The role of the **Laboratory Director** is to provide technical, operational, and administrative leadership through planning, allocation and management of personnel and equipment resources. The Laboratory Director provides leadership and support for the QA program and is responsible for overall laboratory efficiency and the financial performance of the Kelso facility. The Laboratory Director has the authority to stop work in response to quality problems. The Laboratory Director also provides resources for implementation of the QA program, reviews and approves this QA Manual, reviews and approves standard operating procedures (SOPs), and provides support for business development by identifying and developing new markets through continuing support of the management of existing client activities.

- The responsibility of the **Quality Assurance Manager** (QAM) is to oversee implementation of the quality program and to coordinate QA activities within the laboratory. The QAM works with laboratory production units to establish effective quality control and assessment plans. The QAM has the authority to stop work in response to quality problems. The QAM is responsible for maintaining the QA Manual and performing an annual review of it; reviewing and approving SOPs and coordinating the annual review of each SOP; maintaining QA records such as metrological records, archived logbooks, PT sample results, etc.; document control; conducting PT sample studies; approving nonconformity and corrective action reports; maintaining the laboratory's certifications and approvals; performing internal QA audits; preparing QA activity reports; etc. The QAM reports directly to the Laboratory Director. The QAM also interacts with the Columbia Analytical Quality Assurance Director. It is important to note that when evaluating data, the QAM does so in an objective manner and free of outside, or managerial, influence.

  The Chief Quality Officer (CQO) is responsible for the overall QA program at all the Columbia Analytical laboratories. The CQO is responsible for ensuring that annual internal audits are performed at each Columbia Analytical laboratory; maintaining a data base of information about state certifications and accreditation programs; writing laboratory-wide SOPs; maintaining a data base of Columbia Analytical-approved subcontract laboratories; providing assistance to the laboratory QA staff and laboratory managers; preparing a quarterly QA activity report; etc.

- In the case of absence of the Laboratory Director or QA Manager, deputies are assigned to act in that role. Default deputies for these positions are the Client Services Manager or Organics Department Manager (for the Laboratory Director) and the CQO or Laboratory Director (for the QA Manager).

- The **Environmental Health and Safety Officer** (EH&S) is responsible for the administration of the laboratory health and safety policies. This includes the formulation and implementation of safety policies, the supervision of new-employee safety training, the review of accidents, incidents and prevention plans, the monitoring of hazardous waste disposal and the conducting of departmental safety inspections. The EH&S officer is also designated as the Chemical Hygiene Officer. The EH&S Officer has a dotted-line reporting responsibility to Columbia Analytical's EH&S Director.

- The **Client Services and Sample Management Office Manager** is responsible for the Client Services Department (customer services/project chemists, and Electronic Data Deliverables group) and the sample management office/bottle preparation sections. The Client Services Department provides a complete interface with clients from initial project specification to final deliverables. The sample management office handles all the activities associated with receiving, storage, and disposal of samples. The Client Services Manager has the authority to stop subcontractor work in response to quality problems.

- The **Project Chemist** is a senior-level scientist assigned to each client to act as a technical liaison between the client and the laboratory. The project chemist is responsible for ensuring that the analyses performed by the laboratory meet all project, contract, and regulatory-specific requirements. This entails coordinating with the Columbia Analytical laboratory and administrative staff to ensure that client-specific needs are understood, and that the services Columbia Analytical provides are properly executed and satisfy the requirements of the client.

- The Analytical Laboratory is divided into operational units based upon specific disciplines. Each department is responsible for establishing, maintaining and documenting a quality control program based upon the unique requirements within the department. Each **Department Manager and Supervisor** has the responsibility to ensure that quality control functions are carried out as planned, and to guarantee the production of high quality data. Department managers and bench-level supervisors have the responsibility to monitor the day-to-day operations to ensure that productivity and data quality objectives are met. Each department manager has the authority to stop work in response to quality problems in their area. Analysts have the responsibility to carry out testing according to prescribed methods, SOPs, and quality control guidelines particular to the laboratory in which he/she is working.

- The **Sample Management Office** plays a key role in the laboratory QA program by maintaining documentation for all samples received by the laboratory, and by assisting in the archival of all laboratory results. The sample management office staff is also responsible for the proper disposal of samples after analysis.

- **Information Technology** (IT) staff are responsible for the administration of the Laboratory Information Management System (LIMS) and other necessary support services. Other functions of the IT staff include laboratory network maintenance, IT systems development and implementation, education of analytical staff in the use of scientific software, Electronic Data Deliverable (EDD) generation, and data back-up, archival and integrity operations.

Table 6-1
Summary of Technical Experience and Qualifications

Personnel	Years of Experience	Project Role
Jeff Christian, B.S.	30	Laboratory Director
Julie Gish, M.S.	18	Quality Assurance Manager
Lynda Huckestein, B.S.	20	Client Services Manager Sample Management Office Manager
Jeff Coronado, B.S.	19	Metals Department Manager
Nicolas Bloom, M. S.	29	Metals R & D Manager
Harvey Jacky, B.S.	20	General Chemistry Department Manager
Gregory Salata, Ph.D.	9	Extractions Department Manager
Jeff Grindstaff, B.S.	20	Organics Chromatography & Mass Spectrometry Department Manager
Loren Portwood, B.S.	18	Organics Drinking Water Department Manager
Eileen Arnold, B.A.	27	Environmental Health and Safety Officer
Mike Sullivan, B.S.	8	Information Technology Director
Lee Wolf, B.S.	23	Chief Quality Officer
Steve Vincent, B.S.	33	President

7.0 INFORMATION MANAGEMENT

The generation, compilation, reporting, and archiving of electronic data is a critical component of laboratory operations. In order to generate data of known and acceptable quality, the quality assurance systems and quality control practices for electronic data systems must be complete and comprehensive and in keeping with the overall quality assurance objectives of the organization. Columbia Analytical management provides the tools and resources to implement electronic data systems and establishes information technology standards and policies. Appendix C lists major automated data processing equipment.

7.1 Software Quality Assurance Plan

Columbia Analytical has defined practices for assuring the quality of the computer software used throughout all laboratory operations to generate, compile, report, and store electronic data. These practices are described in the *CAS Software Quality Assurance Plan (SQAP)*. The purpose of the SQAP is to describe the policies and practices for the procurement, configuration management, development, validation and verification, data security, maintenance, and use of computer software. The policies and practices described in the plan apply to purchased computer software as well as to internally developed computer software. Key components of this plan are policies for software validation and control.

7.2 IT Support

The local Columbia Analytical Information Technology (IT) department is established to provide technical support for all computing systems. The IT department staff continually monitors the performance and output of operating systems. The IT department oversees routine system maintenance and data backups to ensure the integrity of all electronic data. A software inventory is maintained. Additional IT responsibilities are described in the SQAP.

In addition to the local IT department, Columbia Analytical corporate IT provides support for network-wide systems. Columbia Analytical also has personnel assigned to information management duties such as development and implementation of reporting systems; data acquisition, and Electronic Data Deliverable (EDD) generation.

7.3 Information Management Systems

Columbia Analytical has various systems in place to address specific data management needs. The Columbia Analytical Laboratory Information Management System (LIMS) is used to manage sample information and invoicing. Access is controlled by password. This system defines sample identification, analysis specifications, and provides a means of sample tracking. This system is used during sample login to generate the internal service request. Included on the service request is a summary of client information, sample identification, required analyses, work instructions, deliverable requirements. The LIMS is used to track the status of a sample and is important in maintaining internal chain of custody.

Where possible, instrument data acquired locally is immediately moved to a server (Microsoft Windows2003® domain). This provides a reliable, easily maintained, high-volume acquisition and storage system for electronic data files. With password entry, users may access the system from many available computer stations, improving efficiency and flexibility. The server is also used for data reporting, EDD generation, and administrative functions. Access to these systems is controlled by password. A standardized EDI (electronic data interchange) format is used as a reporting platform, providing functionality and flexibility for end users. With a common standardized communication platform, the EDI provides data reporting in a variety of hardcopy and electronic deliverable formats, including Staged Electronic Data Deliverable (SEDD) format.

7.4 Backup and Security

Columbia Analytical laboratory data is either acquired directly to the centralized acquisition server or acquired locally and then transferred to the server. All data is eventually moved to the centralized data acquisition server for reporting and archiving. Differential backups are performed on all file server information once per day, Sunday through Thursday. Full backups are performed each Friday night. Tapes are physically stored in a locked media cabinet within a locked, temperature controlled computer room, with every other full backup also securely stored offsite.

Access to sample information and data is on a need-to-know basis. Access is restricted to the person's areas of responsibility. Passwords are required on all systems. No direct external, non- Columbia Analytical access is allowed to any of our network systems.

The external e-mail system and Internet access is established via a single gateway to discourage unauthorized entry. Columbia Analytical uses a closed system for company e-mail. Files, such as electronic deliverables, are sent through the external e-mail system only via a trusted agent. The external messaging system operates through a single secure gateway. Email attachments sent in and out of the gateway are subject to a virus scan. Because the Internet is not regulated, we use a limited access approach to provide a firewall for added security. Virus screening is performed continuously on all network systems.

8.0 SAMPLE MANAGEMENT

8.1 Sampling and Sample Preservation

The quality of analytical results is highly dependent upon the quality of the procedures used to collect, preserve and store samples. Columbia Analytical recommends that clients follow sampling guidelines described in 40 CFR 136, 40 CFR 141, USEPA SW-846, and state-specific sampling guidelines, if applicable. Sampling factors that must be taken into account to insure accurate, defensible analytical results include:

- Amount of sample taken
- Type of container used
- Type of sample preservation
- Sample storage time
- Proper custodial documentation

Columbia Analytical uses the sample preservation, container, and holding-time recommendations published in a number of documents. The primary documents of reference are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IV for hazardous waste samples; USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, and Supplements; EPA 40CFR parts 136 and 141; and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples (see Section 18 for complete citations). The container, preservation and holding time information for these references is summarized in Table 8-1 for soil, water, and drinking water. The current EPA CLP Statement of Work should be referred to for CLP procedures. Where allowed by project sampling and analysis protocols (such as Puget Sound Protocols) the holding time for sediment, soil, and tissue samples may be extended for a defined period when stored frozen at -20°C.

Columbia Analytical routinely provides sample containers with appropriate preservatives for our clients. Containers are purchased as precleaned to a level 1 status, and conform to the requirements for samples established by the USEPA. Certificates of analysis for the sample containers are available to clients if requested. Reagent water used for sampling blanks (trip blanks, etc.) and chemical preservation reagents are tested by the laboratory to ensure that they are free of interferences and documented. Our sample kits typically consist of foam-lined, precleaned shipping coolers, (cleaned inside and out with appropriate cleaner, rinsed thoroughly and air-dried), specially prepared and labeled sample containers individually wrapped in protective material, (VOC vials are placed in a specially made, foam holder), chain-of-custody (COC) forms, and custody seals. Container labels and custody seals are provided for each container.

Figure 8-1 shows the chain-of-custody form routinely used at Columbia Analytical and included with sample kits. For large sample container shipments, the containers may be shipped in their original boxes. Such shipments will consist of several boxes of labeled sample containers and sufficient materials (bubble wrap, COC forms, custody seals, shipping coolers, etc.) to allow the sampling personnel to process the sample containers and return them to Columbia Analytical. The proper preservative is added to the sample containers prior to shipment, unless otherwise instructed by the client.

If any returning shipping cooler exhibits an odor or other abnormality after receipt and subsequent decontamination by laboratory personnel, a second, more vigorous decontamination process is employed. Contai ners exhibiting an odor or abnormality after the second decontamination process are promptly and properly discarded. Columbia Analytical keeps client-specific shipping requirements on file and utilizes major transportation carriers to guarantee that sample shipping requirements (same-day, overnight, etc.) are met. Columbia Analytical also provides courier service that makes regularly scheduled trips to the Greater Portland, Oregon Metropolitan area.

When Columbia Analytical ships environmental samples to other laboratories for analysis each sample bottle is wrapped in protective material and placed in a plastic bag (preferably Ziploc®) to avoid any possible cross-contamination of samples during shipping. The sample management office (SMO) follows formalized procedures (SMO-GEN) for maintaining the samples' chain of custody, packaging and shipment. Dry ice gel ice is the only temperature preservative used by Columbia Analytical, unless otherwise specified by the client or receiving laboratory.

8.2 Sample Receipt and Handling

Standard Operating Procedures (SMO-GEN) are established for the receiving of samples into the laboratory. These procedures ensure that samples are received and properly logged into the laboratory, and that all associated documentation, including chain of custody forms, is complete and consistent with the samples received.

Once samples are delivered to the Columbia Analytical sample management office (SMO), a Cooler Receipt and Preservation Check Form (CRF - See Figure 8-2 for an example) is used to assess the shipping cooler and its contents as received by the laboratory personnel. Verification of sample integrity includes the following activities:

- Assessment of custody seal presence/absence, location and signature;
- Temperature of sample containers upon receipt;
- Chain of custody documents properly used (entries in ink, signature present, etc.);
- Sample containers checked for integrity (broken, leaking, etc.);

- Sample is clearly marked and dated (bottle labels complete with required information);
- Appropriate containers (size, type) are received for the requested analyses;
- The minimum amount of sample material is provided for the analysis.
- Sample container labels and/or tags agree with chain of custody entries (identification, required analyses, etc.);
- Assessment of proper sample preservation (if inadequate, corrective action is employed); and
- VOC containers are inspected for the presence/absence of bubbles. (Assessment of proper preservation of VOC containers is performed by lab personnel).

Samples are logged into a Laboratory Information Management System (LIMS). Any anomalies or discrepancies observed during the initial assessment are recorded on the CRF and COC documents. Potential problems with a sample shipment are addressed by contacting the client and discussing the pertinent issues. When the Project Chemist and client have reached a satisfactory resolution, the login process may continue and analysis may begin. During the login process, each sample is given a unique laboratory code and a service request form is generated. The LIMS generates a Service Request that contains client information, sample descriptions, sample matrix information, required analyses, sample collection dates, analysis due dates and other pertinent information. The service request is reviewed by the appropriate Project Chemist for accuracy, completeness, and consistency of requested analyses and for client project objectives.

Samples are stored as per method requirements until they undergo analysis, unless otherwise specified, using various refrigerators or freezers, or designated secure areas. Columbia Analytical has five walk-in cold storage units which house the majority of sample containers received at the laboratory. In addition, there are four additional refrigerators, including dedicated refrigerated storage of VOC samples. The dedicated storage areas for VOC samples are monitored using storage blanks, as described in the *SOP for VOA Storage Blanks (VOC-BLAN)*. Columbia Analytical also has seven sub-zero freezers capable of storing samples at -20 ° C primarily used for tissue and sediment samples requiring specialized storage conditions. The temperature of each sample storage unit is monitored daily and the data recorded in a bound logbook. Continuous-graph temperature recorders have also been placed in the walk-in refrigerators to provide a permanent record of the storage conditions to which samples are exposed.

Columbia Analytical adheres to the method-prescribed or project-specified holding times for all analyses. The sampling date and time are entered into the LIMS system at the time of sample receipt and login. Analysts then monitor holding times by obtaining analysis-specific reports from the LIMS. These reports provide holding time information on all samples for the analysis, calculated from the sampling date and the holding time requirement. To document holding time compliance, the date and time analyzed is printed or written on the analytical raw data. For analyses with a holding time prescribed in hours it is essential that the sample collection time is provided, so holding time compliance can be demonstrated. If not, the sample collection time is assumed as the earliest in the day (i.e. the most conservative).

Unless other arrangements have been made in advance, upon completion of all analyses and submittal of the final report, aqueous samples and sample extracts are retained at ambient temperature for 30 days, soil samples are reta ined at ambient temperature for 60 days, and tissue samples are retained frozen for 3 months. Upon expiration of these time limits, the samples are either returned to the client or disposed of according to approved disposal practices. All samples are characterized according to hazardous/non-hazardous waste criteria and are segregated accordingly. All hazardous waste samples are disposed of according to formal procedures outlined in the *CAS Environmental Health and Safety Manual.* All waste produced at the laboratory, including the laboratory's own various hazardous waste streams, is treated in accordance with applicable local and Federal laws. Documentation is maintained for each sample from initial receipt through final disposal to ensure that an accurate history of the sample from "cradle to grave" is available.

8.3 Sample Custody

Sample custody transfer at the time of sample receipt is documented using chain-of-custody (COC) forms accompanying the samples. During sample receipt, it is also noted if custody seals were present. This is described in the *SOP for Sample Receiving (SMO-GEN)*. Figure 8-1 is a copy of the chain-of-custody form routinely used at Columbia Analytical.

Facility security and access is important in maintaining the integrity of samples received at Columbia Analytical/Kelso. Access to the laboratory facility is limited by use of locked exterior doors with a coded entry, except for the reception area and sample receiving doors, which are manned during business hours and locked at all other times. In addition, the sample storage area within the laboratory is a controlled access area with locked doors with a coded entry. The Columbia Analytical facility is equipped with an alarm system and Columbia Analytical employs a private security firm to provide nighttime and weekend security.

A barcoding system is used to document internal sample custody. Each person removing or returning samples from/to sample storage while performing analysis is required to document this custody transfer. The system uniquely identifies the sample container and provides an electronic record of the custody of each sample. For sample extracts and digestates the analyst documents custody of the sample extract or digestate by signing on the benchsheet, or custody record, that they have accepted custody. The procedures are described in the *SOP for Sample Tracking and Internal Chain of Custody (SMO-SCOC).*

8.4 Project Setup

The analytical method(s) used for sample analysis are chosen based on the client's requirements. Unless specified otherwise, the most recent versions of reference methods are used. For SW-846 methods, some projects may require the most recent *promulgated* version, and some projects may require the most recent *published* version. The Project Chemist will ensure that the correct method version is used. LIMS codes are chosen to identify the analysis method used for analysis. The Project Chemist ensures that the correct methods are selected for analysis, deliverable requirements are identified, and due dates are specified on the service request. To communicate and specify project-specific requirements, a Tier V form (Figure 8-3) is used and accompanies the service request form.

Table 8-1
Sample Preservation and Holding Times

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Bacterial Tests				
Coliform, Colilert (Standard Methods)	W, DW	P, Bottle or Bag	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Coliform, Fecal and Total (Standard Methods)	W, DW	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Fecal Streptococci (SM 9230B)	W	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Inorganic Tests				
Acidity (SM 2310B)	W	P,G	Cool, 4°C	14 days[EPA]
Alkalinity (SM 2320B)	W, DW	P,G	Cool, 4°C	14 days[EPA]
Ammonia (SM 4500NH3)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Biochemical Oxygen Demand (SM 5210B)	W	P,G	Cool, 4°C	48 hours
Bromate (EPA 300.1)	W, DW	P,G	50mg/L EDA, cool to 4°C	28 days
Bromide (EPA 300.1)	W, DW	P,G	None Required	28 days
Chemical Oxygen Demand (SM 5220C)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Chloride (EPA 300.0)	W, DW	P,G	None Required	28 days
Chloride (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Chlorine, Total Residual (SM 4500Cl F)	W, DW	P,G	None Required	24 hours
Chlorite (EPA 300.1)	W, DW	P,G	50mg/L EDA, cool to 4°C	14 days
Chlorophyll-A (SM 11200H)	W	G Amber	Cool, 4°C	Analyze immediately
Chromium VI (EPA 7196A)	W	P,G	Cool, 4°C	24 hours
Color (SM 2120B)	W, DW	P,G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination (EPA 335.4, 9010, 9012) (SM 4500CN E,G)	W, DW	P,G	Cool, 4°C, NaOH to pH>12, plus 0.6 g Ascorbic Acid	14 days
Cyanide, Weak Acid Dissociable (SM 4500CN I)	W	P,G	Cool, 4°C, NaOH to pH >12	14 days
Ferrous Iron (CAS SOP)	W, DW	G Amber	Cool, 4°C	24 hours
Fluoride (EPA 300.0)	W, DW	P,G	None Required	28 days
Fluoride (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Hardness (SM 2340C)	W, DW	P,G	HNO_3 to pH<2	6 months
Hydrogen Ion (pH) (SM 4500H B)	W, DW	P,G	None Required	Analyze immediately
Kjeldahl and Organic Nitrogen (ASTM D3590-89)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Nitrate (EPA 300.0)	W, DW	P,G	Cool, 4°C	48 hours
Nitrate (EPA 353.2)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	48 hours
Nitrate (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Nitrate-Nitrite (EPA 353.2)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrite (EPA 300.0)	W, DW	P,G	Cool, 4°C	48 hours
Nitrite (EPA 353.2)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	48 hours
Nitrite (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Orthophosphate (EPA 365.3)	W, DW	P,G	Cool, 4°C	Analyze immediately
Oxygen, Dissolved (Probe) (SM 4500O G)	W, DW	G, Bottle and Top	None Required	Analyze immediately
Oxygen, Dissolved (Winkler)	W, DW	G, Bottle and Top	Fix on Site and Store in Dark	8 hours
Perchlorate (EPA 314.0)	W, DW	P,G	Protect from temp. extremes	28 days
Phenolics, Total (EPA 420.1)	W	G Only	Cool, 4°C, H_2SO_4 to pH<2	28 days
Phosphorus, Total (EPA 365.3)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Residue, Total (EPA 160.3 & SM 2540B)	W	P,G	Cool, 4°C	7 days
Residue, Filterable (TDS) (SM 2540C)	W	P,G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS) (SM 2540D)	W	P,G	Cool, 4°C	7 days
Residue, Settleable (SM 2540F)	W	P,G	Cool, 4°C	48 hours
Residue, Volatile (EPA 160.4)	W	P,G	Cool, 4°C	7 days
Silica (SM 4500SiO2 C)	W	P Only	Cool, 4°C	28 days
Specific Conductance (EPA 120.1 & SM 2510B)	W, DW	P,G	Cool, 4°C	28 days
Sulfate (EPA 300.0)	W, DW	P,G	Cool, 4°C	28 days
Sulfate (EPA 9056)	W	P,G	Cool, 4°C	Analyze immediately
Sulfide (SM 4500S2 F)	W	P,G	Cool, 4°C, Add Zinc Acetate plus Sodium Hydroxide to pH>9	7 days
Sulfite (SM 4500SO3 B)	W	P,G	None Required	24 hours
Surfactants (MBAS) (SM 5540C)	W	P,G	Cool, 4°C	48 hours
Tannin and Lignin (SM 5550B)	W	P,G	Cool, 4°C	28 days
Turbidity (EPA 180.1)	W, DW	P,G	Cool, 4°C	48 hours

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Metals				
Metals, except CrVI and Mercury	W, DW	P,G	HNO_3 to pH<2	6 months
(EPA 200.7, 200. 8, 200.9, 6010, 6020)	S	G, Teflon-Lined Cap	Cool, 4°C	6 months
Chromium VI (EPA 7195/7191)	W	P,G	Cool, 4°C	24 hours
Mercury	W	P,G	HNO_3 to pH<2	28 days
(EPA 245.1, 7470, 7471)	S	P,G	Cool, 4°C	28 days
1631E	W	F	Cool, 4°C, HCl or H_2SO_4 to pH<2	90 days
1631E	S	F	Freeze < -15ºC	1 Yr
Methyl Mercury 1630	W	F	HCL to pH<2	6 months
Organic Tests				
Oil and Grease, Hexane Extractable Material (EPA 1664)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Carbon, Total (EPA 415.1, 9060 & SM 5310C)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Halogens, Total (EPA 9020)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2, No headspace	28 days
Organic Halogens, Adsorbable (EPA 1650B)	W	G, Teflon-Lined Cap	Cool, 4°C, HNO_3 to pH<2	6 months
Petroleum Hydrocarbons, Total (EPA 8015)	W	G, Teflon-Lined Cap	Cool, 4°C, HCl or H_2SO_4 to pH<2	7 days until extraction; 40 days after extraction
	S	G, Teflon-Lined Cap	Cool, 4°C	14 days until extraction; 40 days after extraction
Pharma Personal Care Products 1694	W	Amber G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2	14 days until extraction; 40 days after extraction
Nitroaromatics and Nitramines 8330, 8330B	W,S	G, Teflon-Lined Cap	Cool, 4°C	S 14, W 7 days until extraction; 40 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Organic Test				
Methanol in Process Liquid NCASI 94.03	L	G, Teflon-Lined Cap	Cool, 4°C	30 days
HAPS – Condensates NCASI 99.01		G, Teflon-Lined Cap	Cool, 4°C	14/30 days
HAPS – Impinger/Canisters NCASI 99.02			Cool, 4°C	21 days
Perfluorinated Compounds HPLC/MS/MS	W	P	Cool, 4°C	14 days until extraction; 40 days after extraction
PBDE/PBB – ROHS GC/MS			RT	40 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Volatile Organics				
Petroleum Hydrocarbons, Volatile (Gasoline-Range Organics) (EPA 8015)	W	G, Teflon-Lined Septum Cap	Cool, 4°C, HCl to pH<2 No Headspace	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C Minimize Headspace	14 days
Purgeable Halocarbons (EPA 624, 8021, 8260)	W	G, Teflon-Lined Septum Cap, No Headspace	**No Residual Chlorine Present**: HCl to pH<2, Cool, 4°C, No Headspace **Residual Chlorine Present**: 10% $Na_2S_2O_3$, HCl to pH<2, Cool, 4°C	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4°C Sodium Bisulfate Cool, 4°C	7 days 48 hrs to prepare from Encore, 14 days after preparation. 48 hrs to prepare from Encore, 14 days after preparation.
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE) (EPA 624, 8021, 8260)	W	G, Teflon-Lined Septum Cap, No Headspace	**No Residual Chlorine Present**: HCl to pH<2, Cool, 4°C, No Headspace **Residual Chlorine Present**: 10% $Na_2S_2O_3$, HCl to pH<2, Cool 4°C	14 days
	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4°C Sodium Bisulfate Cool, 4°C	7 days 48 hrs to prepare from Encore, 14 days after preparation. 48 hrs to prepare from Encore, 14 days after preparation.
Acrolein, Acrylonitrile, Acetonitrile (EPA 624, 8260)	W	G, Teflon-Lined Septum Cap	Adjust pH to 4-5, Cool, 4°C, No Headspace	14 days
EDB and DBCP (EPA 8260)	W,S	G, Teflon-Lined Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	28 days

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Semivolatile Organics				
Petroleum Hydrocarbons, Extractable (Diesel-Range Organics) (EPA 8015)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Alcohols and Glycols (EPA 8015)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Acid Extractable Semivolatile Organics (EPA 625, 8270)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Base/Neutral Extractable Semivolatile Organics (EPA 625, 8270)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Polynuclear Aromatic Hydrocarbons (EPA 625, 8270, 8310)	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction
Organochlorine Pesticides and PCBs (EPA 608, 8081, GC/MS/MS)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Organophosphorus Pesticides (EPA 8141, GC/MS/MS)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Nitrogen- and Phosphorus-Containing Pesticides (EPA 8141)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Herbicides (EPA 8151)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Organotins (CAS SOP)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Chlorinated Phenolics (EPA 1653A)	W	G, Teflon-Lined Cap	H_2SO_4 to pH<2, Cool, 4°C[g]	30 days until extraction; 30 days after extraction
Resin and Fatty Acids (NCASI 85.02)	W	G, Teflon-Lined Cap	NaOH to pH $\geq$10, Cool, 4°C[g]	30 days until extraction; 30 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Drinking Water Organics				
Purgeable Organics (EPA 524.2)	DW	G, Teflon-Lined Septum Cap	Ascorbic Acid, HCl to pH≤2, Cool, 4°C, No Headspace	14 days
EDB, DBCP, and TCP (EPA 504.1)	DW	G, Teflon-Lined Septum Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	14 days
Carbamates, Carbamoyloximes (EPA 531.1)	DW	G, Amber, Teflon-Lined Cap	1.8 mL monochloroacetic acid to pH<3; 80 mg/L $Na_2S_2O_3$ if Res.Cl.; Cool, 4°C	28 days
Chlorinated Herbicides (EPA 515.4)	DW	G, Amber, Teflon-Lined Cap	If Res.Cl, 2mg/4omL NaS; Cool, <6°C	14 days until extraction; 21 days after extraction
Chlorinated Pesticides (EPA 508.1, 525.2)	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH≤ 2; Cool, 4°C	14 days until extraction; 30 days after extraction
Diquat and Paraquat (EPA 549.2)	DW	G, Amber, Teflon-Lined Cap	100 mg/L $Na_2S_2O_3$ if Res.Cl., Cool, 4°C,	7days until extraction; 21 days after extraction
Endothall (EPA 548.1)	DW	G, Amber, Teflon-Lined Cap	Cool, 4°C	7 days until extraction; 14 days after extraction
Glyphosate (EPA 547)	DW	G, Amber, Teflon-Lined Cap	100 mg/L $Na_2S_2O_3$, Cool, 4°C	14 days
Haloacetic Acids (EPA 552.2)	DW	G, Amber, Teflon-Lined Cap	100 mg/L NH_4Cl, Cool, 4°C	14 days until extraction; 7 days after extraction
Semivolatile Organics (EPA 525.2)	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH≤ 2; Cool, 4°C	14 days until extraction; 30 days after extraction
Nitrosoamines (EPA 521)	DW	G, Amber, Teflon-Lined Cap	Dechlorinate at collection[g] Cool, 4°C	14 days until extraction; 28 days after extraction
Selected Pesticides and Flame Retardants (EPA 527)	DW	G, Amber, Teflon-Lined Cap	See method Cool, 4°C	14 days until extraction; 28 days after extraction
Explosives (EPA 529)	DW	G, Amber, Teflon-Lined Cap	See method Cool, 4°C	14 days until extraction; 30 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Toxicity Characteristic Leaching Procedure (TCLP)				
Semivolatile Organics (EPA 1311/8270)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C, Store in Dark[g] TCLP extract: Cool, 4°C, Store in Dark[g]	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Organochlorine Pesticides (EPA 1311/8081)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Chlorinated Herbicides (EPA 1311/8151)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C TCLP extract: Cool, 4°C	14 days until TCLP ext'n; 7 days until extraction; 40 days after extraction
Mercury (EPA 1311/7470)	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	28 days until extraction; 28 days after extraction
Metals, except Mercury (EPA 1311/6010)	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	180 days until extraction; 180 days after extraction
Volatile Organics (EPA 1311/8260)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C Minimize Headspace TCLP extract: Cool, 4°C, HCl to pH<2, No Headspace	14 days until extraction; 14 days after extraction

a For EPA SW-846 methods the method number is listed generically, without specific revision suffixes.
b DW = Drinking Water, W = Water; S = Soil or Sediment; HW = Hazardous Waste
c P = Polyethylene; G = Glass, F- Fluoropolymer
d For chlorinated water samples
e The maximum holding time is dependent upon the geographical proximity of sample source to the laboratory.
f Fourteen days until extraction for soil, sediment, and sludge samples.
g If the water sample contains residual chlorine, 10% sodium thiosulfate is used to dechlorinate.

Figure 8-1
Chain of Custody Form

Columbia Analytical Services™

CHAIN OF CUSTODY

1317 South 13th Ave. • Kelso, WA 98626 • (360) 577-7222 • (800) 695-7222x07 • FAX (360) 636-1068

SR#: _____

PAGE _____ OF _____ COC #_____

PROJECT NAME
PROJECT NUMBER
PROJECT MANAGER
COMPANY/ADDRESS
CITY/STATE/ZIP
E-MAIL ADDRESS
PHONE # | FAX #
SAMPLER'S SIGNATURE

SAMPLE I.D.	DATE	TIME	LAB I.D.	MATRIX	NUMBER OF CONTAINERS	Semivolatile Organics by GC/MS 625☐ 8270☐ 8270LL☐	Volatile Organics 624☐ 8260☐ 8021☐ BTEX☐	Hydrocarbons (*see below) Gas☐ Diesel☐ Oil☐	☐ Fuel Fingerprint (FIQ) ☐ NW-HCID Screen	Oil & Grease/TRPH 1664 HEM☐ 1664 SGT☐	PCB's Aroclors☐ Congeners☐	Pesticides/Herbicides 608☐ 8081A☐ 8141A☐ 8151A☐	Chlorophenolics - 8151M Tri☐ Tetra☐ PCP☐	PAHS 8310☐ SIM☐	Metals, Total or Dissolved (See list below)	Cyanide☐ Hex-Chrom☐	pH, Cond., Cl, SO4, PO4, F, NO_2, NO_3, BOD, TSS, TDS (circle)	NH_3-N, COD, Total-P, TKN, TOC, DOC (circle) NO_2+NO_3	TOX 9020☐ AOX 1650☐ 506☐					REMARKS

REPORT REQUIREMENTS

___ I. Routine Report: Method Blank, Surrogate, as required
___ II. Report Dup., MS, MSD as required
___ III. Data Validation Report (includes all raw data)
___ IV. CLP Deliverable Report
___ V. EDD

INVOICE INFORMATION

P.O. # _____
Bill To: _____

TURNAROUND REQUIREMENTS

____ 24 hr. ____ 48 hr.
____ 5 Day
____ Standard (10-15 working days)
____ Provide FAX Results

_____
Requested Report Date

Circle which metals are to be analyzed:

Total Metals: Al As Sb Ba Be B Ca Cd Co Cr Cu Fe Pb Mg Mn Mo Ni K Ag Na Se Sr Tl Sn V Zn Hg

Dissolved Metals: Al As Sb Ba Be B Ca Cd Co Cr Cu Fe Pb Mg Mn Mo Ni K Ag Na Se Sr Tl Sn V Zn Hg

*INDICATE STATE HYDROCARBON PROCEDURE: AK CA WI NORTHWEST OTHER:_____ (CIRCLE ONE)

SPECIAL INSTRUCTIONS/COMMENTS:

RELINQUISHED BY:	RECEIVED BY:	RELINQUISHED BY:	RECEIVED BY:
Signature / Date/Time	Signature / Date/Time	Signature / Date/Time	Signature / Date/Time
Printed Name / Firm	Printed Name / Firm	Printed Name / Firm	Printed Name / Firm

RCOC #1 06/08

Figure 8-2

Columbia Analytical Services, Inc.
Cooler Receipt and Preservation Form

PC_____

Client / Project:_____ Service Request *K09*_____

Received:_____ Opened:_____ By:_____

1. Samples were received via? *US Mail* *Fed Ex* *UPS* *DHL* *GH* *GS* *PDX* *Courier* *Hand Delivered*
2. Samples were received in: (circle) *Cooler* *Box* *Envelope* *Other*_____ *NA*
3. Were custody seals on coolers? NA Y N If yes, how many and where?_____
   If present, were custody seals intact? Y N If present, were they signed and dated? Y N
4. Is shipper's air-bill filed? If not, record air-bill number:_____ NA Y N
   _____ _____ _____
5. **Temperature of cooler(s) upon receipt (°C):** _____ _____ _____ _____
   Temperature Blank (°C): _____ _____ _____ _____
   Thermometer ID: _____ _____ _____ _____
6. If applicable, list Chain of Custody Numbers: _____ _____ _____ _____
7. Packing material used. *Inserts* *Baggies* *Bubble Wrap* *Gel Packs* *Wet Ice* *Sleeves* *Other*_____
8. Were custody papers properly filled out (ink, signed, etc.)? NA Y N
9. **Did all bottles arrive in good condition (unbroken)?** *Indicate in the table below.* NA Y N
10. Were all sample labels complete (i.e analysis, preservation, etc.)? NA Y N
11. Did all sample labels and tags agree with custody papers? *Indicate in the table below* NA Y N
12. **Were appropriate bottles/containers and volumes received for the tests indicated?** NA Y N
13. Were the pH-preserved bottles tested* received at the appropriate pH? *Indicate in the table below* NA Y N
14. Were VOA vials and 1631 Mercury bottles received without headspace? *Indicate in the table below.* NA Y N
15. **Are CWA Microbiology samples received with >1/2 the 24hr. hold time remaining from collection?** NA Y N
16. Was C12/Res negative? NA Y N

Sample ID on Bottle	Sample ID on COC	Sample ID on Bottle	Sample ID on COC

Sample ID	Bottle Count	Bottle Type	Out of Temp	Head-space	Broken	pH	Reagent	Volume added	Reagent Lot Number	Initials

*Does not include all pH preserved sample aliquots received. See sample receiving SOP (SMO-GEN).

Additional Notes, Discrepancies, & Resolutions:_____

_____

_____

Page 1 of: *1* *2*

Figure 8-3
Tier V Form

Client :

Project Chemist :

Project Name :

Service Request :

Project Number :

SMO LimsTemplate ID :

Project Description :

QAPP/SOW Information :

Reporting

Tier Level :

PDF:

Report to :

In result field use :

EDD :

Flagging Requirements :

Other Requirements :

Sample Considerations

Sample Limitations :

Sample Prep/Analysis :

Non-Standard Holdtimes :

Historical Data :

Comments :

9.0 ANALYTICAL PROCEDURES

Columbia Analytical employs methods and analytical pr ocedures from a variety of external sources. The primary method references are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IVA, IVB, and online updates for hazardous waste samples, and USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, and Supplements; and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples. Complete citations for these references can be found in Section 18.0. Other published procedures, such as state-specific methods, program-specific methods (such as Puget Sound Protocols), or in-house methods may be used. Several factors are involved with the selection of analytical methods to be used in the laboratory. These include the method detection limit, the concentration of the analyte being measured, method selectivity, accuracy and precision of the method, the type of sample being analyzed, and the regulatory compliance objectives. The implementation of methods by Columbia Analytical is described in SOPs specific to each method. A list of NELAP-accredited methods is given in Appendix E. Further details are described below.

9.1 Standard Operating Procedures (SOPs) and Laboratory Notebooks.

Columbia Analytical maintains SOPs for use in both technical and administrative functions. SOPs are written following standardized format and content requirements. Each SOP is reviewed and approved by a minimum of two managers (the Laboratory Director and/or Department Manager and the Quality Assur ance Manager). All SOPs undergo a documented annual review to make sure current practices are described. The QA Manager maintains a comprehensive list of current SOPs. The document control process ensures that only the most currently prepared version of an SOP is being used. The QA Manual, QAPPs, SOPs, standards preparation logbooks, maintenance logbooks, et al., are controlled documents. The procedures for document control are described in the *SOP for Document Control* (ADM-DOC_CTRL). In addition to SOPs, each laboratory department maintains a current file, accessible to all laboratory staff, of the current methodology used to perform analyses. Laboratory notebook entries are standardized following the guidelines in the *SOP for Making Entries into Logbooks and onto Benchsheets* (ADM-DATANTRY). Entries made into laboratory notebooks are reviewed and approved by the appropriate supervisor at a regular interval.

9.2 Deviation from Standard Operating Procedures

When a customer requests a modification to an SOP (such as a change in reporting limit, addition or deletion of target analyte(s), etc.), the project chemist handling that project must discuss the proposed deviation with the department manager in charge of the analysis and obtain their approval to accept the project. The project chemist is responsible for documenting the approved or allowed deviation from the SO P by placing a detailed description of the deviation attached to the quotation or in the project file and also providing an appropriate comment on the service request when the samples are received.

For circumstances when a deviation or departu re from company policies or procedures involving any non-technical function is found necessary, approval must be obtained from the appropriate supervisor, manager, the laboratory director, or other level of authority. Frequent departure from policy is not encouraged. However, if frequent departure from any policy is noted, the laboratory director will address the possible need for a change in policy.

9.3 Modified Procedures

Columbia Analytical strives to perform published methods as described in the referenced documents. If there is a material deviation from the published method, the method is cited as a "Modified" method in the analytical report. Modifications to the published methods are listed in the standard operating procedure. Standard operating procedures are available to analysts and are also available to our clients for review, especially those for "Modified" methods. Client approval is obtained for the use of "Modified" methods prior to the performance of the analysis.

9.4 Analytical Batch

The basic unit for analytical quality control is the analytical batch. The definition that Columbia Analytical has adopted for the analytical batch is listed below. The overriding principle for describing an analytical batch is that all the samples in a batch, both field samples and quality control samples are to be handled exactly the same way, and all of the data from each analysis is to be manipulated in exactly the same manner. The minimum requirements of an analytical batch are:

1) The number of (field) samples in a batch is not to exceed 20.
2) All (field) samples in a batch are of the same matrix.
3) The QC samples to be processed with the (field) samples include:
   a) Method Blank (a.k.a. Laboratory Reagent Blank)

      Function: Determination of laboratory contamination.

   b) Laboratory Control Sample

      Function: Assessment of method performance

   c) Matrix Spiked (field) Sample (a.k.a. Laboratory Fortified Sample Matrix)*

      Function: Assessment of matrix bias

   d) Duplicate Matrix Spiked (field) Sample or Duplicate (field) Sample (a.k.a. Laboratory Duplicate)*

      Function: Assessment of batch precision

   * A sample identified as a field blank, an equipment blank, or a trip blank is not to be matrix spiked or duplicated.
4) A single lot of reagents is used to process the batch of samples.
5) Each operation within the analysis is performed by a single analyst, technician, chemist, or by a team of analysts/technicians/chemists.

6) Samples are analyzed in a continuous manner over a timeframe not to exceed 24-hours.
7) (Field) samples are assigned to batches commencing at the time that sample processing begins. For example: for analysis of metals, sample processing begins when the samples are digested. For analysis of organic constituents, it begins when the samples are extracted.
8) The QC samples are to be analyzed in conjunction with the associated field samples prepared with them. However, for tests which have a separate sample preparation step that defines a batch (digestion, extraction, etc.), the QC samples in the batch do not require analysis each time a field sample within the preparation batch is analyzed (multiple instrument sequences to analyze all field samples in the batch need not include re-analyses of the QC samples).
9) The batch is to be assigned a unique identification number that can be used to correlate the QC samples with the field samples.
10) Batch QC refers to the QC samples that are analyzed in a batch of (field) samples.
11) Project-specific requirements may be exceptions. If project, program, or method requirements are more stringent than these laboratory minimum requirements, then the project, program, or method requirements will take precedence. However, if the project, program, or method requirements are less stringent than these laboratory minimum requirements, these laboratory minimum requirements will take precedence.

9.5 Specialized Procedures

Columbia Analytical not only strives to provide results that are scientifically sound, legally defensible, and of known and documented quality; but also strives to provide the best solution to analytical challenges. Procedures using specialized instrumentation and methodology have been developed to improve sens itivity (provide lower detection limits), selectivity (minimize interferences while maintaining sensitivity), and overall data quality for low concentration applications. Examples ar e trace-level Mercury and Methylmercury analyses, reductive precipitation metals analysis, specialized GC/MS analyses, LC/MS analyses, and ultra-low level organics analyses (including PAHs, pesticides and PCBs).

9.6 Sample Cleanup

Columbia Analytical commonly employs several cleanup procedures to minimize known common interferences prior to analysis. EPA methods (3620, 3630, 3640, 3660, and 3665) for cleanup of sample extracts for organics analysis are routinely used to minimize or eliminate interferences that may adversely affect sample results and data usability.

10.0 CALIBRATION PROCEDURES AND FREQUENCY

All equipment and instruments used at Columbia Analytical are operated, maintained and calibrated according to the manufacturer's guidelines and recommendations, as well as to criteria set forth in the applicable analytical methodology. Operation and calibration are performed by personnel who have been properly trained in these procedures. Documentation of calibration information is maintained in appropriate reference files. Brief descriptions of the calibration procedures for our major laboratory equipment and instruments are described below. Calibration verification is performed according to the applicable analytical methodology. Calibration verification procedures and criteria are listed in laboratory Standard Operating Procedures. Documentation of calibration verification is maintained in appropriate reference files.

Records are maintained to provide traceability of reference materials.

Laboratory support equipment (thermometers, balances, and weights) are routinely verified on an annual basis by a vendor accredited to A2LA or ISO /IEC 17025:2005 International Standards. All analytical measurements generated at Columbia Analytical are performed using materials and/or processes that are traceable to a reference material. Metrology equipment (analytical balances, thermometers, etc.) is calibrated using reference materials traceable to the National Institute of Standards and Technology (NIST). These primary reference materials are themselves recertified on an annual basis. Vendors used for metrology support are required to verify compliance to International Standards by supplying the laboratory with a copy of their scope of accreditation.

All sampling containers provided to the client by the laboratory are purchased as precleaned (Level 1) containers, with certificates of analysis available for each bottle type. This information is provided to the client when requested.

Equipment subjected to overloading or mishandling, or has been shown by verification to be defective; is taken out of service until it is repaired. The equipment is placed back in service only after verifying, by calibration, that the equipment performs satisfactorily.

10.1 Temperature Control Devices

Temperatures are monitored and recorded for all of the temperature-regulating support equipment such as sample refrigerators, freezers, and standards refrigerators. Bound record books are kept which contain daily-recorded temperatures, identification and location of equipment, acceptance criteria and the initials of the technician who performed the checks. The procedure for performing these measurements is provided in the *SOP for Support Equipment Monitoring and Calibration (SOP ADM-SEMC).* The SOP also includes the use of acceptance criteria and correction factors.

Where the operating temperature is specified as a test condition (such as ovens, incubators, evaporators) the temperature is recorded on the raw data. All thermometers are identified according to serial number, and the calibration is checked annually against a National Institute of Standards and Technology (NIST) certified thermometer. The NIST thermometer is recertified by a vendor accredited to A2LA or ISO/IEC 17025:2005 International Standard on an annual basis.

10.2 Analytical Balances

The calibration of each analytical balance is checked by the user each day of use with three Class S or S-1 weights, which assess the accuracy of the balance at low, mid-level and high levels bracketing the working range. Records are kept which contain the recorded measurements, identification of the balance, acceptance criteria, and the initials of user who performed the check. The procedure for performing these measurements and use of acceptance criteria is described in the SOP ADM-SEMC. The weights are recertified using NIST traceable standards by an accredited metrology organization on an annual basis.

As needed, the balances are recalibrated using the manufacturers recommended operating procedures. Analytical balances are serviced on a semi-annual basis by an accredited metrology organization.

10.3 Water Purification Systems

Columbia Analytical uses two independent water purification systems is designed to produce deionized water meeting method specifications. One system consists of a series of pumps, filters, and resin beds designed to yield deionized water meeting the specifications of ASTM Type II water, and *Standard Methods for the Examination of Water and Wastewater* (SM1080, 20th Ed.) *High Quality* water. Activated carbon filters are also in series with the demineralizers to produce "organic-free" water. A second system consists of pumps, filters, and treatment components designed to yield deionized water meeting the specifications of ASTM Type I water, and *Standard Methods for the Examination of Water and Wastewater* (SM1080, 20th Ed.) *High Quality* water. Following a written SOP, the status of each system is monitored continuously for conductivity and resistivity with an on-line meter and indicator light, and readings recorded daily in a bound record book. The meter accuracy is verified annually. Deionizers are rotated and replaced on a regular schedule. Microbiology water is checked on a daily basis at a point downstream of the purification system at a tap in the laboratory.

10.4 Source and Preparation of Standard Reference Materials

Consumable reference materials routinely purchased by the laboratories (e.g., analytical standards) are purchased from nationally rec ognized, reputable vendors. All vendors have fulfilled the requirements for ISO 9001 certification and/or are accredited by A $_2$LA. Columbia Analytical relies on a primary vendor for the majority of its analytical supplies. Consumable primary stock standards are obtained from certified commercial sources or from sources referenced in a specific method. Supelco, Ultra Scientific, AccuStandard, Chem Services, Inc., Aldrich Chemical Co., Baker, Spex, etc. are exam ples of the vendors used. Reference material information is recorded in the appropriate logbook(s) and materials are stored under conditions that provide maximum protection against deterioration and contamination. The logbook entry includes such information as an assigned logbook identification code, the source of the material (i.e. vendor identification), solvent (if applicable) and concentration of analyte(s), reference to the certificate of analysis and an assigned expiration date. The date that the standard is received in the laboratory is marked on the container. When the reference material is used for the first time, the date of usage and the initials of the analyst are also recorded on the container.

Stock solutions and calibration standard solutions are prepared fresh as often as necessary according to their stability. All standard solutions are properly labeled as to analyte concentration, solvent, date, preparer, and expiration date; these entries are also recorded in the appropriate notebook(s) following the *SOP for Making Entries into Logbooks and onto Benchsheets* (SOP No. ADM-DATANTRY). Prior to sample analysis, all calibration reference materials are verified with a second, independent source of the material (see section 11.3.5).

10.5 Inductively Coupled Plasma-Atomic Emission Spectrograph (ICP-AES)

Each emission line on the ICP is calibrated daily against a blank and against standards. Analyses of calibration standards, initial and continuing calibration verification standards, and inter-element interference check samples are carried out as specified in the applicable method SOP and analytical method (i.e. EPA 200.7, 6010B, 6010C, CLP SOW, etc.).

10.6 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)

Each element of interest is calibrated for using a blank and a single standard. Prior to calibration, a short-term stability check is performed on the system. Following calibration, an independent check standard is analyzed, and a continuing calibration verification standard (CCV) is analyzed with every ten samples.

10.7 Atomic Absorption Spectrophotometers (AAS)

These instruments are calibrated daily using a minimum of four standards and a blank. Calibration is validated using reference standards, and is verified at a minimum frequency of once every ten samples. Initial calibration points cannot be “dropped” from the resulting calibration curve.

10.8 GC/MS Systems

All GC/MS instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise) using procedures outlined in Standard Operating Procedures and/or appropriate USEPA method citations. All reference materials used for this function are vendor-certified standards. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method. For isotope dilution procedures, the internal standard response(s) and labeled compound recovery must meet method criteria. Method-specific instrument tuning is regularly checked using bromofluorobenzene (BFB) for volatile organic chemical (VOC) analysis, or decafluorotriphenylphosphine (DFTPP) for semi-volatile analysis. Mass spectral peaks for the tuning compounds must conform both in mass numbers and in relative intensity criteria before analyses can proceed. Calibration policies for organics chromatographic analyses are described in the *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

10.9 Gas Chromatographs and High Performance Liquid Chromatographs

Calibration and standardization follow SOP guidelines and/or appropriate USEPA method citations. All GC and HPLC instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise). The lowest standard is equivalent to the method reporting limit; additional standards define the working

range of the GC or LC detector. Results are used to establish response factors (or calibration curves) and retention-time windows for each analyte. Calibration is verified at a minimum frequency of once every ten samples, unless otherwise specified by the reference method. *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

10.10 LC/MS Systems

Calibration and tuning procedures are included in analytical SOPs written specifically for these tests. In general, multiple concentration levels for the analytes of interest are used to generate calibration curves. All reference materials used for this function are vendor-certified standards. Calibration and tuning verification is performed at SOP-defined intervals. Any other system performance checks are described in the applicable SOP. Calibration policies for organics chromatographic analyses are described in the *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

10.11 UV-Visible Spectrophotometer (manual colorimetric analyses)

Routine calibrations for colorimetric and turbidimetric analyses involve generating a 5-point calibration curve including a blank. Initial calibration points cannot be "dropped" from the resulting calibration curve. Correlation coefficients must meet method or SOP specifications before analysis can proceed. Independent calibration verification standards (ICVs) are analyzed with each batch of samples. Continuing calibration is verified at a minimum frequency of once every ten samples. Typical UV-Visible spectrophotometric methods at Columbia Analytical include total phenolics, phosphates, surfactants and tannin-lignin.

10.12 Flow Injection Analyzer (automated colorimetric analysis)

A minimum of six standards and a blank are used to calibrate the instrument for cyanide analysis. A blank and (minimum of) five standards are used to calibrate the instrument for all other automated chemistries. Initial calibration points cannot be "dropped" from the resulting calibration curve. Standard Columbia Analytical acceptance limits are used to evaluate the calibration curve prior to sample analysis.

10.13 Ion Chromatographs

Calibration of the ion chromatograph (IC) involves generating a calibration curve with the method-specified number of points (or more). Initial calibration points cannot be "dropped" from the resulting calibration curve. A correlation coefficient of $\geq$ 0.995 for the curve is required before analysis can proceed. Quality Control (QC) samples that are routinely analyzed include blanks and laboratory control samples. The target analytes typically determined by the IC include nitrate, nitrite, chloride, fluoride, sulfate and drinking water inorganic disinfection byproducts. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method.

10.14 Turbidimeter

Calibration of the turbidimeter requires analysis of three Nephelometric Turbidity Unit (NTU) formazin standards. Quality Control samples that are routinely analyzed include blanks, Analytical Products Group® QC samples (or equivalent) and duplicates.

10.15 Ion-selective electrode

The method-prescribed numbers of standards are used to calibrate the electrodes before analysis. The slope of the curve must be within acceptance limits before analysis can proceed. Quality Control samples that are routinely analyzed include blanks, LCSs and duplicates.

10.16 Pipets

The calibration of pipets and autopipettors used to make critical-volume measurements is verified following the *SOP Checking Volumetric Labware (ADM-VOLWARE)*. Both accuracy and precision verifications are performed, at intervals applicable to the pipet and use. The results of all calibration verifications are recorded in bound logbooks.

10.17 Other Instruments

Calibration for the total organic carbon (TOC), total organic halogen (TOX), and other instruments is performed following manufacturer's recommendations and applicable SOPs.

11.0 QUALITY CONTROL

A primary focus of Columbia Analytical's Quality Assurance (QA) Program is to ensure the accuracy, precision and comparability of all analytical results. Prior to using a procedure for the analysis on field samples, acceptable method performance is established by performing demonstration of capability analyses. Performance characteristics are established by performing method detection limit studies and assessing accuracy and precision according to the reference method. Columbia Analytical has established Quality Control (QC) objectives for precision and accuracy that are used to determine the acceptability of the data that is generated. These QC limits are either specified in the test methodology or are statistically derived based on the laboratory's historical data. Quality Control objectives are defined below.

11.1 Quality Control Objectives

11.1.2 Demonstration of Capability - A demonstration of capability (DOC) is made prior to using any new test method or when a technician is new to the method. This demonstration is made following regulatory, accreditation, or method specified procedures. In general, this demonstration does not test the performance of the method in real world samples, but in the applicable clean matrix free of target analytes and interferences.

A quality control sample material may be obtained from an outside source or may be prepared in the laboratory. The analyte(s) is (are) diluted in a volume of clean matrix (for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples). Where specified, the method-required concentration levels are used. Four aliquots are prepared and analyzed according to the test procedure. The mean recovery and standard deviations are calculated and compared to the corresponding acceptance criteria for precision and accuracy in the test method or laboratory-generated acceptance criteria (if there are not established mandatory criteria). All parameters must meet the acceptance criteria. Where spike levels are not specified, actual Laboratory Control Sample results may be used to meet this requirement, provided acceptance criteria is met.

11.1.3 Accuracy - Accuracy is a measure of the closeness of an individual measurement (or an average of multiple measurements) to the true or expected value. Accuracy is determined by calculating the mean value of results from ongoing analyses of laboratory-fortified blanks, standard reference materials, and standard solutions. In addition, laboratory-fortified (i.e. matrix-spiked) samples are also measured; this indicates the accuracy or bias in the actual sample matrix. Accuracy is expressed as percent recovery (% REC.) of the measured value, relative to the true or expected value. If a measurement process produces results whose mean is not the true or expected value, the process is said to be biased. Bias is the systematic error either inherent in a method of analysis (e.g., extraction efficiencies) or caused by an artifact of the measurement system (e.g., contamination). Columbia Analytical utilizes several quality control measures to eliminate analytical bias, including systematic analysis of method blanks, laboratory control samples and independent calibration verification standards. Because bias can be positive or negative, and because several types of bias can occur simultaneously, only the net, or total, bias can be evaluated in a measurement

11.1.4 Precision - Precision is the ability of an analytical method or instrument to reproduce its own measurement. It is a measure of the variability, or random error, in sampling, sample handling and in laboratory analysis. The American Society of Testing and Materials (ASTM) recognizes two levels of precision: repeatability - the random error associated with measurements made by a single test operator on identical aliquots of test material in a given laboratory, with the same apparatus, under constant operating conditions, and reproducibility - the random error associated with measurements made by different test operators, in different laboratories, using the same method but different equipment to analyze identical samples of test material.

"Within-batch" precision is measured using replicate sample or QC analyses and is expressed as the relative percent difference (RPD) between the measurements. The "batch-to-batch" precision is determined from the variance observed in the analysis of standard solutions or laboratory control samples from multiple analytical batches.

11.1.5 Control Limits - The control limits for accuracy and precision originate from two different sources. For analyses having enough QC data, control limits are calculated at the 99% confidence limits. For analyses not having enough QC data, or where the method is prescriptive, control limits are taken from the method on which the procedure is based. If the method does not have stated control limits, then control limits are assigned method-default or reasonable values. Control limits are updated periodically when new statistical limits are generated for the appropriate surrogate, laboratory control sample, and matrix spike compounds (typically once a year) or when method prescribed limits change. The updated limits are reviewed by the Quality Assurance Manager. The new control limits replace the previous limits and data is assessed using the new values. Current acceptance limits for accuracy and precision are available from the laboratory. For inorganics, the precision limit values listed are for laboratory duplicates. For organics, the precision limit values listed are for duplicate laboratory control samples or duplicate matrix spike analyses.

11.1.6 Representativeness - Representativeness is the degree to which the field sample, being properly preserved, free of contamination, and analyzed within holding time, represents the overall sample site or material. This can be extended to the sample itself, in that representativeness is the degree to which the subsample that is analyzed represents the entire field sample submitted for analysis. Columbia Analytical has sample handling procedures to ensure that the sample used for analysis is representative of the entire sample.

These include the *SOP for Subsampling and Compositing of Samples* and the *SOP for Tissue Sample Preparation*. Further, analytical SOPs specify appropriate sample handling and sample sizes to further ensure the sample aliquot that is analyzed is representative in entire sample.

11.1.7 Comparability – Comparability expresses the confidence with which one data set can be compared to another and is directly affected by data quality (accuracy and precision) and sample handling (sampling, preservation, etc). Only data of known quality can be compared. The objective is to generate data of known quality with the highest level of comparability, completeness, and usability. This is achieved by employing the quality controls listed below and standard operating procedures for the handling and analysis of all samples. Data is reported in units specified by the client and using Columbia Analytical or project-specified data qualifiers.

11.2 Method Detection Limits and Method Reporting Limits

Method Detection Limits (MDL) for methods performed at Columbia Analytical/Kelso is determined during initial method set up and if any significant changes are made. If an MDL study is not performed annually, the established MDL is verified by performing a limit of detection (LOD) verification on every instrument used in the analysis. The MDLs are determined by following the *SOP for Performing Method Detection Limits Studies and Establishing Limits of Detection and Quantitation (ADM-MDL),* which is based on the procedure in 40 CFR Part 136, Appendix B. As required by NELAP and DoD protocols, the validity of MDLs is verified using LOD verification samples.

The Method Reporting Limit (MRL) is the lowest amount of an analyte in a sample that can be quantitatively determined with stated, acceptable precision and accuracy under stated analytical conditions (i.e. limit of quantitation- LOQ). LOQ are analyzed on an annual basis and cannot be lower than the lowest calibration standard. Current MDLs and MRLs are available from the laboratory.

11.3 Quality Control Procedures

The specific types, frequencies, and processes for quality control sample analysis are described in detail in method-specific standard operating procedures and listed below. These sample types and frequencies have been adopted for each method and a definition of each type of QC sample is provided below.

11.3.1 Method Blank (a.k.a. Laboratory Reagent Blank)

The method blank is an analyte-free matrix (water, soil, etc.) subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand, or an acceptable substitute is used. The method blank is analyzed to demonstrate that the analytical system itself does not introduce contamination. The method blank results should be below the Method Reporting Limit (MRL) or, if required for DoD projects, < ½ MRL for the analyte(s) being tested. Otherwise, corrective action must be taken. A method blank is included with the analysis of every sample preparation batch, every 20 samples, or as stated in the method, whichever is more frequent.

11.3.2 Calibration Blanks

For some methods, calibration blanks are prepared along with calibration standards in order to create a calibration curve. Calibration blanks are free of the analyte of interest and, where applicable, provide the zero point of the calibration curve. Additional project-specific requirements may also apply to calibration blanks.

11.3.3 Continuing Calibration Blanks

Continuing calibration blanks (CCBs) are solutions of either analyte-free water, reagent, or solvent that are analyzed in order to verify the system is contamination-free when CCV standards are analyzed. The frequency of CCB analysis is either once every ten samples or as indicated in the method, whichever is greater. Additional project-specific requirements may also apply to continuing calibration blanks.

11.3.4 Calibration Standards

Calibration standards are solutions of known concentration prepared from primary standard or stock standard materials. Calibration standards are used to calibrate the instrument response with respect to analyte concentration. Standards are analyzed in accordance with the requirements stated in the particular method being used.

11.3.5 Initial (or Independent) Calibration Verification Standards

Initial (or independent) calibration verification standards (ICVs) are standards that are analyzed *after* calibration with newly prepared standard(s) but *prior to* sample analysis, in order to verify the validity and accuracy of the standards used in the calibration. Once it is determined that there is no reference material defect or systematic error in preparation of the calibration standard(s), standards are considered valid and may be used for subsequent calibrations and quantitative determinations (as expiration dates and methods allow). The ICV standards are prepared from materials obtained from a source independent of that used for preparing the calibration standards ("second-source"). ICVs are also analyzed in accordance with method-specific requirements.

11.3.6 Continuing Calibration Verification Standards

Continuing calibration verification standards (CCVs) are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. The frequency of CCV analysis is either once every ten samples, or as indicated in the method.

11.3.7 Internal Standards

Internal standards are known amounts of specific compounds that are added to each sample prior to instrument analysis. Internal standards are generally used for GC/MS and ICP-MS procedures to correct sample results that have been affected by changes in instrument conditions or changes caused by matrix effects. The requirements for evaluation of internal standards are specified in each method and SOP.

11.3.8 Surrogates

Surrogates are organic compounds which are similar in chemical composition and chromatographic behavior to the analytes of interest, but which are not normally found in environmental samples. Depending on the analytical method, one or more of these compounds is added to method blanks, calibration and check standards, and samples (including duplicates, matrix spike samples, duplicate matrix spike samples and laboratory control samples) prior to extraction and analysis in order to monitor the method performance on each sample. The percent recovery is calculated for each surrogate, and the recovery is a measurement of the overall method performance.

Recovery (%) = (M/T) x 100

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

11.3.9 Laboratory Control Samples

The laboratory control sample (LCS) is an aliquot of analyte-free water or analyte-free solid (or anhydrous sodium sulfate or equivalent) to which known amounts of the method analyte(s) is (are) added. A reference material of known matrix type, containing certified amounts of target analytes, may also be used as an LCS. An LCS is prepared and analyzed at a minimum frequency of one LCS per 20 samples, with every analytical batch or as stated in the method, whichever is more frequent. The LCS sample is prepared and analyzed in exactly the same manner as the field samples.

The percent recovery of the target analytes in the LCS is compared to established control limits and assists in determining whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required reporting limit. Comparison of batch-to-batch LCS analyses enables the laboratory to evaluate batch-to-batch precision and accuracy.

Recovery (%) = (M/T) x 100

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

11.3.10 Laboratory Fortified Blanks - LFB

A laboratory blank fortified at the MRL used to verify the minimum reporting limit. The LFB is carried through the entire extraction and analytical procedure. A LFB is required with every batch of drinking water samples.

11.3.11 Matrix Spikes (a.k.a. Laboratory Fortified Sample Matrix)

Matrix spiked samples are aliquots of samples to which a known amount of the target analyte (or analytes) is (are) added. The samples are then prepared and analyzed in

the same analytical batch, and in exactly the same manner as are routine samples. For the appropriate methods, matrix spiked samples are prepared and analyzed and at a minimum frequency of one spiked sample (and one duplicate spiked sample, if appropriate) per twenty samples. The spike recovery measures the effects of interferences caused by the sample matrix and reflects the accuracy of the method for the particular matrix in question. Spike recoveries are calculated as follows:

$$Recovery\ (\%) = (S - A) \times 100 \div T$$

Where: S = The observed concentration of analyte in the spiked sample,
A = The analyte concentration in the original sample, and
T = The theoretical concentration of analyte added to the spiked sample.

11.3.12 Laboratory Duplicates and Duplicate Matrix Spikes

Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed. The relative percent difference between duplicate analyses or between an MS and DMS is a measure of the precision for a given method and analytical batch. The relative percent difference (RPD) for these analyses is calculated as follows:

$$Relative\ Percent\ Difference\ (RPD) = (S1 - S2) \times 100 \div S_{ave}$$

Where S1 and S2 = The observed concentrations of analyte in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike, and

S_{ave} = The average of observed analyte concentrations in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike.

Depending on the method of analysis, either duplicates (and/or matrix spikes) or MS/DMS analyses are performed at a minimum frequency of one set per 20 samples. If an insufficient quantity of sample is available to perform a laboratory duplicate or duplicate matrix spikes, duplicate LCSs will be prepared and analyzed.

11.3.13 Interference Check Samples

An interference check sample (ICS) is a solution containing both interfering and analyte elements of known concentration that can be analyzed to verify background and interelement correction factors in metals analyses. The ICS is prepared to contain known concentrations (method or program specific) of elements that will provide an adequate test of the correction factors. The ICS is analyzed at the beginning and end of an analytical run or at a method-specified frequency. Results must meet method criteria and any project-specific criteria.

11.3.14 Post Digestion Spikes

Post digestion spikes are samples prepared for metals analyses that have an analyte spike added to determine if matrix effects may be a factor in the results. The spike addition should produce a method-specified minimum concentration above the method reporting limit. A post digestion spike is analyzed with each batch of samples and recovery criteria are specified for each method.

11.3.15 Control Charting

The generation of control charts is routinely performed at Columbia Analytical. Surrogate, Matrix Spike and LCS recoveries are all monitored and charted. In addition, the laboratory also monitors the Relative Percent Difference (RPD) measurement of precision. Control charts are available to each individual laboratory unit to monitor the data generated in its facility using control charts that have been programmed to identify various trends in the analytical results. If trends in the data are perceived, various means of corrective action may then be employed in order to prevent future problems with the analytical system(s). Finally, data quality reports using control charts are generated for specific clients and projects pursuant to contract requirements. The control charting procedure is described in the SOP for *Control Charting Quality Control Data* (ADM-CHRT).

11.3.16 Glassware Washing

Glassware washing and maintenance play a crucial role in the daily operation of a laboratory. The glassware used at Columbia Analytical undergoes a rigorous cleansing procedure prior to every usage. A number of SOPs have been generated that outline the various procedures used at Columbia Analytical; each is specific to the end-use of the equipment as well as to the overall analytical requirements of the project. In addition, other equipment that may be routinely used at the laboratory is also cleaned following instructions in the appropriate SOP.

12.0 DATA REDUCTION, VALIDATION, AND REPORTING

Columbia Analytical reports the analytical data produced in its laboratories to the client via the certified analytical report (CAR). This report includes a transmittal letter, a case narrative, client project information, specific test results, quality control data, chain of custody information, and any other project-specific support documentation. The following procedures describe our data reduction, validation and reporting procedures.

12.1 Data Reduction and Review

Results are generated by the analyst who performs the analysis and works up the data. All data is initially reviewed and processed by analysts using appropriate methods (e.g., chromatographic software, instrument printouts, hand calculation, etc.). Equations used for calculation of results are found in the applicable analytical SOPs. The resulting data set is either manually entered (e.g., titrimetric or microbiological data) into an electronic report form or is electronically transferred into the report from the software used to process the original data set (e.g., chromatographic software). Once the complete data set has been transferred into the proper electronic report form(s), it is then printed. The resulting hardcopy version of the electronic report is then reviewed by the analyst for accuracy. Once the primary analyst has checked the data for accuracy and acceptability, the hardcopy is forwarded to the supervisor or second qualified analyst, who reviews the data for errors. Where calculations are not performed using a validated software system, the reviewer rechecks a minimum of 10% of the calculations. When the entire data set has been found to be acceptable, a final copy of the report is printed and signed by the laboratory supervisor, departmental manager or designated laboratory staff. The entire data package is then placed into the appropriate service request file, and an electronic copy of the final data package is forwarded to the appropriate personnel for archival. Data review procedures are described in the *SOP for Laboratory Data Review Process.*

Policies and procedures for manual editing of data are established. The analyst making the change must initial and date the edited data entry, without obliteration of the original entry. The policies and procedures are described in the *SOP for Making Entries into Logbooks and onto Benchsheets* (SOP ADM-DATANTRY).

Policies and procedures for electronic manual integration of chromatographic data are established. The analyst performing the integration must document the integration change by printing both the “before” and “after” integrations and including them in the raw data records. The policies and procedures are described in the *SOP for Manual Integration of Chromatographic Peaks* (SOP ADM-INT).

12.2 Confirmation Analysis

12.2.1 Gas Chromatographic and Liquid Chromatographic Analyses

For gas chromatographic (GC) and liquid chromatographic (LC) analyses, all positive results are confirmed by a second column, a second detector, a second wavelength (HPLC/UV), or by GC/MS analysis, unless exempted by one of the following situations:

- The analyte of interest produces a chromatogram containing multiple peaks exhibiting a characteristic pattern, which matches appropriate standards. This is limited to petroleum hydrocarbon analyses (e.g., gasoline and diesel) and does not include polychlorinated biphenyls.
- The sample meets all of the following requirements:
    1. All samples (liquid or solid) come from the same source (e.g., groundwater samples from the same well) for continuous monitoring. Samples of the same matrix from the same site, but from different sources (e.g., different sampling locations) are not exempt.
    2. All analytes have been previously analyzed in sample(s) from the same source (within the last year), identified and confirmed by a second column or by GC/MS. The chromatogram is largely unchanged from the one for which confirmation was carried out. The documents indicating previous confirmation must be available for review.

12.2.2 Confirmation Data

Confirmation data will be provided as specified in the method. Identification criteria for GC, LC or GC/MS methods are summarized below:

- GC and LC Methods
    1. The analyte must fall within plus or minus three times the standard deviation (established for the analyte/column) of the retention time of the daily midpoint standard in order to be qualitatively identified. The retention-time windows will be established and documented, as specified in the appropriate Standard Operating Procedure (SOP).
    2. When sample results are confirmed by two dissimilar columns or detectors, the agreement between quantitative results must be evaluated. The relative percent difference between the two results is calculated and evaluated against SOP and/or method criteria.
- GC/MS Methods - Two criteria are used to verify identification:
    1. Elution of the analyte in the sample will occur at the same relative retention time (RRT) as that of the analyte in the standard.
    2. The mass spectrum of the analyte in the sample must, in the opinion of a qualified analyst or the department manager, correspond to the spectrum of the analyte in the standard or the current GC/MS reference library.

12.3 Data Review and Validation of Results

The integrity of the data generated is assessed through the evaluation of the sample results, calibrations, and QC samples (method blanks, laboratory control samples, sample duplicates, matrix spikes, trip blanks, etc.). A brief description of the evaluation of these analyses is described below, with de tails listed in applicable SOPs. The criteria for evaluation of QC samples are listed within each method-specific SOP. Other data evaluation measures may include (as necessary) a check of the accuracy check of the QC standards and a check of the system sensitivity. Data transcriptions and calculations are also reviewed.

Note: Within the scope of this document, all possible data assessment requirements for various project protocols cannot be included in the listing below. This listing gives a general description of data evaluation practices used in the laboratory in compliance with NELAP Quality Systems requirements. Additional requirements exist for certain programs, such as projects under the DoD QSM protocols, and project-specific QAPPs.

- Method Calibration – Following the analysis of calibration blanks and standards according to the applicable SOP the calibration correlation coefficient, average response factor, etc. is calculated and compared to specified criteria. If the calibration meets criteria analysis may continue. If the calibration fails, any problems are isolated and corrected and the calibration standards reanalyzed. Following calibration and analysis of the independent calibration verification standard(s) the percent difference for the ICV is calculated. If the percent difference is within the specified limits the calibration is complete. If not, the problem associated with the calibration and/or ICV are isolated and corrected and verification and/or calibration is repeated.
- Continuing Calibration Verification (CCV) – Following the analysis of the CCV standard the percent difference is calculated and compared to specified criteria. If the CCV meets the criteria analysis may continue. If the CCV fails, routine corrective action is performed and documented and a 2nd CCV is analyzed. If this CCV meets criteria, analysis may continue, including any reanalysis of samples that were associated with a failing CCV. If the routine corrective action failed to produce an immediate CCV within criteria, then either acceptable performance is demonstrated (after additional corrective action) with two consecutive calibration verifications or a new initial calibration is performed.
- Method Blank – Results for the method blank are calculated as performed for samples. If results are less than the MRL (<½ MRL for DoD projects), the blank may be reported. If not, associated sample results are evaluated to determine the impact of the blank result. If possible, the source of the contamination is determined. If the contamination has affected sample results the blank and samples are reanalyzed. If positive blank results are reported, the blank (and sample) results are flagged with an appropriate flag, qualifier, or footnote.
- Sample Results (Inorganic) – Following sample analysis and calculations (including any dilutions made due to the sample matrix) the result is verified to fall within the calibration range. If not, the sample is diluted and analyzed to bring the result into calibration range. When sample and sample duplicates are analyzed for precision, the calculated RPD is compared to the specified limits. The sample and duplicate are reanalyzed if the criteria are exceeded. The samples may require re-preparation and reanalysis. For metals, additional measures as described in the applicable SOP may be taken to further evaluate results (dilution tests and/or post-digestion spikes). Results are reported when within the calibration range, or as estimates when outside the calibration range. When dilutions are

performed the MRL is elevated accordingly and qualified. Efforts are made to meet the project MRL's including alternative analysis.

- Sample Results (Organic) – For GC/MS analyses, it is verified that the analysis was within the prescribed tune window. If not, the sample is reanalyzed. Following sample analysis and calculations (including any dilutions made due to the sample matrix) peak integrations, retention times, and spectra are evaluated to confirm qualitative identification. Internal standard responses and surrogate recoveries are evaluated against specified criteria. If internal standard response does not meet criteria, the sample is diluted and reanalyzed. Results outside of the calibration range are diluted to within the calibration range. For GC and HPLC tests, results from confirmation analysis are evaluated to confirm positive results and to determine the reported value. The procedure to determine which result to report is described in the SOP *Confirmation Procedure for GC and HPLC Analysis (SOC-CONF).* If obvious matrix interferences are present, additional cleanup of the sample using appropriate procedures may be necessary and the sample is reanalyzed. When dilutions are performed the MRL is elevated accordingly and qualified. Efforts are made to meet the project MRL's including additional cleanup.
- Surrogate Results (Organic) – Following sample analysis and data reduction, the percent recovery of each surrogate is compared to specified control limits. If recoveries are acceptable, the results are reported. If recoveries do not fall within control limits, the sample matrix is evaluated. When matrix interferences are present or documented, the results are reported with a qualifier that matrix interferences are present. If no matrix interferences are present and there is no cause for the outlier, the sample is reprepared and reanalyzed. However, if the recovery is above the upper control limit with non-detected target analytes, the sample may be reported. All surrogate recovery outliers are appropriately qualified on the report.
- Duplicate Sample and/or Duplicate Matrix Spike Results – The RPD is calculated and compared to the specified control limits. If the RPD is within the control limits the result is reported. If not, an evaluation of the sample is made to verify that a homogenous sample was used. Despite the use of homogenizing procedures prior to sample preparation or analysis, the sample may not be homogenous or duplicate sample containers may not have been sample consistently. If non-homogenous, the result is reported with a qualifier about the homogeneity of the sample. Also, the results are compared to the MRL. If the results are less than five times the MRL, the results are reported with a qualifier that the high RPD is due to the results being near the MRL. If the sample is homogenous and results above five times the MRL, the samples and duplicates are reanalyzed. If re-analysis also produces out-of-control results, the results are reported with an appropriate qualifier.
- Laboratory Control Sample Results – Following analysis of the LCS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits, the analysis is in control and results may be reported. If not, this indicates that the analysis is not in control. Samples associated with the 'out of control' LCS, shall be considered suspect and the samples re-extracted or re-analyzed or the data reported with the appropriate qualifiers. For analysis where a large number of analytes are in the LCS, it becomes more likely that some analytes (marginal exceedences) will be outside the control limits. The procedure described in the 2003 NELAC standards, Appendix D.1.1.2.1 are used to determine if the LCS is effective in validating the analytical system and the associated samples.

- Matrix Spike Results – Following analysis of the MS the percent recovery is calculated and compared to specified control limits. If the recovery is within control limits the results may be reported. If not, and the LCS is within control limits, this indicates that the matrix potentially biases analyte recovery. It is verified that the spike level is at least five times the background level. If not, the results are reported with a qualifier that the background level is too high for accurate recovery determination. If matrix interferences are present or results indicate a potential problem with sample preparation, steps may be taken to improve results; such as performing any additional cleanups, dilution and reanalysis, or re-preparation and reanalysis. Results that do not meet acceptance limits are reported with an appropriate qualifier.

12.4 Data Reporting

When an analyst determines that a data package has met the data quality objectives (and/or any client-specific data quality objectives) of the method and has qualified any anomalies in a clear, acceptable fashion, the data package is reviewed by a trained chemist. Prior to release of the report to the client, the project chemist reviews and approves the entire report for completeness and to ensure that any and all client-specified objectives were successfully achieved. The original raw data, along with a copy of the final report, is filed in project files by service request number for archiving. Columbia Analytical maintains control of analytical results by adhering to standard operating procedures and by observing sample custody requirements. All data are calculated and reported in units consistent with project specifications, to enable easy comparison of data from report to report.

To the extent possible, samples shall be reported only if all QC measures are acceptable. If a QC measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). The *SOP for Data Reporting and Report Generation* addresses the flagging and qualification of data. The Columbia Analytical-defined data qualifiers, state-specific data qualifiers, or project-defined data qualifiers are used depending on project requirements. A case narrative may be written by the project chemist to explain problems with a specific analysis or sample, etc.

For subcontracted analyses, the Project Chemist verifies that the report received from the subcontractor is complete. This includes checking that the correct analyses were performed, the analyses were performed for each sample as requested, a report is provided for each analysis, and the report is signed. The Project Chemist accepts the report if all verification items are complete. Acceptance is demonstrated by forwarding the report to the Columbia Analytical client.

12.5 Documentation

Columbia Analytical maintains a records system which ensures that all laboratory records of analysis data retained and available. Analysis data is retained for 5 years from the report date unless contractual terms or regulations specify a longer retention time. The archiving system is described in the *SOP for Data Archiving*.

12.5.1 Documentation and Archiving of Sample Analysis Data

The archiving system includes the following items for each set of analyses performed:

- Benchsheets describing sample preparation (if appropriate) and analysis;
- Instrument parameters (or reference to the data acquisition method);
- Sample analysis sequence;
- Instrument printouts, including chromatograms and peak integration reports for all samples, standards, blanks, spikes and reruns;
- Logbook ID number for the appropriate standards;
- Copies of report sheets submitted to the work request file; and
- Copies of Nonconformity and Corrective Action Reports, if necessary.

Individual sets of analyses are identified by analysis date and service request number. Since many analyses are performed with com puter-based data systems, the final sample concentrations can be automatically calculated. If additional calculations are needed, they are written on the integration report or securely stapled to the chromatogram, if done on a separate sheet.

For organics analysis, data applicable to all analyses within the batch, such as GCMS tunes, CCVs, batch QC, and analysis sequences; are kept using a separate documentation system. This system is used to archive data on a batch-specific basis and is segregated according to the date of analysis. This system also includes results for the most recent calibration curves, as well as method validation results.

12.6 Deliverables

In order to meet individual project needs, Columbia Analytical provides several levels of analytical reports. Standard specifications for each level of deliverable are described in Table 12-1. Variations may be provided based on client or project specifications. This includes (but is not limited to) to following specialized deliverables:

- ADEC – Alaska Department of Conservation specified data package
- ACOE/HTRW – Army Corps of Engineers specified data package and reporting requirements (HTRW, CERP, FUDS, etc.)
- AFCEE – Air Force Center for Environmental Excellence project-specific reporting

When requested, Columbia Analytical provides Electronic Data Deliverables (EDDs) in the format specified by client need or project specification. Columbia Analytical is capable of generating EDDs with many different formats and specifications. The EDD is prepared by report production staff using the electronic version of the laboratory report to minimize transcription errors. User guides and EDD specification outlines are used in preparing the EDD. The EDD is reviewed and compared to the hard-copy report for accuracy.

Table 12-1
Descriptions of Columbia Analytical Standard Data Deliverables

Tier I. Routine Certified Analytical Report (CAR) includes the following:

1. Transmittal letter
2. Sample analytical results
3. Method blank results
4. Surrogate recovery results and acceptance criteria for applicable organic methods
5. Chain of custody documents
6. Dates of sample preparation and analysis for all tests

Tier II and IIA. In addition to the Tier I Deliverables, this CAR includes the following:

1. Matrix spike result(s) with calculated recovery and including associated acceptance criteria
2. Duplicate or duplicate matrix spike result(s) (as appropriate to method), with calculated relative percent difference
3. Tier IIA also includes Laboratory Control Sample (LCS) result(s) with calculated recovery and including associated acceptance criteria

Tier III. Data Validation Package. In addition to the Tier II Deliverables, this CAR includes the following:

1. Case narrative
2. Calibration records and results of initial and continuing calibration verification standards, with calculated recoveries
3. Results of laboratory control sample (LCS) or Quality Control check sample, with calculated recovery and/or associated acceptance limit criteria
4. Results of calibration blanks or solvent blanks (as appropriate to method)
5. Summary forms for associated QC and calibration parameters
6. Copies of all raw data, including extraction/preparation bench sheets, chromatograms, and instrument printouts. For GC/MS, this includes tuning criteria and mass spectra of all positive hits. Results and spectra of TIC compounds will be included upon request.

Tier IV. CLP-Level Data Validation Package.

A complete Data Validation Package containing all sample results, quality control and calibration results, and raw data necessary to fulfill all deliverable requirements of an EPA Contract Laboratory Program (CLP) data package.

13.0 PERFORMANCE AND SYSTEM AUDITS

Quality audits are an essential part of C olumbia Analytical/Kelso's quality assurance program. There are two types of audits used at the facility: System Audits are conducted to qualitatively evaluate the operational details of the QA program, while Performance Audits are conducted by analyzing proficiency testing samples in order to quantitatively evaluate the outputs of the various measurement systems.

13.1 System Audits

The system audit examines the presence and appropriateness of laboratory systems. External system audits of C olumbia Analytical/Kelso are conducted regularly by various regulatory agencies and clients. Table 13-1 summarizes some of the major programs in which C olumbia Analytical/Kelso participates. Programs and certifications are added as required. Additionally, internal system audits of Columbia Analytical/Kelso are conducted regularly under the direction of the Quality Assurance Manager. The internal audit procedures are described in the *SOP for Internal Audits*. The internal audits are performed as follows:

- Comprehensive lab-wide system audit – performed annually. This audit is conducted such that systems, technical operations, hardcopy data, and electronic data are assessed.
- Hardcopy report audits – minimum of 3 per quarter.
- Electronic audit trail reviews – each applicable instrument per quarter.

All audit findings, and corrective actions are documented. The results of each audit are reported to the Laboratory Director and Department Managers for review. Any deficiencies identified are summarized in the audit report. Managers must respond with corrective actions correcting the deficiency within a defined timeframe. Should problems impacting data quality be found during an internal audit, any client whose data is adversely impacted will be given written notification within the corrective action period (if not already provided).

Electronic data audits may be performed in conjunction with hardcopy data audits. The electronic audits focus on organic chromatographic data and include an examination of audit trails, peak integrations, calibration practices, GCMS tuning data, peak response data, use of appropriate files, and other components of the analysis. The audit also verifies that the electronic data supports the hardcopy reported data.

Additional internal audits or data evaluations may be performed as needed to address any potential data integrity issues that may arise.

13.2 Performance Audits

Columbia Analytical/Kelso also participates in the analysis of interlaboratory proficiency testing (PT) samples. Participation in PT studies is performed on a regular basis and is designed to evaluate all analytical areas of the laboratory. Columbia Analytical routinely participates in the following studies:

- Water Pollution (WP) and additional water parameters, 2 per year.
- Water Supply (WS) PT studies, 2 per year.
- Hazardous Waste/Soil PT studies, 2 per year.
- Underground Storage Tank PT studies, 2 per year.
- Microbiology (WS and WP) PT studies, 2 per year.
- Other studies as required for specific certifications, accreditations, or validations.

PT samples are processed by entering them into the LIMS system as samples (assigned Service Request, due date, testing requirements, etc.) and are processed the same as field samples. The laboratory sections handle samples the same as field samples, performing the analyses following method requirements and performing data review. The laboratory sections submit results to the QA Manager for subsequent reporting to the appropriate agencies or study provider. Results of the performance evaluation samples and audits are reviewed by the Quality Assurance Manager, Laboratory Director, the laboratory staff, and the Columbia Analytical Quality Assurance Director. For any results outside acceptance criteria, the analysis data is reviewed to identify a root cause for the deficiency, and corrective action is taken and documented through nonconformity (NCAR) procedures.

Table 13-1
Current Columbia Analytical Performance and System Audit Programs

Federal and National Programs

- The TNI (The NELAC Institute) National Environmental Laboratory Accreditation Program (NELAP) Accredited Drinking Water, Non-Potable Water, Solid & Hazardous Waste, and Biological Tissue Laboratory
- ANSI-ASQ National Accreditation Board/ACLASS ISO 17025:2005
- DoD- ELAP Environmental Laboratory Accreditation Program
- Naval Facilities Engineering Service Center
  Validated Laboratory for NFESC Parameters
- U.S. Army Corps of Engineers
  Approved Laboratory for USACE Projects
- U.S. EPA Region 8
  Approved Drinking Water Laboratory

State and Local Programs

- State of Alaska, Department of Environmental Conservation
  UST Laboratory, Lab I.D. UST040
- State of Arizona, Department of Health Services
  License No. AZ0339
- State of Arkansas, Department of Environmental Quality
  Certified Environmental Laboratory, Lab I.D. 88-0637
- State of California, Department of Health Services, Environmental Laboratory Accreditation Program
  Certification No. 2286
- State of Colorado, Department of Public Health and Environment
  Certified Drinking Water Laboratory
- State of Florida, Department of Health
  Primary NELAP Accreditation No. E87412
- State of Georgia, Department of Natural Resources
  Certified Drinking Water Laboratory
- State of Hawaii, Department of Health
  Certified Drinking Water Laboratory
- State of Idaho, Department of Health and Welfare
  Certified Drinking Water Laboratory
- State of Indiana, Department of Health
  Certified Drinking Water Laboratory, Lab I.D. C-WA-01
- State of Louisiana, Department of Environmental Quality
  Accredited Environmental Laboratory, Lab I.D. 3016
- State of Louisiana, Department of Health and Hospitals
  Accredited Drinking Water Laboratory, Lab I.D. LA080001
- State of Maine, Department of Human Services
  Certified Environmental Laboratory, Lab I.D. WA0035
- State of Michigan, Department of Environmental Quality
  Certified Drinking Water Laboratory, Lab I.D. 9949

Table 13-1 (continued)

State and Local Programs (continued)

- State of Minnesota, Department of Health
  Certified Environmental Laboratory, Lab I.D. 053-999-368
- State of Montana, Department of Health and Environmental Sciences
  Certified Drinking Water Laboratory, Lab I.D. 0047
- State of Nevada, Division of Environmental Protection
  Certified Drinking Water Laboratory, Lab I.D. WA35
- State of New Jersey, Department of Environmental Protection
  Accredited Environmental Laboratory, Lab I.D. WA005
- State of New Mexico, Environment Department
  Certified Drinking Water Laboratory
- State of North Carolina, Department of Environment and Natural Resources
  Certified Environmental Laboratory, Lab I.D. 605
- State of Oklahoma, Department of Environmental Quality
  General Water Quality/Sludge Testing, Lab I.D. 9801
- State of Oregon, ORELAP Laboratory Accreditation Program
  Accredited Environmental Laboratory, Lab I.D. WA200001
- State of South Carolina, Department of Health and Environmental Control
  Certified Environmental Laboratory, Lab I.D. 61002
- State of Utah, Department of Health, Division of Laboratory Services
  Accredited Environmental Laboratory
- State of Washington, Department of Ecology, Environmental Laboratory Accreditation Program
  Accreditation No. C1203
- State of Wisconsin, Department of Natural Resources
  Accredited Environmental Laboratory, Lab I.D. 998386840

14.0 PREVENTIVE MAINTENANCE

Preventive maintenance is a crucial element of the Quality Assurance program. Instruments at Columbia Analytical (e.g., ICP/MS and ICP systems, GC/MS systems, atomic absorption spectrometers, analytical balances, gas and liquid chromatographs, etc.) are maintained under commercial service contracts or by qualified, in-house personnel. All instruments are operated and maintained according to the instrument operating manuals. All routine and special maintenance activities pertaining to the instruments are recorded in instrument maintenance logbooks. The maintenance logbooks used at Columbia Analytical contain extensive information about the instruments used at the laboratory.

An initial demonstration of analytical control is required on every instrument used at Columbia Analytical before it maybe used for sample analysis. If an instrument is modified or repaired, a return to analytical control is required before subsequent sample analyses can occur. When an instrument is acquired at the laboratory, the following information is noted in a bound maintenance notebook specifically associated with the new equipment:

- The equipment's serial number;
- Date the equipment was received;
- Date the equipment was placed into service;
- Condition of equipment when received (new, used, reconditioned, etc.); and
- Prior history of damage, malfunction, modification or repair (if known).

Preventive maintenance procedures, frequencies, etc. are available for each instrument used at Columbia Analytical. They may be found in the various SOPs for routine methods performed on an instrument and may also be found in the operating or maintenance manuals provided with the equipment at the time of purchase.

Responsibility for ensuring that routine maintenance is performed lies with the section supervisor. The supervisor may perform the maintenance or assign the maintenance task to a qualified bench level analyst who routinely operates the equipment. In the case of non-routine repair of capital equipment, the section supervisor is responsible for providing the repair, either by performing the repair themselves with manufacturer guidance or by acquiring on-site manufacturer repair. Each laboratory section maintains a critical parts inventory. The parts inventories include the items needed to perform the preventive maintenance procedures listed in Appendix D.

This inventory or "parts list" also includes the items needed to perform any other routine maintenance and certain in-house non-routine repairs such as gas chromatography/mass spectrometry jet separators and electron multipliers and ICP/MS nebulizer. When performing maintenance on an instrument (whether preventive or corrective), additional information about the problem, attempted repairs, etc. is also recorded in the notebook. Typical logbook entries include the following information:

- Details and symptoms of the problem;
- Repairs and/or maintenance performed;
- Description and/or part number of replaced parts;
- Source(s) of the replaced parts;
- Analyst's signature and date; and
- Demonstration of return to analytical control.

See the table in Appendix D for a list of preventive maintenance activities and frequency for each instrument.

15.0 CORRECTIVE ACTION

Nonconforming events such as errors, deficiencies, deviations from SOP, proficiency (PT) failure or results that fall outside of established QC limits are documented using a *Nonconformity and Corrective Action Report* form. The laboratorys procedure and responsibilities for addressing nonconforming work is defined in the SOP ADM-CA *Corrective Action.*

The laboratory takes all appropriate steps necessary to ensure all sample results are reported with acceptable quality control results. When sample results do not conform to established quality control procedures, responsible management will evaluate the significance of the nonconforming work and take corrective action to address the nonconformance.

If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). Failure to meet established analytical controls, such as the quality control objectives outlined in Section 11, prompts corrective action. In general, corrective action may take several forms and may involve a review of the calculations, a check of the instrument maintenance and operation, a review of analytical technique and methodology, and reanalysis of quality control and field samples. If a potential problem develops that cannot be solved directly by the responsible analyst, the supervisor, team leader, the department manager, and/or the Quality Assurance Manager may examine and pursue alternative solutions. In addition, the appropriate project chemist is notified in order to ascertain if the client needs to be notified.

In the event that analyses produce nonconformances with data or results, the problem and the corresponding corrective actions taken are documented on a *Nonconformity and Corrective Action Report* (See Figure 15-1) following the requirements in the *SOP for Corrective Action* (SOP No. ADM-CA). This form is utilized to determine the root cause of the nonconformity and to document corrective actions in response to out-of-control situations. The Quality Assurance Manager reviews each problem, ensuring that appropriate corrective action has been taken by the appropriate personnel. The Nonconformity and Corrective Action Report (NCAR) is filed in the associated service request file and a copy is kept by the Quality Assurance Manager. The Quality Assurance Manager periodically reviews all NCARs looking for chronic, systematic problems that need more in-depth investigation and alternative corrective action consideration. In addition, the appropriate project chemist is promptly notified of any problems in order to inform the client and proceed with any action the client may want to initiate.

In addition to internal communication of data issues, the laboratory also maintains a system for dealing with customer complaints. The person who initially receives the feedback (typically the project chemist) is responsible for documenting the complaint. If the project chemist is unable to satisfy the customer, the complaint is brought to the attention of the Client Services Manager, Laboratory Director, or QA Manager for final resolution. The complaint and resolution are documented. The procedure is described in the *SOP for Handling Customer Feedback* (ADM-FDBK).

Figure 15-1

Nonconformity and Corrective Action Report

NCAR No: *Assigned by QA*

PROCEDURE (SOP or METHOD): EVENT DATE: _____

EVENT:
- ☐ Missed Holding Time
- ☐ Method Blank Contamination
- ☐ Equipment Failure
- ☐ SOP Deviation
- ☐ QC Failure
- ☐ Login Error
- ☐ Unacceptable PT Sample Result
- ☐ Other (describe):
- ☐ Lab Error (spilled sample, spiking error, etc.)
- ☐ Project Management Error

INCLUDE NUMBER OF SAMPLES / PROJECTS / CUSTOMERS / SYSTEMS AFFECTED

DETAILED DESCRIPTION

ORIGINATOR: _____ DATE: _____

PROJECT MANAGER(S): _____ NOTIFIED BY: _____ DATE: _____

ROOT CAUSE OF NON-CONFORMITY (POTENTIAL CAUSES COULD BE TRAINING, COMMUNICATION, SPECIFICATIONS, EQUIPMENT, KNOWLEDGE)

What is the cause of the error or finding:

CORRECTIVE ACTION AND OUTCOME

Re-establishment of conformity must be demonstrated and documented. Describe the steps that were taken, or are planned to be taken, to correct the particular Nonconformity and prevent its reoccurrence. Include Project Manager Instructions here.

Is the data to be flagged in the Analytical Report with an appropriate qualifier? ☐ **No** ☐ **Yes**

APPROVAL AND NOTIFICATION

Supervisor Verification and Approval of Corrective Action _____ Date: _____
Comments:

QA PM Verification and Approval of Corrective Action _____ Date: _____
Comments:

Project Manager Verification and Approval of Corrective Action _____ Date: _____
Comments:

Customer Notified by ☐ Telephone ☐ Fax ☐ E-mail ☐ Narrative ☐ Not notified

(Attach record or cite reference where record is located.)

16.0 QUALITY ASSURANCE REPORTS

Quality assurance requires an active, ongoing commitment by Columbia Analytical personnel at all levels of the organization. Communication and feedback mechanisms are designed so that analysts, supervisors and managers are aware of QA issues in the laboratory. Analysts performing routine testing are responsible for generating a data quality narrative or data review document with every analytical batch processed. This report also allows the analyst to provide appropriate notes and/or a narrative if problems were encountered with the analyses. A Non-Conformity and Corrective Action Report (NCAR) (see Section 15.0) may also be attached to the data prior to review. Supervisors or qualified analysts review all of the completed analytical batches to ensure that all QC criteria have been examined and any deficiencies noted and addressed.

It is the responsibility of each laboratory unit to provide the project chemist with a final report of the data, accompanied by signature approval. Footnotes and/or narrative notes must accompany any data package if problems were encountered that require further explanation to the client. Each data package is submitted to the appropriate project chemist, who in turn reviews the entire collection of analytical data for completeness and to ensure that any and all client-specified objectives were successfully achieved. A case narrative is written by the project chemist to explain any unusual problems with a specific analysis or sample, etc.

The Quality Assurance Manager (QAM) provides overview support to the project chemists as required (e.g., contractually specified, etc.). The QAM is also responsible for the oversight of all internal and external audits, for all proficiency testing sample and analysis programs, and for all laboratory certification/accreditation responsibilities. The QAM provides the Laboratory Director with quarterly reports that summarize the various QA/QC activities that occurred during the previous quarter. The report addresses such topics as the following:

- Status, schedule, and results of internal and external audits;
- Status, schedule, and results of internal and external proficiency testing studies;
- Status of certifications, accreditations, and approvals;
- Status of QA Manual and SOP review and revision;
- Status of MDLs studies;
- Discussion of QC problems in the laboratory;
- Discussion of corrective action program issues;
- Status of staff training and qualification; and
- Other topics as appropriate.

The Laboratory Director also performs an annual management review of the quality and management systems to identify any necessary changes or improv ements to the quality system or quality assurance policies. This review is documented in a report *Management Quality System and Testing Review* and sent to senior management.

17.0 PERSONNEL TRAINING

Technical position descriptions are available for all employees, regardless of position or level of seniority. These documents are maintained by the Human Resources personnel and are available for review. In order to assess the technical capabilities and qualifications of a potential employee, all candidates for employment at Columbia Analytical are evaluated, in part, against the appropriate technical description.

Training begins the first day of employment at Columbia Analytical when the company policies are presented and discussed. Safety and QA/QC requirements are integral parts of all technical SOPs and, consequently, are integral parts of all training processes at Columbia Analytical. Safety training begins with the reading of the *Environmental Health and Safety Manual.* Employees are also required to attend periodic safety meetings where additional safety training may be performed by the Environmental, Health and Safety Officer.

Employees are responsible for complying with the requirements of the QA Manual and QA/QC requirements associated with their function(s). Quality Systems training begins with Quality Assurance orientation for new employees and reading the *Quality Assurance Manual.* During the employees first year, the employee attends *Core Ethics* training and learns about Columbia Analytical Services quality systems. Each employee participates in annual *Ethics Refresher* training, which is part of the Columbia Analytical Improper Practices Prevention Program.

Columbia Analytical also encourages its personnel to continue to learn and develop new skills that will enhance their performance and value to the Company. Ongoing training occurs for all employees through a variety of mechanisms. The "CAS University" education system, external and internal technical seminars and training courses, and laboratory-specific training exercises are all used to provide employees with professional growth opportunities.

All technical training is documented and records are maintained in the QA department. Training requirements and its documentation are described in the SOP (ADM-TRANDOC) *Documentation of Training.* A training plan is developed whenever an employee starts a new procedure to new position. The training plan includes a description of the step-by-step process for training an employee and for initial demonstration of capability. Where the analyst performs the entire procedure, a generic training plan may be used.

17.1 Initial Demonstration of Capability (IDOC)

Training in analytical procedures typically begins with the reading of the Standard Operating Procedure (SOP) for the method. Hands-on training begins with the observation of an experienced analyst performing the method, followed by the trainee performing the method under close supervision, and culminating with independent performance of the method on quality control samples. Successful completion of the applicable Demonstration of Capability analysis qualifies the analyst to perform the method independently. Demonstration of Capability is performed by one of the following:

- Successful completion of an Initial Precision and Recovery (IPR) study (required where mandated by the method).
- Analysis of 4 consecutive Laboratory Control Samples, with acceptable accuracy and precision.
- Where spiking is not possible but QC standards are used ("non-spiked" Laboratory Control Samples), analysis of 4 consecutive Laboratory Control Samples with acceptable accuracy and precision.
- Where one of the three above is not possible, special requirements are as follows:
    - Total Settleable Solids: Successful single-blind PT sample analysis and duplicate results with RPD<10%.
    - Color: Four consecutive prepared LCSs with acceptable accuracy and precision of <10% RSD.
    - Physical Tests (Grain size, Corrosivity to Steel, etc.): Supervisor acknowledgement of training and approval.

A flowchart identifying the Demonstration of Proficiency requirements is given in Figure 17-1. The flowchart identifies allowed approaches to assessing Demonstration of Capability when a 4-replicate study is not mandated by the method, when spiking is not an option, or when QC samples are not readily available.

17.2 Continuing Demonstration of Proficiency

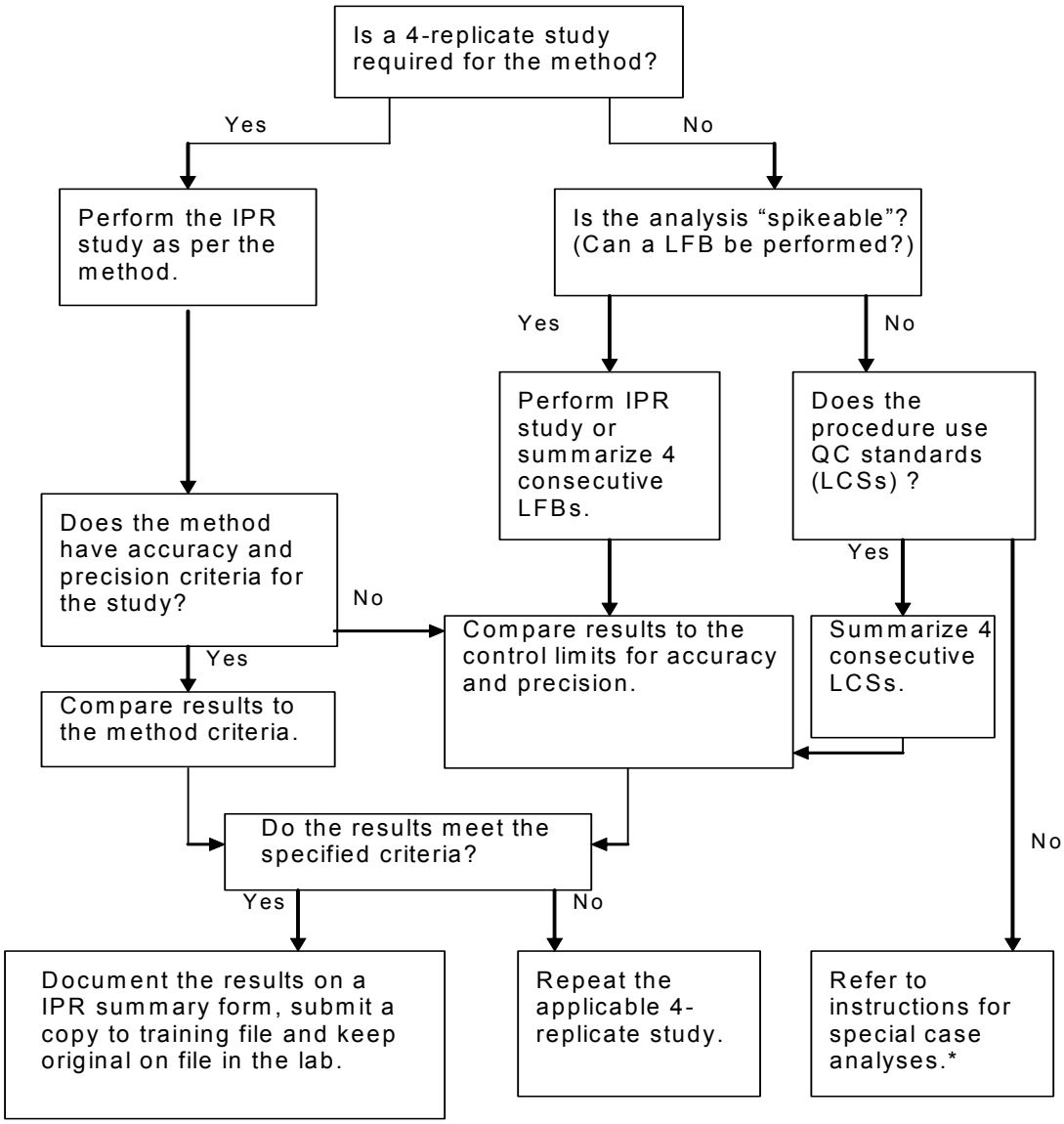
A periodic demonstration of proficiency is required to maintain continuing qualification. Continuing Demonstration of Proficiency is required each year, and may be performed one of the following ways:

- Successful performance on external (independent) single-blind sample analyses using the test method, or a similar test method using the same technology. I.e. PT sample or QC sample blind to the analyst.
- Performing Initial Demonstration of Capability as described above, with acceptable levels of precision and accuracy.
- Analysis of at least 4 consecutive LCSs with acceptable levels of accuracy and precision from in-control analytical batches.
- If the above cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.
- For methods for which PT samples are not available and a spiked analysis (LFB, MDL, etc.) is not possible, analysis of field samples that have been analyzed by another analyst with statistically indistinguishable results.

17.3 Documentation of Training

Records are maintained to indicate the employee has the necessary training, education, and experience to perform their functions. Information of previously acquired skills and abilities for a new employee is maintained in Human Resources personnel files and Columbia Analytical resumes. QA maintains a database to record the various technical skills and training acquired while employed by Columbia Analytical. Information includes the employee's name, a description of the skill including the appropriate method and SOP reference, the mechanism used to document proficiency, and the date the training was completed. General procedures for documenting technical training are described in the *SOP for Documentation of Training (SOP No. ADM-TRANDOC).*

Figure 17-1
Initial Demonstration of Capability Requirements[a]



[a] For IDOC IPR or LFB studies, "second-source" reference materials are used, as per NELAP requirements
*Total Settleable Solids: Successful PT sample analysis and duplicate results with RPD<10%.
*Color: Four consecutive prepared LCSs with acceptable accuracy and precision of <10% RSD.
* Physical Tests (Grain size, Corrosivity to Steel, et c.): Supervisor acknowledgement of training and approval.

18.0 REFERENCES FOR ANALYTICAL PROCEDURES – EXTERNAL DOCUMENTS

The analytical methods used at Columbia Analytical generally depend upon the end-use of the data. Since most of our work involves the analysis of environmental samples for regulatory purposes, specified federal and/or state testing methodologies are used and followed closely. Typical methods used at Columbia Analytical are taken from the following references:

- National Environmental Laboratory Accreditation Program (NELAP), 2003 Quality Standards.
- American National Standard *General requirements for the competence of testing and calibration laboratories,* ANSI/ISO/IEC 17025:2005(E)
- *Department of Defense Quality Systems Manual for Environmental Laboratories*, Final Version 3 (January 2006).
- *DoD Quality Systems Manual for Environmental Laboratories,* Version 4.1, 4/22/2009
- *Good Automated Laboratory Practices, Principles and Guidance to Regulations For Ensuring Data Integrity In Automated Laboratory Operations*, EPA 2185 (August 1995).
- *Manual for the Certification of Laboratories Analyzing Drinking Water*, 4th Edition, EPA 815-B-97-001 (March 1997).
- *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, (September 1986) and Updates I (July 1992), II (September 1994), IIA (August 1993), IIB (January 1995), III (December 1996), Final Update IV (February 2007), and updates posted online at http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm. See Chapters 1, 2, 3, and 4.
- *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, (Revised March 1983).
- *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA/600/R-93/100 (August 1993).
- *Methods for the Determination of Metals in Environmental Samples*, EPA/600/4-91/010 (June 1991) and Supplements.
- *Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater*, EPA 600/4-82-057 (July 1982) and 40 CFR Part 136, Appendix A.
- *Methods for the Determination of Organic Compounds in Drinking Water*, EPA/600/4-88/039 (December 1988) and Supplements.
- *Standard Methods for the Examination of Water and Wastewater*, 18th Edition (1992); 19th Edition (1995), 20th Edition (1998). See Introduction in Part 1000.
- 40 CFR Part 136, Guidelines for Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act.
- 40 CFR Part 141, National Primary Drinking Water Regulations.

- *Analytical Methods for Petroleum Hydrocarbons*, ECY 97-602, Washington State Department of Ecology, June 1997.
- State-specific total petroleum hydrocarbon methods for the analysis of samples for gasoline, diesel, and other petroleum hydrocarbon products (Alaska, Arizona, California, Oregon, Washington, Wisconsin, etc.).
- Annual Book of ASTM Standards, Part 31, Water.
- EPA Contract Laboratory Program, Statement of Work for Organic Analysis, SOW Nos. OLM03.1, OLM03.2, OLM04.2, and OLM04.3.
- EPA Contract Laboratory Program, Statement of Work for Inorganic Analysis, SOW No. ILM04.0, ILM04.1, and ILM05.2.
- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*, EPA-540/R-94/012 (February 1993).
- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, EPA-540/R-94/013 (February 1994).
- National Institute for Occupational Safety and Health (NIOSH) *Manual of Analytical Methods*, Third Edition (August 1987); Fourth Edition (August 1994).
- *Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound*, for USEPA and USACE (March 1986), with revisions through April 1997.
- WDOE 83-13, *Chemical Testing Methods for Complying with the State of Washington Dangerous Waste Regulations* (March 1982) and as Revised (July 1983 and April 1991).
- *Identification and Listing of Hazardous Waste,* California Code of Regulations, Title 22, Division 4.5, Chapter 11.
- *Analytical Methods for the Determination of Pollutants in Pulp and Paper Industry Wastewater*, EPA 821-R-93-017 (October 1993).
- *Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewaters*, EPA 821-B-98-016 (July 1998).
- National Council of the Pulp and Paper Industry for Air and Stream Improvement (NCASI).

APPENDIX A

LIST of QA PROGRAM DOCUMENTS

and

STANDARD OPERATING PROCEDURES

QA Program Files

Quality Assurance Manual	10/2/2009
Software Quality Assurance Plan	7/11/05
CAS-Kelso Certifications/Accreditations	Cert_kel.xls
Columbia Analytical Services MDL Tracking Spreadsheet	Mdl_list.xls
Technical Training Summary Database	TrainDat.mdb
Approved Signatories List	AppSignatories.pdf
Personnel resumes/qualifications	HR Department
Personnel Job Descriptions	HR Department
Quality Control Acceptance Criteria	Qclimits.xls
Master Logbook of Laboratory Logbooks	Masterlog-001
Standard Operating Procedure Database	TrainDat.mdb

Corporate – Policies

POLICY TITLE	POLICY DATE	DATE APPROVED	DATE EFFECTIVE
CAS Quality and Ethics Policy Statement	March 2009	3/19/09	3/19/09
Policy for Data Review and Validation	May 2009	5/5/09	7/1/09
Policy for Internal Quality Assurance Audits	May 2009	5/5/09	7/1/09
Policy for Standards and Reagents Expiration Dates	September 2009	Final draft	9/28/09
Policy for Quality Assurance for Non-Regulated Testing	Draft	-	-
Policy for Use of Accreditation Organization's Name, Symbols, and Logos	Draft	-	-
Policy for Conducting Research, Technical Investigations, and Method Development	In development	-	-

Administrative SOP Corporate

SOP TITLE	SOP Code	Rev	SOP Date
SOP for Checking New Lots of Chemicals for Contamination	ADM-CTMN	4	1/26/09
SOP for Control Limits	ADM-CTRL_LIM	6	9/28/07
SOP for Corrective Action	ADM-CA	5	9/12/07
SOP for Data Recall	ADM-DATARECALL	0	9/21/07
SOP for Document Control	ADM-DOC_CTRL	7	1/27/09
SOP for Documentation of Training	ADM-TRANDOC	10	12/6/07
SOP for Estimation of Uncertainty of Measurements	ADM-UNCERT	4	12/30/08
SOP for Handling Customer Feedback	ADM-FDBK	4	12/10/07
SOP for Making Entries into Logbooks and onto Benchsheets	ADM-DATANTRY	8	9/8/09
SOP for Managerial Review of the Laboratory's Quality Systems	ADM-MGMTRVW	2	11/7/07
SOP for Manual Integration of Chromatographic Peaks	ADM-INT	3	8/28/07
SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation	ADM-MDL	9	9/8/09
SOP for Preparation of Electronic-data for Organic Analyses for Electronic-data Audits	ADM-E_DATA	3	8/29/07
SOP for Preparation of SOPs	ADM-SOP	8	11/14/08
SOP for Preventive Action	ADM-PA	0	11/14/08
SOP for Proficiency Testing Sample Analysis	ADM-PTS	1	9/28/07
SOP for Purchasing Through SOP Purchasing Agent in Kelso	ADM-PUR	2	12/10/07
SOP for Qualification of Subcontract Laboratories Outside of SOP Network	ADM_SUBLAB	4	12/29/08
SOP for Significant Figures	ADM-SIGFIG	8	1/28/09

Administrative SOP Kelso

SOP Title	FILE NAME
CHECKING PIPETTE CALIBRATION	**ADM-CPIP**
CONTINGENCY PLAN FOR LABORATORY EQUIPMENT FAILURE	**ADM-ECP**
CONTROL CHARTING QUALITY CONTROL DATA	**ADM-CHRT**
DATA ARCHIVING	**ADM-ARCH**
DATA REPORTING AND REPORT GENERATION	**ADM-RG**
DEPARTMENT OF DEFENSE PROJECTS LABORATORY PRACTICES AND PROJECT MANAGEMENT	**ADM-DOD**
ELECTRONIC DATA BACKUP AND ARCHIVING	**ADM-EBACKUP**
INTERNAL QUALITY ASSURANCE AUDITS	**ADM-IAUD**
LABORATORY BALANCE MONITORING AND CALIBRATION	**ADM-BAL**
LABORATORY DATA REVIEW PROCESS	**ADM-DREV**
PROJECT MANAGEMENT	**ADM-PCM**
REAGENT LOGIN AND TRACKING	**ADM-RLT**
SUPPORT EQUIPMENT MONITORING AND CALIBRATION	**ADM-SEMC**
SAMPLE BATCHES	**ADM-BATCH**
SAMPLE MANAGEMENT SOPS	**FILE NAME**
BOTTLE ORDER PREPARATION AND SHIPPING	SMO-BORD
FOREIGN SOILS HANDLING TREATMENT	SMO-FSHT
SAMPLE DISPOSAL	SMO-SDIS
SAMPLE RECEIVING	SMO-GEN
SAMPLE TRACKING AND LABORATORY CHAIN OF CUSTODY	SMO-SCOC

Technical SOP Kelso

SOP Title	FILE NAME
COLIFORM, TOTAL (DRINKING WATER)	BIO-9221DW
COLIFORM, FECAL	BIO-9221FC
COLIFORM, TOTAL	BIO-9221TC
COLIFORM, FECAL (MEMBRANE FILTER PROCEDURE)	BIO-9222D
COLILERT® and COLITAG	BIO-9223
FECAL STREPTOCOCCUS/ENTEROCOCCUS	BIO-9230B
COLILERT® COMPLETED TEST VERIFICATION OF E. COLI IN MUG CULTURES	BIO-CCT
ENTEROLERT	BIO-ENT
HEPTEROTROPHIC PLATE COUNT	BIO-HPC
MICROBIOLOGY QUALITY ASSURANCE AND QUALITY CONTROL	BIO-QAQC
SHEEN SCREEN/OIL DEGRADING MICROORGANISMS	BIO-SHEEN
EPA CLP ORGANICS ANALYSES	CLP_ORGA
SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION	EXT-3510
CONTINUOUS LIQUID - LIQUID EXTRACTION	EXT-3520
SOLID PHASE EXTRACTION	EXT-3535
SOXHLET EXTRACTION	EXT-3540
AUTOMATED SOXHLET EXTRACTION	EXT-3541
ULTRASONIC EXTRACTION	EXT-3550
WASTE DILUTION EXTRACTION	EXT-3580
SILICA GEL CLEANUP	EXT-3630
REMOVAL OF SULFUR USING COPPER	EXT-3660
REMOVAL OF SULFUR USING MERCURY	EXT-3660M
SULFURIC ACID CLEANUP	EXT-3665
CARBON CLEANUP	EXT-CARCU
DIAZOMETHANE PREPARATION	EXT-DIAZ
FLORISIL CLEANUP	EXT-FLOR
ORGANIC EXTRACTIONS GLASSWARE CLEANING	EXT-GC
PREPARATION OF REAGENTS AND BLANK MATRICES USED IN SEMIVOLATILE ORGANICS ANALYSIS	EXT-REAG
ADDITION OF SPIKES AND SURROGATES	EXT-SAS
SOLID PHASE DISPERSION IN TISSUES	EXT-SPD
MEASURING SAMPLE WEIGHTS AND VOLUMES FOR ORGANIC ANALYSIS	EXT-WVOL
FACILITY AND LABORATORY CLEANING	FAC-CLEAN
OPERATION AND MAINTENANCE OF LABORATORY REAGENT WATER SYSTEMS	FAC-WATER
FLASHPOINT DETERMINATION - SETAFLASH	GEN-1020
COLOR	GEN-110.2
HARDNESS, TOTAL	GEN-130.2
SOLIDS, TOTAL DISSOLVED (TDS)	GEN-160.1
SOLIDS, TOTAL SUSPENDED (TSS)	GEN-160.2
TOTAL SOLIDS	GEN-160.3
SOLIDS, TOTAL VOLATILE AND PERCENT ASH IN SOIL AND SOLID SAMPLES	GEN-160.4
SETTEABLE SOLIDS	GEN-160.5
HALIDES, ADSORBABLE ORGANIC (AOX)	GEN-1650
DETERMINATION OF INORGANIC ANIONS IN DRINKING WATER BY ION CHROMATOGRAPHY	GEN-300.1
ACIDITY	GEN-305.2
ALKALINITY TOTAL	GEN-310.1

PERCHLORATE BY ION CHROMATOGRAPHY	GEN-314.0
CHLORIDE (TITRIMETRIC, MERCURIC NITRATE)	GEN-325.3
CHLORINE, TOTAL/FREE RESIDUAL	GEN-330.4
TOTAL RESIDUAL CHLORINE - METHOD 330.5	GEN-330.5
TOTAL CYANIDES AND CYANIDES AMENABLE TO CHLORINATION	GEN-335
AMMONIA BY FLOW INJECTION ANALYSIS	GEN-350.1
AMMONIA AS NITROGEN BY ION SPECIFIC ELECTRODE	GEN-350.3
NITRATE/NITRITE, NITRITE BY FLOW INJECTION ANALYSIS	GEN-353.2
NITRITE BY COLORIMETRIC PROCEDURE	GEN-354.1
PHOSPHORUS DETERMINATION USING COLORMETRIC PROCEDURE	GEN-365.3
DISSOLVED SILICA	GEN-370.1
GRAVIMETRIC SULFATE	GEN-375.3
SULFIDE, TITRIMETRIC (IODINE)	GEN-376-1
SULFIDE, METHYLENE BLUE	GEN-376-2
PHENOLICS, TOTAL	GEN-420.1
MBAS	GEN-425.1
HALOGENS TOTAL AS CHLORIDE BY BOMB COMBUSTION	GEN-5050
BIOCHEMICAL OXYGEN DEMAND	GEN-5210B
HALIDES, ADSORBABLE ORGANIC (AOX) - SM 5320B	GEN-5320B
TANNIN AND LIGNIN	GEN-5550
CYANIDE EXTRACTION OF SOLIDS AND OILS	GEN-9013
HALIDES, TOTAL ORGANIC (TOX)	GEN-9020
HALIDES, EXTRACTABLE ORGANIC (EOX)	GEN-9020M
TOTAL SULFIDES BY METHYLENE BLUE DETERMINATION	GEN-9030
TOTAL HALIDES BY OXIDATIVE COMBUSTION AND MICROCOULOMETRY	GEN-9076
CARBON, TOTAL ORGANIC IN SOIL	GEN-ASTM
AUTOFLUFF	GEN-AUTOFLU
SULFIDES, ACIDS VOLATILE	GEN-AVS
HEAT OF COMBUSTION	GEN-BTU
CYANIDE, WEAK ACID DISSOCIABLE	GEN-CNWAD
CHEMICAL OXYGEN DEMAND	GEN-COD
CONDUCTIVITY IN WATER AND WASTES	GEN-COND
CORROSIVITY TOWARDS STEEL	GEN-CORR
HEXAVALENT CHROMIUM - COLORIMETRIC	GEN-CR6
CARBONATE (CO3) BY EVOLUTION AND COLUMETRIC TITRATION	GEN-D513-82M
SULFIDE, SOLUBLE DETERMINATION OF SOLUBLE SULFIDE IN SEDIMENT	GEN-DIS.S2
BULK DENSITY OF SOLID WASTE FRACTIONS	GEN-E1109
FERROUS IRON IN WATER	GEN-FeII
FLUORIDE BY ION SELECTIVE ELECTRODE	GEN-FISE
FORMALDEHYDE COLORIMETRIC DETERMINATION	GEN-FORM
HYDROGEN HALIDES BY ION CHROMATOGTRAPHY (METHOD 26)	GEN-HA26
MERCURY IN COAL SAMPLE PREPARATION BY PARR BOMB COMBUSTION	GEN-HGPREP
HYDAZINE IN WATER USING COLORIMETRIC PROCEDURE	GEN-HYD
TOTAL SULFUR FOR ION CHROMATOGRAPHY	GEN-ICS
ION CHROMATOGRAPHY	GEN-IONC
COLOR, NCASI	GEN-NCAS
OXYGEN CONSUMPTION RATE	GEN-O2RATE
CARBON, TOTAL ORGANIC DETERMINATION (WALKELY BLACK METHOD)	GEN-OSU
Ph IN SOIL AND SOLIDS	GEN-Phs

Ph IN WATER	GEN-Phw
PARTICLE SIZE DETERMINATION - ASTM PROCEDURE	GEN-PSASTM
PARTICLE SIZE DETERMINATION	GEN-PSP
SULFIDES, REACTIVE	GEN-RS
TOTAL SULFIDE BY PSEP	GEN-S2PS
SULFITE	GEN-SO3
SPECIFIC GRAVITY	GEN-SPGRAV
SUBSAMPLING AND COMPOSITING OF SAMPLES	GEN-SUBS
THIOCYANATE	GEN-THIOCN
NITROGEN, TOTAL AND SOLUBLE KJELDAHL	GEN-TKN
POST DIGESTION DETERMINATION OF TOTAL KJELDAHL NITROGEN BY SEMIAUTOMATED COLORIMETRY	GEN-TKNAA
TOTAL ORGANIC CARBON IN WATER	GEN-TOC
TURBIDITY MEASUREMENT	GEN-TURB
ULTIMATE BOD	GEN-UBOD
GLASSWASHING FOR INORGANIC ANALYSES	GEN-WASH
Quantitative Determination of Carbamate Pesticides by High Performance Liquid Chromatography/Tandam Mass Spectrometry (HPLC/MS/MS)	LCP-8321
NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY(HPLC)	LCP-8330B
QUANTITATION OF NITROAROMATICS AND NITRAMINES IN WATER, SOIL, AND TISSUE BY LIQUID CHROMATOGRAPHY AND TANDEM MASS SPECTROMETRY (LC-MS/MS)	LCP-LCMS4
NITROGUANIDINE BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	LCP-NITG
QUANTITATION OF NITROPHENOLS IN SOLIS BY LIQUID CHROMATOGRAPHYAND TANDEM MASS SPECTORMETRY (LC-MS/MS)	LCP-NITRO
METHYL MERCURY IN SOIL AND SEDIMENT BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630S
METHYL MERCURY IN TISSUE BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630T
METHYL MERCURY IN WATER BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630W
MERCURY IN WATER BY OXIDATION, PURGE&TRAP, AND COLD VAPOR ATOMIC FLUORES. SPECTROMETRY	MET-1631
MERCURY IN WATER	MET-245.1
METALS DIGESTION	MET-3005A
METALS DIGESTION	MET-3010A
METALS DIGESTION	MET-3020A
METALS DIGESTION	MET-3050B
CLOSED VESSEL OIL DIGESTION	MET-3051M
DETERMINATION OF METALS & TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 6020)	MET-6020
ARSENIC BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION	MET-7062
METALS DIGESTION	MET-7195
MERCURY IN LIQUID WASTE	MET-7470A
MERCURY IN SOLID OR SEMISOLID WASTE	MET-7471A/B
SELENIUM BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION	MET-7742
CATION-EXCHANGE CAPACITYOF SOILS (SODIUM ACETATE) - METHOD 9081	MET-9081
SAMPLE PREPARATION OF AQUEOUS SAMPLES BY "CLEAN" TECHNIQUES	MET-ACT
BIOACCESSIBILITY OF METALS IN SOIL AND SOLID WASTE	MET-BIOACC
METALS DIGESTION	MET-DIG
FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRIC ANALYSES	MET-FAA
SAMPLE FILTRATION FOR METALS ANALYSIS	MET-FILT
METALS LABORATORY GLASSWARE CLEANING	MET-GC

DETERMINATION OF TRACE METALS BY GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROMETRY (GFAA)	MET-GFAA
DETERMINATION OF METALS AND TRACE ELEMENTS BY ICP/AES	MET-ICP
DETERMINATION OF METALS & TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 200.8)	MET-ICP.MS
MULTIPLE EXTRACTION PROCEDURE	MET-MEP
TRACE METALS IN WATER BY PRECONCENTRATION USING REDUCTIVE PRECIPITATION FOLLOWED BY ICP-MS	MET-RPMS
WASTE EXTRACTION TEST (WET) PROCEDURE (STLC) for NONVOLATILE and SEMIVOLATILE PARAMETERS	MET-STLC
METALS AND SEMIVOLATILES TCLP EXTRACTION (EPA METHOD 1311)	MET-TCLP
SAMPLE PREPARATION OF BIOLOGICAL TISSUES FOR METALS ANALYSIS BY GFAA, ICP-OES, AND ICP-MS	MET-TDIG
TISSUE SAMPLE PREPARATION	MET-TISP
GRAVIMETRIC DETERMINATION OF HEAXANE EXTRACTABLE MATERIAL (1664)	PET-1664
GASOLINE RANGE ORGANICS BY GAS CHROMATOGRAPHY	PET-GRO
ANALYSIS OF WATER, SOLIDS AND SOLUBLE WASTE SAMPLES FOR SEMI-VOLATILE FUEL HYDROCARBONS	PET-SVF
ANALYSIS OF SOLID AND AQUEOUS SAMPLES FOR STATE OF WISCONSIN DIESEL RANGE ORGANICS	PHC-WIDRO
BOTTLE ORDER PREPARATION AND SHIPPING	SMO-BORD
FOREIGN SOILS HANDLING TREATMENT	SMO-FSHT
SAMPLE RECEIVING	SMO-GEN
SAMPLE TRACKING AND INTERNAL CHAIN OF CUSTODY	SMO-SCOC
SAMPLE DISPOSAL	SMO-SDIS
CHLORINATED PHENOLICS BY IN-SITU ACETYLATION AND GC/MS	SOC-1653A
PHARMACEUTICALS, PERSONAL CARE PRODUCTS AND ENDOCRINE DISRUPTING COMPOUNDS IN WATER BY HPLC/TANDEM MASS SPECTROMETRY (HPLC/MS/MS)	SOC-1694
1,8-DIHYDROXYANTHRAQUINONE BY GC/MS SIM	SOC-18DHYDRAQ
GEL PERMEATION CHROMATOGRAPHY	SOC-3640A
ACETAMIDE HERBICIDE DEGRADATES IN DRINKING WATER BY SPE AND HPLC/MS/MS	SOC-535
ORGANOCHLORINE PESTICIDES AND PCBs (METHOD 608)	SOC-608
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS	SOC-625
GLYCOLS	SOC-8015M
ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY: CAPILLARY COLUMN TECHNIQUE	SOC-8081
PCBS AS AROCLORS - METHOD 8082A	SOC-8082AAr
CONGENER-SPECIFIC DETERMINATION OF PCBS BY GC/ECD - METHOC 8082A	SOC-8082ACo
PCBS AS AROCLORS	SOC-8082Ar
CONGENER-SPECIFIC DETERMINATION OF PCBS BY GC/ECD	SOC-8082C
DETERMINATION OF NITROGEN OR PHOSPHORUS CONTAINING PESTICIDES	SOC-8141
CHLORINATED HERBICIDES	SOC-8151
CHLORINATED PHENOLS METHOD 8151 MODIFIED	SOC-8151M
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS	SOC-8270C
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS - METHOD 8270D	SOC-8270D
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS - LOW LEVEL PROCEDURE	SOC-8270L
POLYNUCLEAR AROMATIC HYDROCARBONS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY SIM	SOC-8270P
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTED ION MONITORING	SOC-8270S
POLYNUCLEAR AROMATIC HYDROCARBONS BY HPLC	SOC-8310
ALDEHYDES BY HPLC	SOC-8315A

NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	SOC-8330
NITROGLYCERIN AND PETN BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	SOC-8332
RESIN AND FATTY ACIDS BY GC/MS - NCASI METHOD 85.02 MODIFIED	SOC-85.02
METHANOL IN PROCESS LIQUIDS AND STATIONARY SOURCE EMISSIONS	SOC-9403
HAZARDOUS AIR POLLUTANTS (HAPS) IN PULP AND PAPER INDUSTRY CONDENSATES	SOC-9901
HAPS AND OTHER COMPOUNDS IN IMPINGER/CANISTER SAMPLES FROM WOOD PRODUCTS FACILITIES	SOC-9902
BUTYLTINS	SOC-BUTYL
CALIBRATION OF INSTRUMENTS FOR ORGANICS CHROMATOGRAPHIC ANALYSES	SOC-CAL
CALIBRATION OF INSTRUMENTS FOR ORGANICS CHROMATOGRAPHIC ANALYSES USING EPA 8000C	SOC-CAL8000C
CONFIRMATION PROCEDURE FOR GC AND HPLC ANALYSES	SOC-CONF
CPSC PHTHALATES BY GC/MS SELECTIVE ION MONITORING	SOC-CPSC
DIMP	SOC-DIMP
DMD SYNTHESIS	SOC-DMD
TOTAL OLEANOLIC ACID SAPONINS IN WATER BY ACID HYDROLYSIS AND HPLC/MS/MS	SOC-LCMS3
PERCENT LIPIDS IN TISSUE	SOC-LIPID
MONOCHLOROACETIC ACID BY GC-ECD	SOC-MCA
NONYLPHENOLS ISOMERS AND NONYLPHENOL ETHOXYLATES	SOC-NONYL
ORGANIC ACIDS IN AQUEOUS MATRICES BY HPLC	SOC-OALC
EXTRACTION METHOD FOR ORGANOTINS IN SEDIMENTS, WATER, AND TISSUE	SOC-OSWT
CHLORINATED PESTICIDES BY GC/MS/MS, EPA METHOD 1699 MODIFIED	SOC-PESTMS2
PERFLUORINATED COMPOUNDS BY HPLC/MS/MS	SOC-PFC
PICRIC ACID AND PICRAMIC ACID BY HPLC	SOC-PICRIC
POLYBROMINATED DIPHENYL ETHERS (PBDEs) AND POLYBROMINATED BIPHENYLS (PBBs) BY GC/MS	SOC-ROHS
SEMI-VOLATILE ORGANICS SCREENING	SOC-SCR
1,2-DIBROMOETHANE, 1,2-DIBROMO-3-CHLOROPROPANE, AND 1,2,3-TCP BY GC	SVD-504
ORGANOCHLORINE PESTICIDES AND PCBS IN DRINKING WATER	SVD-508_1
CHLORINATED HEBICIDES IN DRINKING WATER	SVD-515_4
N-NITROSAMINES BY GC/MS/MS	SVD-521
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS (METHOD 525.2)	SVD-525
SELECTED PESTICIDES AND FLAME RETARDANTS IN DRINKING WATER BY GC/MS (EPA METHOD 527)	SVD-527
DETERMINATION OF EXPLOSIVES AND RELATED COMPOUNDS IN DRINKING WATER BY GC/MS	SVD-529
CARBAMATES AND CARBAMOYLOXIMES IN WATER BY POST-COLUMN DERIVITIZATION HPLC	SVD-531 -1
GLYPHOSATE IN DRINKING WATER BY HPLC	SVD-547
ENDOTHALL IN DRINKING WATER BY GC/MS	SVD-548
DIQUAT AND PARAQUAT BY HPLC	SVD-549
HALOACETIC ACIDS IN DRINKING WATER	SVD-552
PURGE AND TRAP FOR AQUEOUS SAMPLES	VOC-5030
PURGE AND TRAP/EXTRACTION FOR VOC IN SOIL AND WASTE SAMPLES , CLOSED SYSTEM	VOC-5035
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-524.2
AROMATIC VOLATILE ORGANICS (BTEX) BY GC - METHOD 602	VOC-602BTEX
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-624
AROMATIC VOLATILE ORGANICS (BTEX) BY GC - METHOD 8021	VOC-8021BTEX
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-8260
VOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTIVE ION MONITORING	VOC-8260S

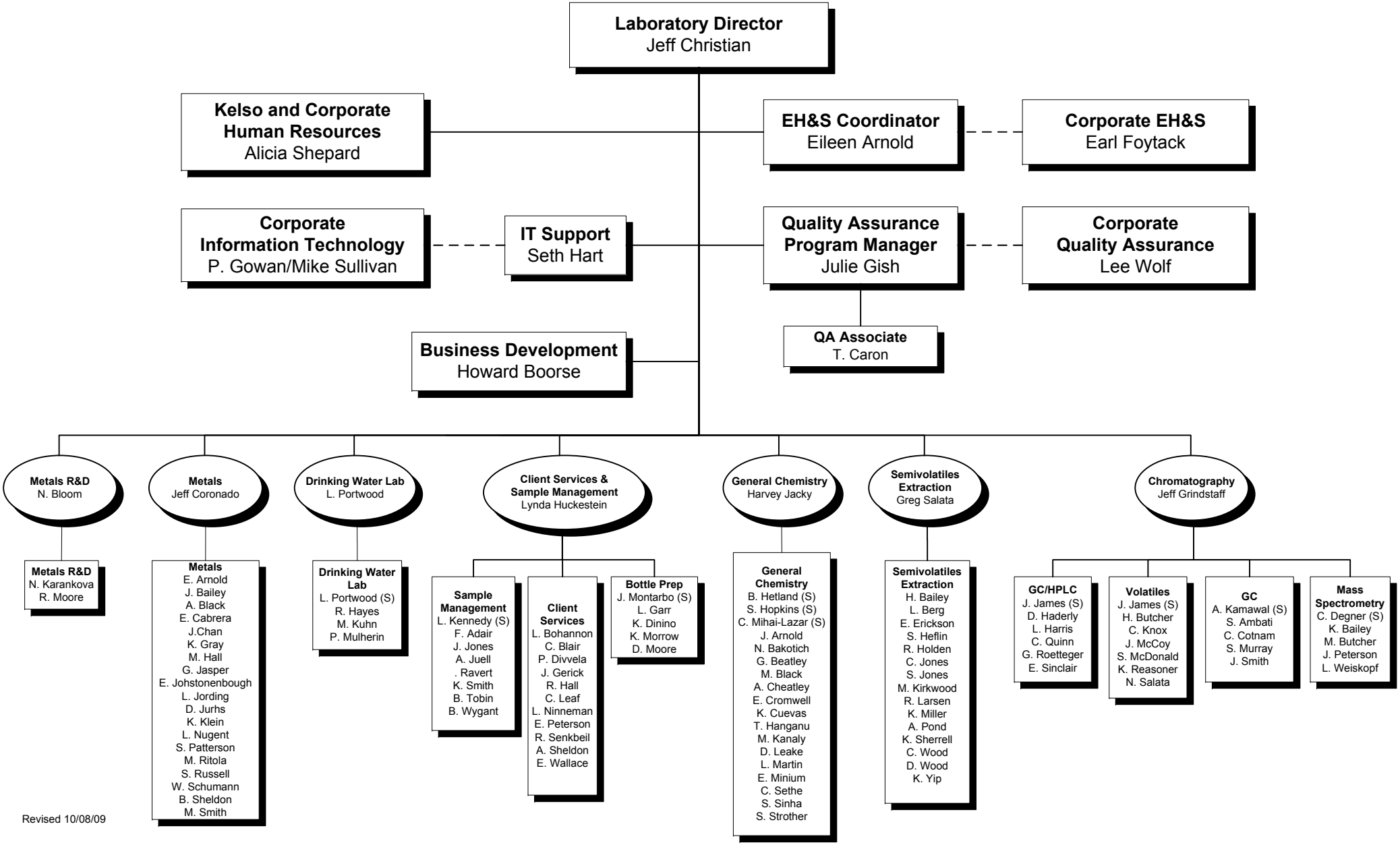
VOA STORAGE BLANKS	VOC-BLAN
SAMPLE SCREENING FOR VOLATILE ORGANIC COMPOUNDS IN SOIL, WATER AND MISC. MATRICES	VOC-BVOC
ZERO HEADSPACE EXTRACTION (EPA METHOD 1311)	VOC-ZHE

APPENDIX B

ORGANIZATIONAL CHARTS and RESUMES OF KEY PERSONNEL

Columbia Analytical Services™

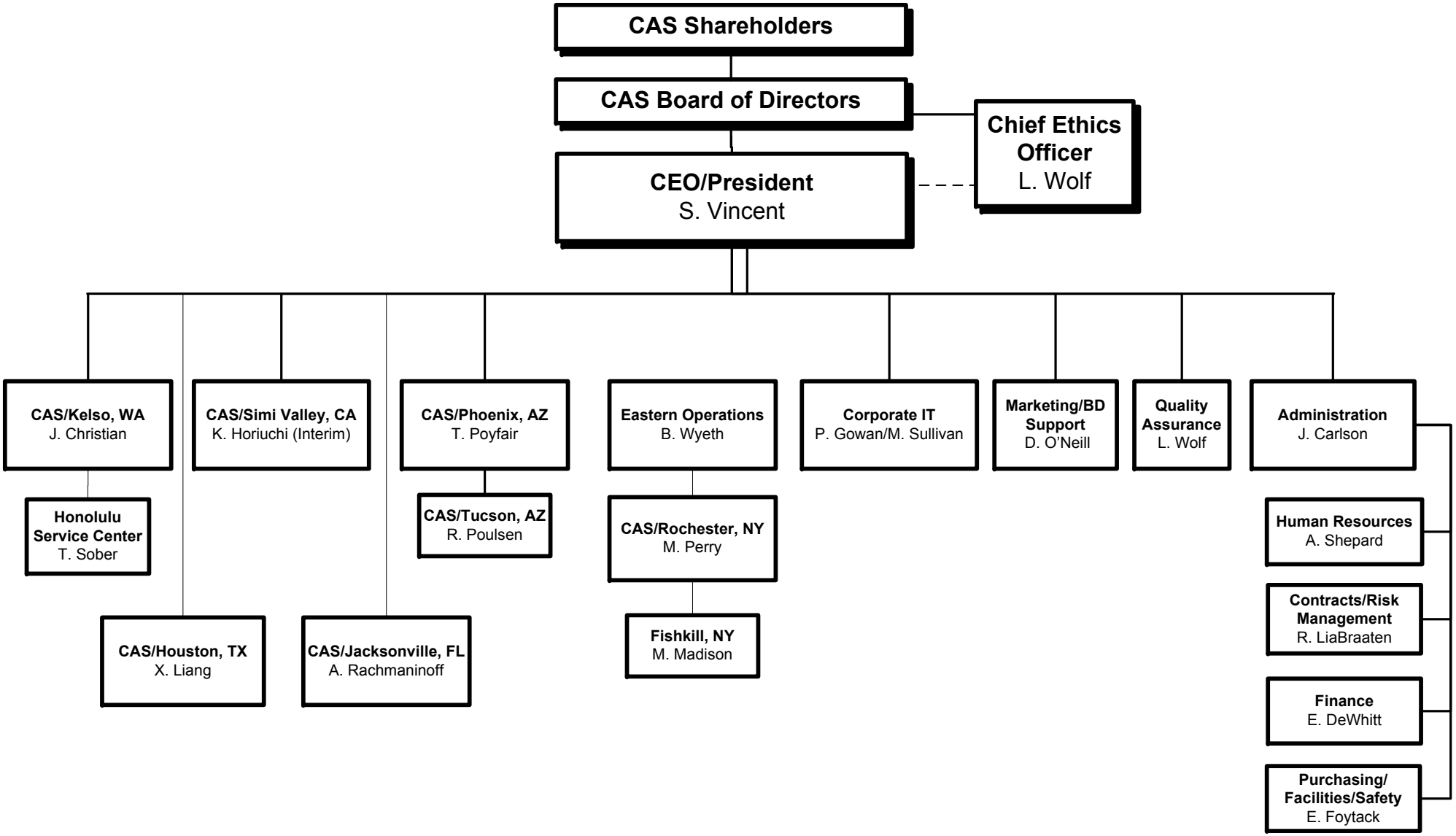
Environmental and General Testing Division
Kelso, Washington
Laboratory Organization



Revised 10/08/09

Columbia Analytical Services™

Laboratory Division Organization



Revised .12/1/09

JEFFREY D. CHRISTIAN

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position

VICE PRESIDENT/NW REGIONAL DIRECTOR – 1996 to Present

Responsibilities

Responsible for all phases of laboratory operations at the Kelso (WA) facility, including project planning, budgeting, and quality assurance. Primary duties include the direct management of the Kelso laboratory (i.e. serves as the Kelso Laboratory Director, 1993- present). Also responsible for additional duties acquired as a member of the Columbia Analytical Services Holdings, Inc., Board of Directors.

Experience

Laboratory Director, Kelso Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1993-1995. Responsible for all phases of laboratory operations, including project planning, budgeting, and quality assurance.

Operations Manager, Kelso Laboratory , *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-1993. Responsibilities included directing the daily operation of the Kelso laboratory. Other responsibilities and duties included functioning as a technical consultant to clients, providing assistance in developing and planning analytical schemes to match client objectives, and writing and developing analytical procedures/methods. Also, served as Project Manager for State of Alaska Department of Environmental Conservation contract and Coordinator for EPA Special Analytical Services (SAS) contracts.

Project Chemist and Manager, Metals Analysis Laboratory, *Columbia Analytical Services, Kelso, Washington,* 1989-1992. Responsible for directing the daily operation of the Metals Laboratory, including the sample preparation, AAS, ICP-OES, and ICP-MS Laboratories.

Scientist, *Weyerhaeuser Technology Center, Federal Way, Washington,* 1986-1989. Responsibilities included supervising atomic spectroscopy laboratory which included flame and furnace AAS, ICP-OES, and sample preparation capabilities to handle a wide variety of sample types. Interfaced with internal and external clients to provide technical support. Wrote and developed analytical procedures/methods.

Lead Technician, Metals Lab, *Weyerhaeuser Technology Center, Federal Way, Washington*, 1981-1986. Responsibilities included primary ICP and AAS analyst for EPA-CLP contract work. Extensive experience in wide variety of environmental and product-related testing.

Research Assistant, *ITT Rayonier, Olympic Research Division, Shelton, Washington,* 1978-1981. Responsibilities included performing water quality tests, product-related analytical tests, corrosion tests (i.e., potentiometric polarization techniques), and operated pilot equipment specific to the pulp and paper industry.

Education

B.S., Chemistry, *Evergreen State College, Olympia, Washington,* 1993.
ICP/MS Training Course, *VG-Elemental,* 1992.
Coursework, *Pacific Lutheran University, Tacoma, Washington.* 1988-1989.
Coursework, *Tacoma Community College, Tacoma, Washington.* 1970-1971, 1988-1989.
Perkin-Elmer Advanced Furnace, *Norwalk, Connecticut,* 1986.
CERTIFICATION, Chemistry, *L.H. Bates Technical, Tacoma, Washington,* 1978.
Coursework, *Central Washington University, Ellensburg, Washington.* 1969-1970.

Publications/ Presentations

Mr. Christian has a number of publications and presentations. For a list of these publications and presentations, please contact CAS.

JULIE GISH

1996 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position

TECHNICAL MANAGER I, KELSO LAB QUALITY ASSURANCE MANAGER – 2008 to Present

Responsibilities

Responsible for the overall implementation of the laboratory QA program. Responsible for the Quality Assurance Manual, certifications, documenting SOPs, and maintaining proficiency testing (PT) records. Oversee balance calibration and sample storage temperature control. Maintain certifications/accreditations for regulatory agencies and client certifications or approval programs. Act as primary point of contact during laboratory audits and provides audit responses and initiates any corrective actions. Coordinate the analysis and reporting of PT samples. Conduct internal audits and make recommendations for corrective action.

Experience

Scientist IV, Semi-Volatile Mass Spectrometry Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 2002-2008. Primary responsibilities were analysis, interpretation and report generation for semivolatile organics by GC/MS. Analyses included EPA 625, 8270, SIM, and other miscellaneous methodology.

Technical Manager I, Semi-Volatile GC Organics Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1999-2002. Primary responsibilities include supervision and oversight of semi-volatile GC department. This includes initiating new methods, staff training, workload management, and instrument maintenance/troubleshooting. Duties include departmental compliance with CAS QA and Safety policies. Responsible for analysis, interpretation and report generation for pesticides and PCB's by EPA Methods 608, 8080, 8081, 8082, EPA 8141A, Organotins, and CLP Pesticides.

Scientist III, Semi-Volatile Organics Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1996-1999. Primary responsibilities were analysis, interpretation and report generation for pesticides and PCB's by EPA Methods 608, 8080, 8081, 8082, and CLP-Pesticides. Secondary responsibilities include organics semi-volatile sample preparation.

Scientist, Volatile Organics Sample Preparation, *Employer's Overload, Longview, Washington – assigned to the Columbia Analytical Services, Inc., Kelso, Washington facility,* 1996. Primary duties included the preparation of water, soil, sediment and tissue samples using EPA Methods 3510, 3520, 3540, 3550, and 3545. Other duties were the further clean up of extracts using EPA Methods 3620 (Florsil), 3610 (Alumina), 3630 (Silica gel), 3650 (Acid/Base Partitioning), and 3660 (Sulfur).

Organics Chemist and GC/MS Chemist, *Coffey Laboratories, Portland, Oregon,* 1990-1996. Primary responsibilities included sample preparation and analysis for EPA FID, ECD, and HPLC using various EPA SW-846 and 500-series methods, as well as other methodology. Later, moved to GC/MS position which included sample preparation, analysis, and associated instrument maintenance for EPA Methods 625, 8027, and 525 BNA's. Also responsible for data review and approval of data packages.

QC Manager/QC Supervisor and Product Manager, Corn Products, *Frito-Lay, Inc., Vancouver, Washington,* 1982-1990. Manager of the QC department overseeing three supervisors and approximately 30 technicians. Responsible for department cost, accuracy, timeliness of data and safety performance. Later, responsible for production oversight of brand name snacks. Responsible for cost, quality and safety performance over three shifts. Managed four supervisors directly and approximately 60 employees indirectly.

Food Technologist, QA Department, *Kraft, Inc., Buena Park, California*, 1978-1981. Responsible for audits, formulations, finished product evaluation, batch reviews and technical support.

Education

MS, Food Science, Minor in Industrial Engineering, *Oregon State Univ. Corvallis, Oregon,* 1978.
BS, Food Science, Minor in Business Administration, *Utah State University, Logan, Utah,* 1975

Publications/ Presentations

Quality Improvement Team Leader, Coffey Laboratories, Portland, Oregon. 1991

Methods Improvement Program, Coffey Laboratories, Portland, Oregon. Seminars on Development and Implementation 1990.

Statistical Process Control and Total Quality Management, Frito-Lay, Vancouver, Washington. Routine Training Classes 1986-1988.

GREGORY G. SALATA

2003 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**PROJECT/EXTRACTIONS MANAGER V** – 2003 to Present
Responsibilities	Responsibilities include Project Management, including quotation preparation and data reporting, as well as providing technical support to the laboratory as needed. Responsibilities also include oversight of the organic extractions lab, managing resources and providing technical support for all organic preparation work flows. 2003-Present.
Experience	**Project Manager,** *B&B Laboratories, College Station, Texas,* 1999-2003. Supervisor/responsible for analysis of TPH (waters, tissues, sediments), organotins (waters, tissues, sediments), Atterberg Limits (sediments), and total organic/inorganic carbon (sediments, waters). Also responsible for report generation on specific projects. Instrumentation operated included GCs with FID and FPD detectors, Combustion TOC, Water TOC, and Dionex Accelerated Solvent Extractor.
	Graduate Student, *Texas A&M University, College Station, Texas,* 1991-1999. While working toward MS in Oceanography, performed organic extractions for pesticides, PCBs, PAHs, and butyltins. While working toward Ph.D. in Oceanography determined stable carbon isotope ratios in sediments, waters, and bacterial phospholipid fatty acids. Other responsibilities included field sample collection, and operation/maintenance of FinniganMAT 252 isotope ratio MS.
	Analytical Chemist, *Science Applications International (SAIC), San Diego, California,* 1989-1990. Performed organic extraction and GC/FID analysis on sediment/rock samples for the Exxon Valdez oil spill.
	GC Chemist, *Analytical Technologies, San Diego, California,* 1987-1989. Responsible for analysis of volatile organics using purge and trap and GC/PID/ELCD.
Education	**Ph.D., Oceanography,** *Texas A&M University, College Station, Texas.* 1999 **MS, Oceanography,** *Texas A&M University, College Station, Texas.* 1993 **BA, Chemistry,** *University of California San Diego, Revelle College, La Jolla, California.* 1987
Publications/ Presentations	*Dr. Salata has a number of publications and published abstracts. For a list of these publications and published abstracts, please contact CAS.*
Affiliations	Society of Environmental Toxicology and Chemistry (SETAC)
	American Chemical Society

JEFFREY A. CORONADO

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position

TECHNICAL MANAGER IV, INORGANICS DEPARTMENT MANAGER – 2001 to Present

Responsibilities

Oversee the operation of the Metals Group. Responsible for the quality and timeliness of the inorganic laboratories analytical reports, departmental budgets, workload coordination, method development efforts, cost-effectiveness, and resource allocation.

Documentation of Demonstration of Capabilities is available for review.

Experience

Metals Department Manager, *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-2001. Responsibilities included management of all aspects of the metal laboratory operation, including personnel training and evaluation, review of all metals data, and report generation. Also responsible for client service on a number of ongoing CAS accounts. Technical duties include primary analytical responsibility for trace level metals analysis by ICP/MS. Analyses range from routine water and soil analysis, to marine tissues, as well as industrial applications such as ultra-trace QA/QC work for various semiconductor clients. Also responsible for a number of specialized sample preparation techniques including trace metals in seawater by reductive precipitation, and arsenic and selenium speciation by ion-exchange chromatography. Developed methodology for performing mercury analysis at low part per trillion levels by cold vapor atomic fluorescence..

Supervisor, GFAA Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1989-1992. Responsibilities included supervision of metals analysis by graphite furnace atomic absorption following SW-846 and EPA CLP methodologies. Duties include workload scheduling, data review, instrument maintenance, personnel training and evaluation.

Education

Field Immunoassay Training Course, *EnSys Inc*., 1995.
Winter Conference on Plasma Spectrochemistry, *San Diego, California,* 1994.
ICP-MS Training Course, *VG-Elemental*, 1992.
BS, Chemistry, *Western Washington University, Bellingham, Washington,* 1988.
BA, Business Administration, *Western Washington University, Bellingham, Washington,* 1985.

LYNDA A. HUCKESTEIN

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**CLIENT SERVICES MANAGER IV** – 1998 to Present
Responsibilities	Management of the Client Services Departments: Project Management, Electronic Data Deliverables and Report Generation, and Sample Management. Personally responsible for approximately 1.5 million dollars of client work annually performing technical project management and client service. Provides technical and regulatory interpretation assistance, as well as project organization of work received by the laboratory.
	Documentation of Demonstration of Capabilities is available for review.
Experience	**Project Chemist,** *Columbia Analytical Service, Inc., Kelso, Washington,* 1992-1998. Primary responsibilities included technical project management and client service in areas of pulp & paper, marine services, mining, and DOD. Also responsible for providing technical and regulatory interpretation assistance as-well-as project organization to work received by the laboratory
	Project Chemist and Department Manager, General Chemistry Laboratory, *Columbia Analytical Services, Inc.,* 1989-1992. Responsible for management of the General Chemistry laboratory for routine wastewater, bioassay, and microbiological analyses. Also responsible for supervision of staff, data review, and reporting.
	Analyst III, *Columbia Analytical Services, Inc., Kelso, Washington*, 1989. Primary responsibilities included coliform testing, total recoverable petroleum hydrocarbon extractions and analysis, BODs, ammonias, and TKN, in addition to miscellaneous wet chemistry analyses.
	Microbiologist/Chemist, *Coffey Laboratories, Portland, Oregon,* 1983. Coliform analysis; water chemistry.
	Laboratory Assistant, *Oregon State University, Corvallis, Oregon,* 1983. Wheat spike dissection and tissue culture.
Education	**BS, Microbiology,** *Oregon State University, Corvallis, Oregon,* 1983.

HARVEY L. JACKY

1999TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position

TECHNICAL MANAGER II – 2008 to Present

Responsibilities

Oversee the operation of the General Chemistry and Microbiology groups. Responsible for the quality and timeliness of the inorganic laboratories analytical reports, departmental budgets, workload coordination, method development efforts, cost-effectiveness, and resource allocation.

Documentation of Demonstration of Capabilities is available for review.

Experience

Project Manager III, *Columbia Analytical Services, Inc., Kelso, WA*, 1999-2008. Responsible for technical project management, ensuring overall data quality and compliance with customer requirements, and providing technical support to clients regarding laboratory application to projects. Additionally, acts as a consultant to clients regarding industrial/environmental compliance issues; serving as liaison between clients and regulatory agencies.

Director of Project Management, *Coffey Laboratories, Portland, Oregon*, 1997-1999. Responsible for technical project management. Communicated with clients to determine needs and expectations. Monitored laboratory production and ensured the timely completion of analytical projects. Technical consultant for clients regarding environmental compliance. Supervised and managed other members of the project management team. Served as a member of the senior management team for oversight of general operations, strategic planning, finances, and policy.

Project Manager/Chemist, *Coffey Laboratories, Portland, Oregon*, 1997-1999. Served as primary liaison between Coffey Laboratories and major clients. Ensured that work was completed in a timely manner and done to client specifications. Served as technical consultant regarding environmental chemistry, soil remediation, and waste water industrial compliance. Clients included the Oregon Department of Transportation, Hazmat Unit, Portland, Oregon; Raythion Demilitarization Co., Umatilla, Oregon; Hydroblast - Wastewater Evaporator Systems, Vancouver, Washington; and Union Pacific Railroad, Northwest Region, Klamath Falls, Oregon.

Technical Sales Representative, *Coffey Laboratories, Portland, Oregon*, 1995-1997. Responsible for marketing and sales, including actively prospecting for new potential clients. Additional responsibilities included procurement and preparation of all major project bids; ensuring that client expectations were met; and maintaining customer satisfaction. Served as consultant regarding industrial compliance issues, environmental remediation projects, and hazardous waste management.

Senior Chemist/Laboratory Chemical Hygiene Officer, *Coffey Laboratories, Portland, Oregon*, 1988-1995. Performed analytical tests including Anions by Ion Chromatography (EPA 300.0), PAHs by HPLC (EPA 8310), Cyanides (EPA 335), and other inorganic, wet chemistry, and organic analytical tests on a wide variety of sample matrices. Responsible for the initial quality assurance review of work performed, supervised and managed personnel. Developed and implemented Laboratory Chemical Hygiene Plan. Directed personnel in regards to safety issues and hazardous waste management. Served as consultant and teacher regarding analytical methodology, environmental compliance, and industrial hygiene.

Education

40-Hour Hazmat Certification, *PBS Environmental,* 1996.
Industrial Emergency Response, *SFSP Seminar,* 1991
BS, Zoology, *Oregon State University, Corvallis, Oregon,* 1988.
BS, General Science, *Oregon State University, Corvallis, Oregon,* 1988.
COURSEWORK, General Studies, *Linfield College, McMinnville, Oregon,* 1981-1982.

Publications/ Presentations

Biochemical and Physical Factors Involved in the Application and Measurement of a Soil Bioremediation System. Biogeochemistry, Portland State University, 1996

Affiliations

American Chemical Society, Member since 1988

JEFFERY A. GRINDSTAFF

1991 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position

TECHNICAL MANAGER III, PHARMACEUTICAL, GC/MS VOA AND SEMI-VOA LABORATORIES, – 1997 to Present

Responsibilities

Primary responsibilities include leadership of the Pharmaceutical, GC/MS VOA and Semi-VOA staff, management of method development, training, data review, tracking department workload, scheduling analyses. Responsible for ensuring data quality and timeliness. Also responsible for project management and coordination for pharmaceutical clients.

Documentation of Demonstration of Capabilities is available for review.

Experience

Manager, GC/MS VOA Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1994-1997. Responsible for supervision of GC/MS VOA staff, method development, training, data review, tracking department workload, scheduling analyses, and general maintenance and troubleshooting of GC/MS systems.

Scientist III, GC/MS VOA Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1991-1994. Responsibilities included scheduling workload, data review, instrument maintenance and troubleshooting, and personnel training and evaluation. Also responsible for supervision of extraction personnel and instrument analysts. Additional supervisory duties included report generation and data review for GC analyses. Responsibilities also included project management and customer service.

Chemist, *Enseco-CRL, Ventura, California,* 1990-1991. Established GC/MS department including inventory maintenance, preparation of state certification data packages, method development, SOPs, and extended data programs. Performed daily maintenance and troubleshooting of GC and GC/MS instrumentation. Scheduled and performed routine and non-routine VOA analyses.

GC/MS Chemist, VOA Laboratory *Coast-to-Coast Analytical Service, San Luis Obispo, California,* 1990-1991. Responsible for standard preparation for VOA analyses, instrument calibration, tuning, and maintenance. Also implemented and further developed EPA methods for quantitative analysis of pesticides and priority pollutants.

Education

Sampling and Testing of Raw Materials, *PTI International, 2004*.
Leadership Training, *Richard Rogers Group, 1996*
Mass Selective Detector Maintenance, *Hewlett Packard Education Center, 1993*
Interpretation of Mass Spectra I, *Hewlett-Packard Analytical Education Center,* 1992.
B.S., Chemistry, *California Polytechnic State University, San Luis Obispo, California,* 1989.
A.A., Liberal Arts, *Allan Hancock College, Santa Maria, California.* 1986

Publications/ Presentations

Low Level Analysis of 1,4-Dioxane by GC/MS SIM using Large Volume Injection, with J. Peterson and R. Holden. SETAC National Meeting Poster Session, Portland, OR 2004.

Low Level Determination of N-nitrosodimethylamine by Chemical Ionization GC/MS with Large Volume Injection, with C. Degner and J. Peterson. SETAC National Meeting Poster Session, Portland, OR 2004.

Analysis of Polybrominated Diphenyl Ethers by GC/MS with Large Volume Injection, J. Peterson and M.Thompson SETAC National Meeting Poster Session, Portland, Oregon, 2004.

Alternate Method to Lower Detection Limits to Satisfy Regulatory Action Levels for Volatiles in Groundwater, with David Edelman, Kairas Parvez, and Paul Laymon. TAPPI National Meeting, Orlando, FL 1996

Affiliations

American Chemical Society. 1989

NICOLAS BLOOM

2008 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**Scientist VII** – 2008 to Present
Responsibilities	**Senior Research Scientist**
	Mr. Bloom has been involved in research on the biogeochemistry of trace metals in the environment for 30 years. After graduating from the University of Washington in 1979, he entered the graduate program in the Civil Engineering Department, where he worked as a full time researcher, investigating the sorption behavior of ultra-trace concentrations of cations and anions on ferric hydroxide suspensions. In 1980, Mr. Bloom was hired by the Battelle Marine Research Laboratory to develop sampling and analytical techniques to quantify a wide range of trace metals in sea water at ambient levels and apply those methods to the biogeochemical cycling of Hg, As, Ag, Pb, Cd, and Cu in Puget Sound. In 1984 Mr. Bloom returned to graduate school at the University of Connecticut, where he developed analytical techniques to allow the speciation of Hg at the sub-picogram level by GC-CVAFS. These methods have since been applied to investigate the cycling of Hg and its various compounds in lacustrine and marine systems throughout the world.
	In 1991, Mr. Bloom founded Frontier Geosciences Inc., where he continued research into ultra-low level metals speciation in sediments, air, and fossil fuels, as well as mentored the development of IC-ICP/MS and IC-HG-AFS methods for most other trace metals and for Se, As, and Cr speciation. From 2001-2005, Mr. Bloom collaborated extensively with the Universita Ca'Foscari di Venezia in a study of Hg speciation and dynamics in the Venice Lagoon. In 2004, Mr. Bloom founded Studio Geochimica LLC, continuing his studies of the biogeochemistry of trace metals in the environment and industry. In 2008, Mr. Bloom joined Columbia Analytical Services, as Director of the Trace Metals Research and Development Department. In this position, Mr. Bloom is responsible for the development and validation of new trace metals speciation methodologies as well as working with clients and staff having biogeochemical questions or particularly perplexing analytical issues.
Experience	**Research Scientist,** *Battelle Pacific Northwest Laboratory, Marine Sciences Lab, Sequim, WA,* 1980-1989. As an analyst, developed and validated ultra-clean sampling methods and techniques for the analysis of all 13 EPA priority trace metals in water, sediment, and tissues with detection limits below the ambient background concentrations. As a researcher, emphasized biogeochemical processes of trace metals, particularly at the air/sea and sediment/water interfaces. Supervised two technicians.
	Owner/Manager/Sr. Scientist, *Studio Geochimica LLC, Seattle, WA*, 2004 - 2008. Set up the scientific agenda, marketing, sales, inventing new analytical methods, mentoring, working in the lab as scientist and analyst, etc. Staff varied from 4 to 9 people.
	Owner/Manager/Sr. Scientist, *Frontier Geosciences Inc., Seattle, WA*, 1991 – 2004. Set up the scientific agenda, marketing, sales, inventing new analytical methods, mentoring, working in the lab as scientist and analyst, etc. Staff varied from 3 in 1991 to 87 people in 2003.
Education	**BS, Chemistry**, *University of Washington, Seattle, WA*, 1979.
	MS, Chemical Oceanography, *University of Connecticut, Storrs, CT* 1986.
Publications/ Presentations	*Nicolas Bloom Mr. Bloom has approximately 120 publications on the biogeochemistry and analysis of trace metals in the environment (please inquire for publication list or copies of key papers), and has over 400 presentations at conferences and symposia world-wide.*
Affiliations	ASTM, ACS (past member), ASLO (past member)

LOREN E. PORTWOOD

1992 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position

Technical Manager I, DRINKING WATER LABORATORY – 2008 to Present

Responsibilities

Responsible for the overall operation and supervision of the Organic Drinking Water department. Also responsible for implementation and oversight of UCMR2 analyses. Perform method development. Project management of drinking water accounts. Development of Standard Operating Procedures for Drinking Water methods. Operation of Varian GC/MS, Agilent GC/ECD and Agilent HPLC.

Documentation of Demonstration of Capabilities is available for review.

Experience

Scientist IV, Drinking Water Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 2002-2008. Plan, conduct, and, as lead analyst, supervise analyses using advanced instrumentation such as HPLC with post column derivatization, GC/MS, and GC/ECD. Responsible for data interpretation, quality control and data reporting. Additional responsibilities include preparation of SOPs and specifications for processes and tests; handling routine and advanced maintenance and troubleshooting of instrumentation; and assisting in the training of staff department analysts. Assists the department manager and/or other senior scientists in setting up more complex procedures. Serves as senior technical advisor for teams and projects.

Technical Manager I, Petroleum Hydrocarbon Laboratory Supervisor, *Columbia Analytical Services, Inc., Kelso, Washington,* 1998-2002. Primary responsibilities include organizing and prioritizing the workload for the petroleum hydrocarbon team, initiating new methods and process improvements, and staff development and training. Other duties include department wide compliance with CAS quality assurance guidelines, routine system checks, assist and encourage staff in troubleshooting equipment and procedural problems, and lead by example in a manner that is consistent with company, state and federal guidelines. Also responsible for duties listed below under Scientist II and Scientist III.

Scientist III, Petroleum Hydrocarbon Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1997-1998. Duties primarily as listed below.

Scientist II, **Petroleum Hydrocarbon Laboratory,** Columbia Analytical Services, Inc., Kelso, Washington, 1996-1997. Primary responsibilities included analysis, reporting, and archiving of water, soil, and product samples for semi-volatile petroleum hydrocarbons and miscellaneous FID tests. Methods of analysis include EPA methods 8100, 8310, 8315, 8330, 8040, 8015 and various state modifications of 8015 (OR, WA, CA, AK). Additional analyses include solvent scans, alcohols, glycols, and EPA methods 413.2 and 418.1. Other responsibilities include sample preparation and instrument maintenance.

Scientist I, Petroleum Hydrocarbon Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1993-1996. Primary responsibilities included the analysis, reporting, and archiving of water, soil, and product samples for semi-volatile petroleum hydrocarbons. Methods of analysis include EPA method 8015 and various state modifications thereof (OR, WA, CA, AK). Additional responsibilities include sample preparation, instrument maintenance, and assistance with other departmental analyses, including EPA methods 413.2 & 418.1.

Bench Chemist I, Organic Extractions Laboratory, Columbia Analytical Services, Inc., Kelso, Washington, 1992-1993. Primary responsibilities included the performance of a full range of semi-volatile sample preparations for water, soil, and oil to be analyzed in the GC, GC/MS, and Petroleum Hydrocarbon Laboratories. These extraction methods included hazardous waste, wastewater, and drinking water procedures. Other responsibilities included extract cleanup via Florisil®, GPC, and Hg.

Chemist, Treclen Laboratories, Spokane, Washington, 1990-1992. Primary responsibilities included inorganic water and soil testing by EPA methods. As Chemist, I developed the testing which was accredited by the EPA, which included everything from metal digestions, to phosphates, to TSS and TDS.

Education

Comprehensive HPLC Training, *Restek,* 2002.
Purge & Trap Theory and Troubleshooting, *Full Spectrum Analytics, Inc.,* 2001.
HP5890 GC Advanced Operations, *Hewlett Packard,* 1996.
HP6890 Fast GC, *Hewlett Packard,* 1996.
Quality Training, *Roger Tunks,* 1996.
Capillary Chromatography Training, *Restek,* 1993.
HP5890 GC Maintenance and Troubleshooting, *Hewlett Packard,* 1993.
BS, Chemistry, Emphasis in Biochemistry, *Whitworth College, Spokane, Washington*, 1990.

EILEEN M. ARNOLD

1987 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**SCIENTIST IV, METALS LABORATORY, KELSO HEALTH AND SAFTEY OFFICER –** 1994 to Present
Responsibilities	Duties include the operation and maintenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits.
	Documentation of Demonstration of Capabilities is available for review.
Experience	**Project Chemist, Client Serv ices Group, Kelso Health and Safety Officer,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-1994. Duties included technical project management and customer service. Responsible for meeting the clients' needs of timely and appropriate analyses, and to act as liaison for all client-related activities wi thin Columbia Analytical Services, Inc. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, ma intenance of all safety related equipment and documents, and performance of monthly safety audits.
	Scientist IV, Metals Laboratory, Health and Safety Officer, *Columbia Analytical Services, Inc., Kelso, Washington, 1987-1992.* Duties include the operation and main tenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. Health and Safety Officer responsibilities included development and implementati on of the Kelso Health and Safety program, including accident investigation and incident review , maintenance of all safety related equipment and documents, and performance of monthly safety audits.
	Chemist, *Dow Corning Corporation, Springfield, Oregon,* 1986-1987. Responsibilities included ICP and atomic absorption work in silicon manufacturing. Methods development for ICP analysis of minor impurities found in silicon.
	Chemist, *Ametek, Inc., Harleysville, Pennsylvania,* 1982-1985. Responsibilities included product research and development chemist involved in production of thin-film semiconductors for use as solar cells. Work involved AA and SEM techniques.
	Chemist, *Janbridge, Inc., Philadelphia, Pennsylvania*, 1978-1982. Responsibilities included maintaining electroplating process lines through wet chemical analysis techniques, and performed Quality Assurance testing on printed circuit boards.
Education	**BA, Chemistry,** *Immaculata College, Immaculata, Pennsylvania,* 1977.
Affiliations	American Chemical Society, Member since 1987.

APPENDIX C

MAJOR ANALYTICAL EQUIPMENT

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balances (10):			
Precisa and Mettler models	1988-2008	MM	15
Autoclave - Market Forge Sterilmatic	1988	LM	5
Autotitrator – Thermo Orion 500	2007	LM	3
Calorimeters (2):			
Parr 1241 EA Adiabatic	1987	LM	4
Parr 6300 Isoparabolic	2005	LM	4
Centrifuge - Damon/IEC Model K	1992	LM	15
Colony Counter - Quebec Darkfield	1988	LM	4
Conductivity Meters (2):			
YSI Model 3200	2004	LM	4
VWR	2001	LM	4
Digestion Systems (5):			
COD (4)	1987, 1989	LM	5
Kjeldahl, Lachat 46-place (1)	1999	LM	3
Dissolved Oxygen Meter - YSI Model 58 (3)	1987, 1988, 1991	LM	5
Distillation apparatus (Midi) - Easy Still (2)	1996, 2000	LM	7
Drying Ovens (11):			
Shel-Lab and VWR models	1988 - 2003	LM	15
Flash Point Testers (2):			
ERDCO Setaflash Tester	1991	LM	4
Petroleum Systems Services	2005	LM	4
Flow-Injection Analyzers (2):			
Bran-Leubbe	2002	LM	4
Lachat 8500	2007	LM	4
Ion Chromatographs (4)			
Dionex 2000i with Peaknet Data Systems	1988	LM	3
Dionex DX-120 with Peaknet Data System	1998	LM	3
Dionex ICS-2500 with Chromchem Data System	2002	LM	3
Dionex ICS-2000 with Chromchem Data System	2006	LM	3
Ion Selective Electrode Meters (5)			
Fisher Scientific Accument Model 50	1997	LM	6
Fisher Scientific Accument Model 25	1993	LM	6
Fisher Scientific Accument Model 20	2000	LM	6
Orion Model 920A	1990	LM	6
Corning pH/ion Meter Model 135	1992	LM	6
Microscope - Olympus	1988	LM	1
Muffle Furnace- Sybron Thermolyne Model F-A1730	1991	LM	15
pH Meters (2):			
Fisher Scientific Accument Model 20	1993	LM	6
Fisher Scientific Accument Model AR25	2005	LM	6

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY (continued)			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Shatter Box - GP 1000	1989	LM	5
Sieve Shakers (2):			
CE Tyler - Portable RX 24	1990	LM	5
WS Tyler - RX 86	1991	LM	5
Thomas-Wiley Laboratory Mill, Model 4	1989	LM	7
Total Organic Carbon (TOC) Analyzers (2)			
Coulemetrics Model 5012	1997	LM	3
O-I Corporation Model 1010	2002	LM	3
Total Organic Halogen (TOX) Analyzers (3):			
Mitsubishi TOX-Sigma	1995	LM	4
Mitsubishi TOX-100 (2)	2001	LM	4
Turbidimeter - Hach Model 2100N	1996	LM	8
UV-Visible Spectrophotometers (3):			
Hitachi 100-40 Single Beam	1986	LM	5
Beckman-Coulter DU520	2005	LM	5
Perkin Elmer Lambda 25	2008	LM	5
Vacuum Pumps (2):			
Welch Duo-Seal Model 1376	1990	LM	13
Busch R-5 Series Single Stage	1991	LM	13
Water Baths/Incubators (6):			
Hach Model 15320 Incubator	1986	LM	15
Precision Model L-6 (2)	1989, 1990	LM	15
VWR 1540	1991	LM	15
Fisher 11-680-626M Incubator	1992	LM	15
Fisher Isotemp Incubator	2001	LM	15

METALS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance (6)			
Mettler AE 200 analytical balance	1990	MM	12
Various Mettler, Sartorius, and Ohaus models (5)	1988	MM	12
Atomic Absorption Spectrophotometers (5):			
Varian SpectrAA Zeeman/220 AA w/Data Systems (2)	2000	LM	3
CETAC Mercury Analyzer	2000	LM	2
Perkin Elmer AAnalyst 200 Flame AA	2005	MM	2
Atomic Fluorescence Spectrophotometer			
Brooks-Rand Model III (2)	1996, 2005	LM	3
Leeman Mercury Analyzer (1)	2006	LM	2
Centrifuge - IEC Model Clinical Centrifuge	1990	LM	12
Drying Oven - VWR Model 1370F	1990	LM	12
Freeze Dryers (2) - Labconco	1992, 2006	LM	5
Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES) (3)			
Thermo Jarrell Ash Model 61E	1988	LM	4
Thermo Jarrell Ash, Model IRIS	2000	MM	4
Thermo Scientific Model iCAP 6500	2007	MM	3
Inductively Coupled Plasma Mass Spectrometers (ICP-MS):			
VG Excell	2001	MM	3
Thermo X-Series	2006	MM	2
Muffle Furnace - Thermolyne Furnatrol Model 53600 (2)	1991, 2005	LM	5
Shaker - Burrell Wrist Action Model 75	1990	LM	12
TCLP Extractors (3)	1989, 2002	LM	5

SEMIVOLATILE ORGANICS SAMPLE PREPARATION LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance (4)			
Mettler PM480, AE166, BB300	1999 - 2005	MM	18
OHaus EP613	2006	MM	18
Centrifuge - Sorvall Model GLC-1	1988	LM	18
Drying Ovens (2)			
Fisher Model 655G	1991	LM	18
VWR Model 1305U	1999	LM	18
Evaporators (14):			
Organomation N-Evap (7)	1989-98, 2001, 2006	LM	18
Organomation S-Evap (7)	1989-1991, 2006	LM	18
Extractor Heaters: Lab-Line Multi-Unit Models for Continuous Liquid-Liquid and Soxhlet Extractions (102)	1987-1992, 2007	LM	12
Extractors (52):			
Branson Model 450 Sonifier (2)	1991	LM	6
Tekmar Sonicator	1994	LM	6
Fisher Scientific Sonicator	1994	LM	6
Soxhtherm (48)	2000, 2008	LM	8
Extractors, TCLP (10):			
Millipore TCLP Zero Headspace Extractors (10)	1987-1992	LM	2
TCLP Extractor - Tumbler (12 position)	1989	LM	2
Gel Permeation Chromatography (GPC) (5)			
ABC single column (3)	1998, 1999, 2007	LM	4
ABC Autoprep 1000	1995	LM	4
J2 Scientific	2005	LM	4
Muffle Furnace - 4	1994-2006	LM	4
Solid Phase Extractors (8) – Horizon SPE-Dex 4790	2003, 2006	LM	4
Ultrasonic Water Bath – VWR 550D	2007	LM	18
Vacuum Pump – Edwards	1992	LM	8

GC SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler AT 250	1989	MM	7
Chromatography Data Systems (12)			7
HP Enviroquant (8)	1994-2002	LM	
Thruput Target (4)	1998-2000	LM	
Gas Chromatographs (11):			
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual ECD Detectors (4)	1990 – 1995	LM	7
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual FPD Detectors	1991	LM	7
Agilent 6890 GC with Agilent 7683 Autosampler and Dual ECD Detectors (5)	2001, 2005, 2007	LM	7
Agilent 6890 GC with Agilent 7683 Autosampler and Dual FPD Detectors	2003	LM	7
Agilent 7890A Dual ECD Detectors Agilent 7683B autosampler	2008	LM	7

GC/MS SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Accelerated Solvent Extractor - Dionex ASE 200	1996	LM	5
HP Enviroquant Chromatography Data Systems (9)	1994-2002	LM	5
Gas Chromatograph: Hewlett-Packard 5890 with HP 7673 autosampler and FID Detector	1994	LM	5
Semivolatile GC/MS Systems (9):			
Agilent 6890/5973 with ATAS Optic2 LVI and HP 7673 Autosampler (2)	1997, 2001	LM	5
Agilent 5890/5970 and HP 7673 Autosampler	1990	LM	5
Agilent 5890/5970 with ATAS Optic2 LVI and HP 7673 Autosampler	1994	LM	5
Agilent 5890/5972 with ATAS Optic2 LVI and HP 7673 Autosampler (3)	1993, 1994, 1998	LM	5
Agilent 6890/5973 with ATAS Optic3 LVI and 7683 Autosampler	2004	LM	
Agilent 6890/5973 with Agilent PTV Injector and 7683 Autosampler	2007	LM	4
Semivolatile GC/MS/MS – Waters Quattro Micro GC Micromass with Agilent 6890, Agilent PTV Injector, 7683B Autosampler	2008	MM	1

PETROLEUM HYDROCARBONS GC/HPLC LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler BB240	1994	MM	6
Aspirator pump – GAST	2004	LM	6
Drying Oven - Fisher Model 630F	1991	LM	6
Evaporator - Organomation N-Evap	1990	LM	6
HP Enviroquant Chromatography Data Systems (8)	1994-2002	LM	6
Gas Chromatographs (6):			
Hewlett-Packard 5890 Series II with PID/PID/FID(2)	1991	LM	4
EST-ENCON Purge and Trap Concentrator	1991	LM	4
Dynatech Archon 5100 Autosampler	1992	LM	4
Hewlett-Packard 5890 GC with HP 7673 Autosampler and FID Detector	1995	LM	4
Agilent 6890 with Dual FID Detectors and Agilent 7873 Autosampler (3)	2001, 2005	LM	4
High-Performance Liquid Chromatographs (2):			
HP 1090M Series II with Diode Array UV Detector	1999	LM	4
HP 1050/1100 Series with Fluorescence & Diode Array UV Detectors	2004	LM	4
High-Performance Liquid Chromatograph/Mass(2)			
Spectrometer - Thermo Electron TSQ Quantum LC/MS/MS and Autosampler	2005	MM	2
API 5000 LC/MS/MS and SIL-20AC Autosampler	2008	MM	2

VOLATILE ORGANICS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler PE 160	1989	MM	5
Fisher Vortex Mixer	1989	LM	5
HP Enviroquant Chromatography Data Systems (10)	1994-2002	LM	5
Drying Ovens (2):			
Narco 420	1989	LM	5
VWR 1305 U	1991	LM	5
Sonic Water Bath - Branson Model 2200	1989	LM	5
Volatile GC/MS Systems (7):			
Agilent 5890/5970	1989	LM	5
Tekmar 3000 Purge and Trap Concentrator	1995	LM	5
Dynatech ARCHON 5100 Autosampler	1996	LM	5
Agilent 5890/5971	1991	LM	5
Tekmar 3000 Purge and Trap Concentrator	2001	LM	5
Dynatech ARCHON 5100 Autosampler	1995	LM	5
Agilent 5890/5972A	1993	LM	5
Tekmar 3000 Purge and Trap Concentrator	1995	LM	5
Dynatech ARCHON 5100 Autosampler	1996	LM	5
Agilent 6890/5973	2001	LM	5
Tekmar 3100 Purge and Trap Concentrator	2001	LM	5
Varian Archon Autosampler	2001	LM	5
Agilent 6890/5973	2005	LM	5
Tekmar Velocity Purge and Trap Concentrator	2005	LM	5
Tekmar Aquatech Autosampler	2005	LM	5
Agilent 6890/5973 (2)	2007	LM	5
Tekmar 3000 Purge and Trap Concentrator	2007	LM	5
Varian Archon 5100 Autosampler	2007	LM	5

DRINKING WATER ORGANICS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler BB300	1991	MM	2
Extractors (10) – Horizon SPE-DEX Solid Phase Extractor	2003/2008	LM	2
Aglinet Enviroquant Chromatography Data Systems (2)	2003	LM	2
Varian Saturn Chromatography Data System	2003	LM	2
Evaporator - Organomation N-Evap	2003	LM	2
Agilent 1100 HPLC w/post-column derivitization: UV/Fluoescence detectors Pickering PCX-5200 Post-column derivitization unit	2003 2003 2003	LM LM LM	2 2 2
Agilent 6890N GC/Dual ECD system w/ autosamplers Agilent 7890 GC/Dual ECD w/autosamplers	2003 2008	LM LM	2 2
Varian Ion trap GC/MS: Varian 3800 GC w/CP8400 autosampler Varian Saturn 2100T mass spectrometer	2003 2006 2003	LM LM LM	2 2 2
Thremo Ion Trap GC/MS w/TriPlus autosampler	2008	LM	2

Metals Method Development Laboratory			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Perkin-Elmer ICP/MS Elan 9000 w/ Perkin-Elmer AS-93+ Autosampler	2008	LM	2
Perkin-Elmer Series 200 IC	2008	LM	2
Brooks Rand III Atomic Fluoresence Spectrophotometer - 2	2008	LM	2
Oriel Atomic Fluoresence Spectrophotometer – Lab Designed	2008	LM	2
Balances - 4	2008	LM	2
Ovens - 2	2008	LM	2
Buck AA Spectrophotometer Model 205	2008	LM	2
Forma Scientific Bio Freezer	2008	LM	2
Digital Shaker SK-71	2008	LM	2

AUTOMATED DATA PROCESSING EQUIPMENT			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
1-WAN: LIMS Sample Manager using Oracle 10g DBMS running on Redhat Advanced Server 3.0 (Linux) platform connected/linked on a frame relay WAN environment	1994-2004	LM	NA
1 - Network Server Pentium 4 class, 1 for Reporting and Data Acquisition running Windows 2003 Advanced Server, 1 for Applications running Windows 2003 Advanced Server. Data acquisition capacity at 65GB with redundant tape and disk arrays.	2004	LM	NA
Approximately 50+ HP and Dell Laserjet printers (various types including models III, 4, 5, 8150, 4000, 4050, 4250, 8150, 1720dn, W5300)	1991 - 2007	LM	NA
Approximately 180 Gateway/Dell PC/Workstations running Windows 2000/XP on LAN connected via 10BT/100BT and TCP/IP for LIMs Terminal Emulation	1993 - 2004	LM	NA
Microsoft Office 2003 Professional as the base application for all PC/Workstations. Some systems using Office 2000/97.	1996 - 2004	LM	NA
E-Mail with link to SMTP for internal/external messaging. Web mail via Outlook Web Access interface. Microsoft Outlook 2003.	1994 - 2006	LM	NA
Standard Excel (R) reporting platform application linked to LAN/WAN for data connectivity and EDD generation.	1996 - 2004	LM	NA
Standard Excel (R) reporting platform application linked to LAN/WAN for data connectivity and EDD generation.	1996 - 2004	LM	NA
Facsimile Machines - Brother 4750e (2); Brother SuperG3 (1); Canon CFX-L4000 (1)	1991 - 2007	LM	NA
Copiers/Scanners: Konica BizHub 420 (1), BizHub 600 (1), BizHub 920 (2), BizHub Pro 1050 (3). The 920s and 1050s are accessible via LAN for network scanning.	2000 - 2007	LM	NA
Dot Matrix Epson FX-880, LQ-1050, LX-300	1991 - 2004	LM	NA
Thruput, MARRS, Stealth, Harold, Blackbird, EDDGE, StarLIMS reporting software systems.	1998 - 2004	LM	NA

NA: Not applicable. This equipment administered by IT staff but may be used by all staff.

APPENDIX D

PREVENTIVE MAINTENANCE PROCEDURES

Instrument	Activity	Frequency
Refrigerators and Coolers	Record temperatures Clean coils Check coolant	Daily Annually Annually or if temperature outside limits
Vacuum Pumps	Clean and change pump oil	Every month or as needed
Fume Hoods	Face velocity measured Sash operation Change filters Inspect fan belts	Quarterly As needed Annually Annually
Ovens	Clean Record temperatures	As needed or if temperature outside lim. Daily, when in use
Incubators	Record temperatures	Daily, morning and evening
Water Baths	Record temperatures Wash with disinfectant solution	Daily, morning and evening When water is murky, dirty, or growth appears
Autoclave	Check sterility Check temperature Clean	Every month Every month When mold or growth appears
Analytical Balances	Check alignment Check calibration Clean pans and compartment	Before every use Daily After every use
Dissolved Oxygen Meter	Change membrane	When fluctuations occur
pH probes	Condition probe	When fluctuations occur
Fluoride ISE	Store in storage solution	Between uses
Ammonia ISE	Store in storage solution	Between uses
UV-visible Spectrophotometer	Wavelength check	Annually
Total Organic Carbon Analyzers	Check IR zero Check digestion/condensation vessels Clean digestion chamber Clean permeation tube Clean six-port valves Clean sample pump Clean carbon scrubber Clean IR cell	Weekly Each use Every 2000 hours, or as needed Every 2000 hours, or as needed Every 200 - 2000 hours, or as needed Every 200 - 2000 hours, or as needed Every 200 - 2000 hours, or as needed Every 2000 - 4000 hours, or as needed

Instrument	Activity	Frequency
Total Organic Halogen Analyzers	Change cell electrolyte	Daily
	Change electrode fluids	Daily
	Change pyrolysis tube	As needed
	Change inlet and outlet tubes	As needed
	Change electrodes	As needed
Flow Injection Analyzer	Check valve flares	Each use
	Check valve ports	Each use
	Check pump tubing	Each use
	Check light counts	Each use
	Check flow cell flares	Quarterly
	Change bulb	As needed
	Check manifold tubing	Each use
	Check T's and connectors	Each use
Ion Chromatographs	Change column	Every six months or as needed
	Change valve port face & hex nut	Every six months or as needed
	Clean valve slider	Every six months or as needed
	Change tubing	Annually or as needed
	Eluent pump	Annually
Atomic Absorption Spectro-photometers - FAA and CVAA	Check gases	Daily
	Clean burner head	Daily
	Check aspiration tubing	Daily
	Clean optics	Every three months
	Empty waste container	Weekly
Atomic Absorption Spectro-photometers - GFAA	Check gases	Daily
	Check argon dewar	Daily
	Change graphite tube	Daily, as needed
	Clean furnace windows	Monthly
ICP - AES	Check argon dewar	Daily
	Replace peristaltic pump tubing	Daily
	Empty waste container	Weekly
	Clean nebulizer, spray chamber, and torch	Every two weeks
	Replace water filter	Quarterly
	Replace vacuum air filters	Monthly

Instrument	Activity	Frequency

Instrument	Activity	Frequency
ICP - MS	Check argon dewar	Daily
	Check water level in chiller	Daily
	Complete instrument log	Daily
	Replace peristaltic pump tubing	Daily
	Clean sample and skimmer cones	As needed
	Clean RF contact strip	As needed
	Inspect nebulizer, spray chamber, and torch	Clean as needed
	Clean lens stack/extraction lens	As needed
	Check rotary pump oil	Monthly
	Change rotary pump oil	Every six months
Gel-Permeation Chromatographs	Clean and repack column	As needed
	Backflush valves	As needed
High Pressure Liquid Chromatographs	Backflush guard column	As needed
	Backflush column	As needed
	Change guard column	As needed when back pressure too high
	Change column	Annually or as needed
	Change in-line filters	As needed
	Leak check	After column maintenance
	Change pump seals	As needed
	Change pump diaphragm	Annually
	Clean flow cell	As needed
	Fluorescence detector check	Daily
	Diode array absorbance check	Daily
Gas Chromatographs, Semivolatiles	Check gas supplies	Daily, replace if pressure reaches 50psi
	Change in-line filters	Quarterly or after 30 tanks of gas
	Change septum	Daily
	Change injection port liner	Weekly or as needed
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Check system for gas leaks	After changing columns and after any power failure
	Clean FID	Weekly or as needed
	Clean ECD	Quarterly or as needed
	Leak test ECD	Annually

Instrument	Activity	Frequency
Gas Chromatograph/Mass Spectrometers, Semivolatiles	Check gas supplies	Daily, replace if pressure reaches 50psi
	Change in-line filters	Annually or as needed
	Change septum	Daily, when in use
	Change injection port liner	Weekly or as needed
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Clean source	As needed when tuning problems
	Change pump oil	As specified by service specifications
Purge and Trap Concentrators	Change trap	Every four months or as needed
	Change transfer lines	Every six months or as needed
	Clean purge vessel	Daily
Gas Chromatographs, Volatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Quarterly or after 30 tanks of gas
	Change septum	Daily
	Clip first 6-12" of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Check system for gas leaks	After changing columns and after any power failure
	Clean PID lamp	As needed
	Clean FID	As needed
	Change ion exchange resin	Every 60 days
	Replace nickel tubing	Quarterly or as needed
Gas Chromatograph/Mass Spectrometers, Volatiles	Check gas supplies	Daily, replace when pressure reaches 50 psi
	Change in-line filters	Annually or as needed
	Change septum	Daily
	Clip first foot of capillary column	As needed
	Change guard column	As needed
	Replace analytical column	As needed when peak resolution fails
	Clean jet separator	As needed
	Clean source	As needed when tuning problems
	Change pump oil	As specified by service specifications

APPENDIX E

CORPORATE POLICY STATEMENTS

Policy for Data Review and Validation

May 2009
Effective July 1, 2009

The purpose of this policy is to identify the requirements for performing data review and validation prior to releasing data and reports to customers of Columbia Analytical Services. It is a requirement of NELAC (TNI) quality system standards and Department of Defense (DoD) agencies to have data review procedures established.

This policy is applicable to the review of raw and reported data generated in all laboratories. Specific data review and validation processes or logistics may vary somewhat from facility to facility, or vary for data generated using different methodologies however; the policies described here are to be followed. The documentation practices should be consistent within the facility. Automated validation processes are encouraged, but must be sufficiently described in an SOP.

In general, the data review and validation practices used at each facility will meet the requirements of NELAP quality system standards, the DoD Quality System Manual (QSM), and ISO 17025. Specific data review and validation policies are as follows:

1. Each laboratory facility will have a written and approved standard operating procedure (SOP) for conducting data review/validation that meets the standard CAS requirements for administrative SOPs. The SOP will list details of data review practices for the facility. The SOP will also give a detailed explanation of the review documentation procedures for each type of data.

2. Data review will be performed by qualified personnel who have documented training on either the analysis itself or training specific to the data review SOP. Personnel preparing reports who may do some level of clerical review or proofreading do not need technical knowledge of the test, but must be knowledgeable of reporting systems and requirements.

3. All data will be reviewed by a minimum of two persons. Data generated or reported by one person may not be released without another person's review.

4. However defined, one review (typically a "primary" technical review) must focus on the validity of the analysis and raw data generated, the technical accuracy and correctness of the analysis (the analytical procedure is in control), use of valid and approved procedures and methods, and interpretation of sample results.

5. The secondary review will be performed by someone other than the technical reviewer. The secondary review will make the same assessments as the primary reviewer, and check the interpretations, data manipulations, and decisions made by the primary reviewer. Additionally, the secondary reviewer will review the outputs from the initial review to the raw data. This includes such things as data processing results/outputs, calculations, runlogs, bench sheets, QC analyses, etc. The secondary review verifies the completeness and validity of the data to be reported.

6. All client-ready final reports will be reviewed in the format, and as presented to, the client; either by analysis fraction or in their entirety. This review will include verification of the accurate and correct reporting of sample and QC results; including accurate translation of results from data to report forms, report format, use of qualifiers and flags, and method citations. This review will also include verification of the correct project information; such as client name, project name, sample I.D.s, etc. The report review should ensure that the report is error-free and contains no inconsistencies. For upper tier deliverables, this review will verify that all deliverables are included in the report package.

7. The Project Manager will review all complete reports prior to signing the report and submitting to the client. The review of the reported data will focus on the following items:
    a. Consistency with client, contract, and/or project specifications.
    b. Acceptability of any data qualifiers or footnotes.
    c. Accuracy and completeness of explanations or discussion in the report cover letter or case narrative.
    d. As needed depending on the scope of testing, an additional level of technical review of all data generated.
    e. A general overview of the completed service request file with respect to overall reasonableness, and if available, with historical project information.

8. Data review must be documented. Persons performing data and report review must sign (or initial) and date the applicable data reviewed. Checklists or review summaries should be used for guidance and documentation. Documentation processes must be described in the laboratory SOP.

Lee Wolf, Corporate Director of Quality Assurance — Date: 5-5-09

Steve Vincent, President — Date: 5-5-09

Policy for Conducting Research, Method Development, and Method Investigations

December 2009

Columbia Analytical Services (CAS) often develops test procedures internally by conducting research and development or method development based on published procedures. This type of testing may not fall under common laboratory regulations which describe benchmarks, or minimum requirements, for procedure development and implementation. Also, it may be necessary at certain times to conduct investigations into the quality of existing methods. Therefore, a policy is necessary to identify and establish those minimum requirements.

The purpose of this Policy is to identify the CAS requirements for performing internal research and subsequent method development, performing method development from published references, and performing investigations into method performance.

For the purpose of this policy, the following Definitions are provided:

Research and development (R&D) – The practice of independently evaluating analytical options and procedures and applying them to a sample analysis challenge; resulting in an internally developed analysis method. For this policy, R&D is limited to that performed by CAS personnel.

Method development – The practice of implementing a CAS analysis procedure based on published references.

Method investigation – For the purpose of this policy, this is defined as the evaluation of major changes in methodology outside the scope of published methods or SOPs. This is generally done to improve method performance or troubleshoot a significant analytical problem; and done outside of the routine maintenance, troubleshooting, and nonconformance/corrective action process.

The intent of this policy is to ensure that CAS R&D, method development, and method investigations are performed in an unbiased manner, ensure data integrity, use common scientific practices; and ensure that these activities are peer reviewed.

General Provisions

- When conducting any of the activities covered by this policy, employees will follow standard CAS procedures for maintaining documentation and analysis records.
- Initial and final review of statements, plans, and summaries will be done by two persons; the applicable Technical Director (TD) and the Laboratory Director (LD). If the TD is the LD, then a Peer will conduct the second review.
- Once development is concluded, the adoption of Standard Operating Procedures (SOPs), conducting personnel training, etc., will be done following routine CAS QA protocols.

Research and Development

When conducting research on new analyses and developing in-house procedures not based on reference methods or published methods, the research and development effort will include the following components:

1) There will be a written *Development Statement* detailing the intent of the research and development effort. This will state the purpose of the work, the resources and references expected to be used, the experimentation that will be performed, and the anticipated result. The following items will be included in the statement:
   a) Equipment to be used.
   b) Quality Control measures to be incorporated into the analysis.
   c) Method Performance (validation) measures to be taken and expectations.
2) There will be an initial internal review and acceptance of the statement by the Technical Director and the Laboratory Director.
3) The person leading a R&D effort will gather information, references, and resources as described in the Development Statement and document those resources.
4) The experimentation will be performed and documented.
5) Once data is collected, it will be interpreted objectively using common assessments of bias and precision. Tests for false negative and false positive results will be used as well as measurements of accuracy and precision.
6) The developer will draw conclusions, and if successful, summarize the results in a brief R&D summary.
7) The summary report will include a documented approval by the Technical Director and the Laboratory Director. The supporting data should be submitted with the report to facilitate the review.
8) Following approval, an SOP will be written for subsequent implementation.

Method Development

When developing and implementing new methods based on reference or published methods, the method development effort will include the following components:

Non-certified (nor certifiable) methods

1) There will be a written *Development Statement* detailing the method development effort. This will state the purpose of the work, the reference method, references expected to be used, the experimentation that will be performed, and the anticipated result. The following items will be included in the statement:
   a) The reference method being implemented and the application(s).
   b) Equipment to be used.
   c) Quality Control measures to be incorporated into the analysis.
   d) Method Performance (validation) measures to be taken and expectations.
   e) Modifications to the reference method.
2) There will be an initial internal review and acceptance of the statement by the applicable Technical Director and the Laboratory Director.

3) The experimentation will be performed and documented.
4) Once data is collected, it will be interpreted objectively using common assessments of bias and precision. Tests for false negative and false positive results will be used as well as measurements of accuracy and precision.
5) The developer will draw conclusions, and if successful, summarize the results in a brief method development summary.
6) The summary report will include a documented approval by the Technical Director and the Laboratory Director. The supporting data should be submitted with the report to facilitate the review.
7) Following approval, an SOP will be written for subsequent implementation on the stated applications.

Certified (certifiable) methods

1) There will be a written *Experimental Plan* detailing the method development effort. This will state the method being implemented, references expected to be used, the experimentation that will be performed, and the anticipated result. The following items will be included in the Plan:
   a) The reference method being implemented.
   b) Equipment to be used.
   c) Quality Control measures to be incorporated into the analysis.
   d) Method Performance (validation) measures to be taken and expectations. This will include method and certification requirements for accuracy and precision, sensitivity, selectivity, calibration/linear range, etc. For methods where NELAC accreditation is being pursued, the requirements of the NELAC Standard (2003 Standard, Quality Systems section 5, Appendix C.3) will be met.
   e) Modifications to the reference method.
2) There will be an initial internal review and acceptance of the Plan by the applicable Technical Director and the Laboratory Director.
3) The method will be set up and run following the procedural steps of the method and the Plan; and will be documented.
4) Once data is collected, it will be interpreted objectively using common assessments of bias and precision. Tests for false negative and false positive results will be used as well as measurements of accuracy and precision.
5) The developer will draw conclusions, and if the results meet the method performance criteria in the method and/or Experimental Plan, the results will be summarized in a brief method development summary.
6) The summary report will include a documented approval the Technical Director and the Laboratory Director. The supporting data should be submitted with the report to facilitate the review.
7) Following approval, an SOP will be written for subsequent implementation.

Method Investigations

1) There will be a written *Investigation Statement* detailing the method investigation effort. This will state the purpose of the investigation, the CAS procedure, the targeted problem, the experimentation that will be performed, and the desired improvement result. The following items will be included in the statement:
   a) The CAS procedure being investigated and the equipment used.
   b) A brief discussion of the problem, the solutions being investigated, and the impact on method compliance and data quality.
   c) The experimentation used to perform the investigation.
   d) The Method Performance (validation) measures that will be taken to re-establish conformity to QA/QC criteria.
2) There will be an initial internal review and acceptance of the statement by the applicable Technical Director and the Laboratory Director.
3) Once data is collected, it will be interpreted objectively using the assessments applicable to that analysis and CAS SOP.
4) The investigator will draw conclusions, and if the results meet the method performance criteria in the method and SOP, the results will be summarized in a brief method investigation summary.
5) The summary report will include a documented approval by the Technical Director and the Laboratory Director. The supporting data should be submitted with the report to facilitate the review.
6) Following approval, the CAS SOP will be revised to implement the changes to procedure.

Documentation

The developer or investigator will generate the written Development or Investigation statements, or Experimental Plan, and provide them for initial review prior to beginning experimentation and data collection. The initial review and acceptance of the Statement will be documented. The laboratory QA PM will keep this documentation on file.

The developer or investigator will generate the written summary report and validation package, and will submit supporting data for review. The approval of the development or investigation (and SOP changes) will be documented and the laboratory QA PM will keep this documentation on file.

Steve Vincent — 12-15-09

Steve Vincent, President/CEO — Date

Lee Wolf — 12-15-09

Lee Wolf, Chief Quality/Ethics Officer — Date

Policy for Standards and Reagents Expiration Dates

September 2009
Effective September 28, 2009

The purpose of this policy is to state the standardized requirements for assigning expiration dates to standards and reagents used in the laboratories of Columbia Analytical Services. It is a requirement of NELAP Quality System standards, the DoD Quality System Manual (QSM), and ISO 17025 to have written protocols to ensure the use of standards and reagents of appropriate quality. Additionally, documentation of the expiration date of reagents and standards is required. This policy is intended to meet the requirements of NELAC, DOD, and ISO 17025.

This policy is applicable to all purchased and prepared standards and reagents used by the laboratory to generate reported data. This includes raw (neat) materials, stock, intermediate, working, and calibration standards and/or reagents. This does not include solvents and acids.

In general, the expiration date is the date after which a standard or reagent shall not be used. It is either the date assigned by the manufacturer, the date (duration) specified by the applicable reference method, or it is a date assigned by the laboratory under this policy.

General Policies:

1. All standard and reagent expiration dates/periods shall be listed in the applicable laboratory SOP.

2. When establishing an expiration date, the following hierarchy will be used:

   - If the cited analytical method specifies the expiration date/period, that date shall be used.
   - If the cited analytical method does not specify the expiration date/period, then the date assigned by the manufacturer will be used.
   - If the cited analytical method does not specify the expiration date/period, and an expiration date is not assigned by the manufacturer, then the laboratory will assign the expiration date according to the CAS Standardized Expiration Dates tables below.

CAS Expiration Dates for Reagents	
Chemical	Expiration Date
Purchased neat reagents	5 years after receipt
Inorganic reagent solutions	1 year from preparation or receipt
Organic reagent solutions	6 months from preparation or receipt

CAS Expiration Dates for Standards	
Chemical	Expiration Date
Purchased neat standards	5 years after receipt
Inorganic stock standard solutions	1 year from preparation or receipt
Inorganic secondary, intermediate, or working standard solutions	6 months from preparation or receipt
Purchased semivolatile organic stock standard solutions	1 year from receipt
Prepared semivolatile organics stock standards	1 year from preparation
Semivolatile organic secondary, intermediate, or working standard solutions	6 months from preparation or receipt
Purchased volatile organics stock standards – **unopened** ampules	1 year from receipt
Purchased volatile organics stock standards – **opened** ampules	≤2000 mg/L 1 month after opening >2000 mg/L 3 months after opening
Prepared volatile organics stock standards	1 year from preparation
All volatile organics secondary, intermediate, or working standards* * note: common 'gases' standards and standards used for calibration should not be older than 7 days	≤20 mg/L 7 day expiration date >20 and ≤200 mg/L 1 month expiration date >200 mg/L 3 month expiration date
Dioxin/Furan and PCB stock standards	5 years from receipt
Dioxin/Furan and PCB working standards	1 year from preparation or receipt
Derivatized (prepared) semivolatile organics standard solutions	1 year from date of derivatization

3. The expiration date of a prepared reagent or standard cannot exceed the expiration date of the starting material, with the exception of standards prepared via in-lab derivatization to yield a different compound. The expiration date of a reagent or standard cannot be extended by preparing a dilution of it. For example, a purchased standard has an expiration date of July 15, 2009. A standard prepared on February 20, 2009 from this purchased standard would ordinarily have an expiration date of six months (namely, 8/20/2009), but since the purchased standard expires before six months, the prepared standard would be assigned an expiration date of July 15, 2009.

4. A multicomponent prepared reagent or standard will be assigned an expiration date not to exceed the expiration date of any of the components' expiration date. For example, a prepared standard is made from purchased standard A (with an expiration date of August 5, 2009) and from purchased standard B (with an expiration date of December 15, 2009). Consequently, the prepared standard will have an expiration date of August 5, 2009.

5. The stability and concentration of the reagent or standard are to be taken into account when assigning the expiration date. Certain solutions, depending on use and storage, may have shorter usable life time than defined by the method, manufacturer, or this policy; and should be assigned expiration dates accordingly. Reagents and standards must be stored under conditions specified by the test method and outlined in the analytical SOP.

6. Expiration dates can be extended under the following conditions:
   - A new, replacement reagent or standard is not readily available from vendors and,
   - The cited analytical method does not specify the expiration date/period and,
   - The material has been stored under conditions specified by the analysis method and outlined in the analytical SOP and,
   - The material is not reactive, volatile, or prone to degradation under the specified storage conditions and,
   - The suitability of the material is verified by the laboratory as follows, under the same valid analysis conditions used for sample analysis, and meet the following criteria:

     a. For reagents:
        i. Perform a blank and LCS pair of analysis three times using three different subaliquots of the reagent.
        ii. Each LCS result must be within the specified control limits for the test.
        iii. The %RSD for the three LCS's must be <10%.
        iv. Each blank result must be < 1/2MRL for every compound to be reported from subsequent analysis.
     b. For standards:
        i. Analyze three separate dilutions of the standard at a concentration near the midpoint of the calibration range. (Note that standards below this concentration cannot be re-verified).
        ii. The average result must be within ± 5% of the original true value.
        iii. The %RSD for the three results must be <10%.

     If these conditions and criteria are met and documented, the material may be assigned a new expiration period the same as newly prepared material.

Lee Wolf, Corporate Director of Quality Assurance — Date: 9-10-09

Steve Vincent, President — Date: 9-15-09

Policy for the Use of Accreditation Organization Names, Symbols, and Logos
September 2009
Effective October 1, 2009

The purpose of this policy is to state Columbia Analytical Services' (CAS) requirements and restrictions for the company use of the name, symbols, and logos of accreditation organizations. In general, the names, symbols, and logos used by these organizations are the property of the organization. Therefore, it is a policy that CAS will comply with the requirements and policies of the organizations that accredit our laboratories.

The NELAC Institute (TNI): The TNI Board of Directors approves and oversees the use of TNI logos and marks (TNI, NELAC, NELAP) by programs, members, and other entities. In consideration that CAS is a member of TNI, CAS will abide by the following TNI policy and be subject to the TNI Consequences of Misuse.

All persons and entities that use or reproduce TNI logos and marks:

1. *Shall restrict access to them by unauthorized parties.*
2. *Shall use them only for purposes and activities authorized by the TNI Board of Directors.*[a]
3. *Shall endeavor to avoid statements in relation to their use that the TNI Board of Directors may consider misleading or unauthorized.*
4. *May not imply endorsement or approval by TNI in communication media such as the Internet, documents, brochures, or advertising without the expressed consent of the TNI Board of Directors.*
5. *May not imply an association or partnership with TNI when such an arrangement has not been authorized by the TNI Board of Directors.*

[a] Authorized uses and activities are listed in the 2003 NELAC Standard, Section 6.8

American Association for Laboratory Accreditation (A2LA): CAS will comply with A2LA policy *P101 – Reference to A2LA Accredited Status – A2LA Advertising Policy*[b].

- CAS will only use the A2LA logo and symbol/phrase "A2LA Accredited" at individual CAS laboratory locations which have demonstrated to be in compliance with A2LA quality system requirements for the applicable A2LA accreditation program (e.g. Testing Laboratory).
- The "A2LA Accredited" symbol will not be used by a CAS laboratory that is not A2LA accredited and the symbol will not be used by a CAS laboratory that has only applied for accreditation.

- When promoting A2LA accreditation, CAS will follow the requirements of the A2LA policy.
- Where the "A2LA Accredited" symbol is used to endorse results on reports, it will always be accompanied by the A2LA certificate number and an indication of the type of laboratory (i.e., testing laboratory).

[b] The A2LA policy can be found at http://www.a2la.org/policies/A2LA_P101.pdf

International Organization for Standardization (ISO): ISO does not perform assessments and therefore is not a certification or accreditation organization. ISO is a standards development organization and compliance with an ISO standard does not imply ISO endorsement. ISO's statement on the use of the name and logo is listed below, and can be found at the following URL: http://www.iso.org/iso/support/name_and_logo.htm ISO has also provided a guide for how to publicize certification to an ISO standard: http://www.iso.org/iso/publicizing2005-en.pdf

Use of ISO's name®

Within the context of international standardization or related activities (such as consultancy, training or conformity assessment including certification) "ISO" (or "iso") is the short name of the International Organization for Standardization. The name is registered within this context as the sole property of ISO and the Organization will protect its name on behalf of all ISO's members - the national standards institutes of some 150 countries. In particular, ISO will not authorize the use of the name "ISO" (or "iso") by any organization other than its members in Internet domain names, names of Web sites, trademarks, companies / organizations, products, etc. Such use could mislead third parties into believing that the domain name / Web site / trademark / company / organization / product concerned represents ISO, or has been approved or authorized to act on behalf of ISO or belongs to ISO.

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The organizations specifically discussed in this policy do not comprise a complete list of organizations to which the policy applies. It is reiterated that, with regards to the use of names, symbols, and logos; it is a policy that CAS will comply with the policies of the organizations that accredit our laboratories.

Lee Wolf, Corporate Director of Quality Assurance	9-21-09 Date
Steve Vincent, President	9-21-09 Date

Policy for Internal Quality Assurance Audits

May 2009
Effective July 1, 2009

The purpose of this policy is to identify the requirements for performing internal systems audits and data audits in the laboratories of Columbia Analytical Services. Internal audits are necessary to ensure that laboratory operations work within the quality systems and that these systems yield data of high quality. Internal audits are also necessary in order to meet certification and accreditation requirements. The internal auditing practices used at each facility will meet the requirements of NELAC quality system standards, the Department of Defense (DoD) Quality System Manual (QSM), and ISO 17025.

For systems audits, the concept of this policy is that corporate quality assurance audits will evaluate the laboratory QA systems and operation horizontally, or as an overall 'umbrella' assessment, whereas local QA audits will be 'drill down' audits focused on technical correctness and data validity. It is practical to verify related systems implementation as these audits are conducted.

For electronic data auditing, the concept is to assess critical data from high liability steps of procedures, from all applicable instruments, in a frequent manner (quarterly) so as to identify any potential problems relatively quickly. This is in contrast to performing 100% data assessment from a subset of instruments quarterly and taking a long period of time to assess all instruments.

Definitions

- System audits are audits used to evaluate quality system implementation, policies, procedures, laboratory practices, and testing activities of the laboratory.

- Data audits are used to assess reported laboratory data. This includes all data used to generate the reported results and the final report itself. These are performed as 'desk audits' of reported data packages, and supporting data if not included in the reported data.

- Electronic data audits are used to assess laboratory data that is processed, interpreted, used by the analyst in electronic format. This is generally limited to electronic chromatographic data.

    - "Critical" and "high liability" data – Data related to the tuning, calibration, calibration verification, and QC analyses for an analysis; as well as data vulnerable to improper manipulation (improper processing/reprocessing of files, clock changes, poor interpretation of control data, peak integrations, etc., as described in CAS Ethics policies).

Specific internal auditing policies are as follows:

1. A comprehensive internal audit will be conducted annually (approximately every 12 months) at each laboratory. The audit will address all elements of the quality system and will include environmental testing activities, and used to meet the annual internal audit requirements of NELAC, DoD, and ISO 17025. In general, the comprehensive audit will be conducted and lead by the Quality Assurance Director (QAD), with assistance from the laboratory Quality Assurance Program Manager (QA PM).

   The laboratory QA PM will not be required to conduct an additional comprehensive audit. While performing the system and data audits described below, the QA PM will verify ongoing implementation of many QA systems.

2. Each laboratory QA PM will conduct three technical systems audits per calendar quarter. These audits will be technically-focused audits of three different test procedures and technologies.
   a. The three procedures will be varied throughout the year such that analytical disciplines (e.g. digestion, extraction, ICP, ICP/MS, titrimetric, colorimetric, GC, GC/MS, HPLC, microbiology, etc) from all sections of the laboratory are assessed in a year (for laboratories with fewer than 12 tests performed, the same tests will be audited more than once).
   b. The audits will assess SOP and method compliance.
   c. The audits will assess the use of sound analytical techniques and practices.
   d. The audits will assess the analyst(s) training and documentation of /proficiency.
   e. The audit will assess all aspects of the test being evaluated, including sample handling/preparation, calibration, sample batching/run sequences, standards, quality control, instrument operation/maintenance, data interpretation, data review/reporting, and applicable quality assurance.

3. Each laboratory will conduct two complete hardcopy data audits per quarter. These audits will focus on data validity, accuracy, and completeness. Data audits will be performed on hardcopy raw and reported data (or electronic version of) and on a 'Service Request basis'.
   a. The audits will be performed on data generated no earlier than three months prior to the audit.
   b. Service requests are to be chosen at random to encompass various analytical disciplines of the laboratory over the course of a year.
   c. The audit will assess the validity of the laboratory procedures used to generate the results reported, from sample receipt to analysis to data reporting, and the accuracy and completeness of the final report.
   d. The audit may be used as a convenient way to assess training documentation for the analysts who performed the analyses.

4. DoD report reviews will be conducted quarterly at the frequency required by the DoD QSM.

5. Electronic data auditing

    a. Each laboratory will conduct random screening of chromatographic data using Mint Miner software (where analytical software is compatible) every quarter on every instrument on data generated that quarter.
    b. Mint Miner software will be adequately configured in order to make screening effective.
    c. Using the screening results, data files will be selected for auditing from each instrument each quarter. Two sequences will be audited, one an initial calibration and one a typical sample analysis sequence. Test methods are to be chosen at random to encompass various methods performed.
    d. The audits will focus on calibration and QC data, including the evaluation of proper processing of files, interpretation of data, peak integrations, and comparison of raw electronic data to 'interpreted' and approved data.
    e. If screening results indicate significant potential problems, additional files should be inspected. The QA PM will conduct these added audits as needed.
    f. If Mint Miner software is not compatible with instrument software, auditing will be performed manually by the QA PM by auditing the data from two sequences per quarter, including one initial calibration sequence, per instrument.

6. As with any audit, additional auditing and investigation may be necessary based on the audits performed and magnitude of findings.

7. Each laboratory facility will have a written and approved standard operating procedure (SOP) for conducting their internal audits. The SOP will include detailed procedures for technical system audits, data audits, and electronic data audits as defined in this policy. In addition to meeting the standard CAS requirements for administrative SOPs, the SOP will include details of the audit processes, use of checklists, documentation, audit reporting, corrective action, and resolution of audit findings.

Lee Wolf, Corporate Director of Quality Assurance — Date: 5-5-09

Steve Vincent, President — Date: 5-5-09

Columbia Analytical Services

CAS Quality and Ethics Policy Statement
March 2009

Columbia Analytical Services (CAS) vision is simple. We strive to be the best in everything we do. This includes ethics and professional practice where CAS is committed to the highest standards of ethical behavior and quality of its analytical testing.

Unethical behavior carries a heavy price - one that we do not want to bear. This includes loss of reputation, loss of business, civil and criminal penalties, and government and customer sanctions.

CAS is committed to excellence and superior performance in everything we do. We will not sacrifice our ethical principles in order to achieve business success. This means we will always strive to conduct business honestly and with integrity. We will always follow and obey the law of the land in which we are operating our business. We will always follow, to the best of our ability, standard operating procedures, rules and regulations that apply to our industry and specifically to our laboratory operations. Our customers, employees, suppliers and communities that we serve expect and deserve nothing less than the highest standards of conduct and compliance.

The following are the critical elements of the Quality and Ethics program at CAS.

- The Executive Management and Board of Directors of CAS sponsor and support the Quality and Ethics program through their personal commitment and by providing the necessary resources to promote this program throughout the organization.
- Chief Quality and Ethics Officer. The position is responsible for the quality and ethics program, ensures that appropriate resources are provided, reviews and recommends changes in the program, and resolves ethical and quality issues brought to management attention. This Officer reports directly to the Board of Directors Audit Committee on quality and ethics.
- Core Values. The CAS Statement of Core Values was developed internally with input from the entire company. We are committed to ensuring the integrity and quality of data, and meeting the needs of our clients, while conducting business with high ethical standards. We hold strong to the core values of Honor, Truth, and Fairness. We are committed to these values and rely on them when confronted by difficult choices.
- Ethical Code of Conduct. As a member of the American Council of Independent Laboratories (ACIL) and part of the laboratory industry, CAS subscribes to and supports the core values and ethical codes established by this industry organization.

- CAS Code of Conduct. CAS requires its employees to be introduced to and to sign the "CAS Commitment to Excellence in Data Quality" statement and to comply with standards outlined in Section 6, Employee Conduct, of our Employee Handbook. All personnel concerned with analytical testing activities within the laboratory are required to acquaint themselves with the quality documentation and to implement these policies and procedures in their work.
- Open Door Policy. Employees have the right and obligation for open communications to ask questions, seek guidance, and report incorrect practices and wrong doing without fear of retribution. As described in the CAS Open Door Policy; CAS believes in using the chain-of-command channels for this dialogue. However, if there is fear or a concern that using this approach is not appropriate, employees are free to take their concerns to the President, the Director of Human Resources, the Chief Administrative Officer, the Chief Quality Officer, or the company Ombudsman. Employees may do so without fear of retribution.
- Ombudsman Program. CAS has implemented an external ombudsman/hotline program through EthicsPoint, a phone and internet-based reporting system, to enhance communication and empower employees to promote safety, security, and ethical behavior. Employees can file a report anonymously to address issues in the workplace and to cultivate a positive work environment.
- Internal Audits. Policies are established to ensure that internal systems and data audits are conducted periodically in addition to external agency and client audits. The data audits include a detailed in-depth review of hardcopy data and electronic data to ensure compliance with the CAS Quality program and on-going data integrity.
- NELAP Accreditation. CAS management is committed to compliance with the NELAP standards. CAS maintains NELAP accreditation and as such includes quality systems documented in QA Manuals, documented procedures in Standard Operating Procedures (SOPS) and policies, and documented training for demonstration of capabilities.
- Ethics Training. CAS has the obligation to provide training to its employees with respect to company policies concerning business conduct. This includes introductory training on this, and related policies, at the time of hire; in-depth "core" training within one year of hire, and on-going refresher training on a semi-annual basis.

The CAS Quality and Ethics Program has been in place for several years. However, this is a "living" program that will change and improve as the company grows and changes.

Steve Vincent, President/CEO — Date: 3-19-09

Lee Wolf, Chief Quality/Ethics Officer — Date: 3-19-09

STANDARD OPERATING PROCEDURE

SOXHLET EXTRACTION

EXT-3540

Revision 10

October 15, 2009

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Approved By: _____ Supervisor — 10/5/09 Date

_____ QA Manager — 10/5/09 Date

_____ Laboratory Manager — 10/5/09 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

SOXHLET EXTRACTION

1. SCOPE AND APPLICATION

1.1. This procedure uses techniques described in EPA Method 3540C for extracting nonvolatile and semi-volatile organic compounds from solids such as soils, sediments, sludges, and wastes.

1.2. This method is applicable to the isolation and concentration of water insoluble and slightly water soluble organics in preparation for a variety of chromatographic procedures.

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2. SUMMARY OF METHOD

2.1. This procedure describes the Soxhlet extraction process. The Soxhlet extraction ensures intimate contact of the sample matrix with the extraction solvent over a period of time. The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor above a plug of glass wool, and extracted using an appropriate solvent for a prescribed amount of time.

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2.2. Following the extraction period, the resulting extract is then dried, concentrated, and as necessary, exchanged into a solvent compatible with the cleanup or determinative step being employed.

3. INTERFERENCES

3.1. Phthalate esters can pose difficulties when performing sample extractions for organochlorine pesticides, PCBs, and other semi-volatile organics. Phthalates are easily extracted or leached from materials containing plastics during laboratory operations. Interferences from phthalates can best be minimized by avoiding contact with any plastic materials.

3.2. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. All apparatus must be cleaned prior to use on individual samples.

3.3. Refer to SW-846 Method 3500 for additional discussion of interferences. Additional cleanup procedures are described in the applicable CAS SOP.

4. DEFINITIONS

4.1. Surrogate - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to extraction and analysis. Percent recoveries are calculated for each surrogate.

4.2. Method Blank (MB) - The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

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4.3. Laboratory Control Sample (LCS) - A laboratory control sample is an analyte-free matrix to which a known quantity of analyte is added. The LCS is subjected to the same processing as field samples and is carried through the entire analytical process. The percent recovery of the analyte in the LCS is used to assess method performance.

4.4. Matrix Spike/Duplicate Matrix Spike (MS/DMS) Analysis - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the method used for the analysis. Samples are split into duplicates, spiked, and analyzed as a MS/DMS pair. Percent recoveries are calculated for each of the analytes detected. The relative percent difference (RPD) between the duplicate spikes (or samples) is calculated and used to assess analytical precision.

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5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. This method uses Dichloromethane, a known human carcinogen. Viton brand gloves should be used while rinsing, pouring or transferring the solvent

6. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1. Refer to the applicable section in the determinative SOP for sample collection, preservation, and holding times.

6.2. The extract holding time is 40 days from sample preparation to analysis.

7. APPARATUS AND MATERIALS

7.1. Soxhlet extractor - 40 mm ID, with 500 ml Erlenmeyer flask.

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7.2. Vacuum filtration funnel - funnel has a glass wool plug in bottom, and is filled with approximately 100 g anhydrous sodium sulfate.

7.3. Kuderna-Danish (K-D) apparatus

7.3.1. Concentrator tube - 10 ml, graduated (Kontes K-570050-1025 or equivalent)

7.3.2. Evaporation flask - 500 ml (Kontes K570001-500 or equivalent). Attach to concentrator tube with springs, clamps or equivalent.

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7.3.3. Snyder column - Three ball macro (Kontes K-503000-0121 or equivalent).

7.3.4. Springs - 1/2 inch (Kontes K-662750 or equivalent).

7.4. Boiling chips - approximately 10/40 mesh (silicon carbide or equivalent), pre-cleaned by solvent rinsing

7.5. Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

7.6. Vials - Glass, 2 ml capacity, with Teflon lined screw or crimp top.

7.7. Glass thimble or glass wool - contaminant free.

7.8. Heating mantle - Rheostat controlled.

7.9. Disposable glass Pasteur pipet and bulb.

7.10. Drying oven.

7.11. Desiccator.

7.12. Crucibles - Porcelain or disposable aluminum.

7.13. Apparatus for grinding.

7.14. Analytical balance - 0.001 g.

7.15. Graduated pipets, 1, 2, 5 and 10 mL. Pipets are pre-tested by lot for accuracy.

7.16. Eppendorf-type pipet

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8. REAGENTS

8.1. Pesticide grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2. Organic-free reagent water. All references to water in this method refer to organic-free reagent water, as defined in Chapter One of SW-846.

8.3. Sodium sulfate (granular, anhydrous), Na_2SO_4. Purify by heating at 400°C for 4 hours.

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8.4. Extraction Solvents - Samples are extracted using one of the following solvent systems:

8.4.1. Acetone/Hexane (1:1) (v/v), CH_3COCH_3/C_6H_{14}. Pesticide quality or equivalent. Soil, sediment, and aqueous sludge samples.

8.4.2. Dichloromethane/Acetone (1:1 v/v), CH_2Cl_2/CH_3COCH_3. Pesticide quality or equivalent. Soil, sediment, and aqueous sludge samples.

8.4.3. Dichloromethane/Acetone (4:1 v/v), CH_2Cl_2/CH_3COCH_3. Pesticide quality or equivalent. Soil, sediment, and aqueous sludge samples.

8.4.4. Dichloromethane, CH_2Cl_2. Pesticide quality or equivalent. Other miscellaneous sample matrices.

8.5. Exchange Solvents

8.5.1. Hexane, C_6H_{14}. Pesticide quality or equivalent.

8.5.2. Methyl–tert-butyl ether (MTBE). Pesticide quality or equivalent.

9. PREVENTIVE MAINTENANCE

Routine cleaning of the extraction apparatus is necessary, including all parts exposed to contact with samples, especially extraction thimbles and the Soxhlet apparatus.

10. RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

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11. PROCEDURE

11.1. Test-specific benchsheets are attached. These benchsheets list such information as solvents, solvent exchanges, weights, and volumes specified for the determinative method. Use the correct benchsheet and record all extraction and sample information. To assist the analyst, a brief description of the procedure is given on the backside of the benchsheet.

11.2. Sample Handling. Also refer to the SOP for *Subsampling and Compositing of Samples*.

11.2.1. Sediment/soil samples – In general, decant and discard any water layer on a sediment or soil sample. However, for sediment samples it is strongly recommended that the analyst consult with the Project Chemist to confirm the procedure to be used for the project (see SOP GEN-SUBS). If water is to be included in the sediment sample, mix the water into the sample. Mix all samples thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

11.2.2. Waste samples - Samples consisting of multi phases have each phase extracted separately. If sample contains a significant portion of water, water is pulled off and extracted. Oil phases are pulled off, usually diluted in hexane or iso-octane and extracted. This procedure is for solids only.

11.2.3. Dry waste samples amenable to grinding - Grind or otherwise subdivide the waste so that it passes through a 1 mm sieve.

11.2.4. Gummy, fibrous, or oily materials not amenable to grinding should be cut, shredded, or otherwise broken up to allow mixing, and maximum exposure of the sample surfaces for extraction. The professional judgment of the analyst is required for handling these difficult matrices.

11.3. Determination of sample % dry weight - In certain cases, sample results are desired based on dry weight basis. Refer to the *SOP for Total Solids* (GEN-160.3). If the determination is performed by the organics preparation personnel, a portion of the sample for this determination should be weighed out at the same time as the portion used for analytical determination.

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11.4. Sample extraction

11.4.1. Place approximately 300 ml of the extraction solvent into a 500 ml Erlenmeyer flask containing one or two clean boiling chips. Attach the flask to the extractor.

11.4.2. Set up the sample thimbles

11.4.2.1. Refer to the determinative SOP (see Table 1 for a list of applicable SOPs) for the preparation, concentration, storage, and expiration for the surrogate, LCS, and MS spiking solutions. These SOPs also list the resulting final spike concentrations.

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11.4.2.2. Weigh out the specified amount of the solid sample to be extracted in a beaker, along with MB (anhydrous sodium sulfate) LCS (clean sand) samples. Blend samples with an equal amount of anhydrous sodium sulfate and place directly into a Soxhlet extractor with a glass wool plug. If the sample possesses high moisture content, additional sodium sulfate should be added to produce a free-flowing solid. The glass wool should plug the unit so that the sample is held in the extractor, but allow the solvent to drain freely for the duration of the extraction period.

11.4.2.3. Add the surrogate standard spiking solution onto the samples. For the sample(s) in each analytical batch selected for QC spiking (LCS and MS samples), add the appropriate volume of the appropriate spiking standard. Each standard should be brought to room temperature before using. Addition of surrogate and spike is routinely witnessed by a second analyst to assure completeness.

11.4.3. Extract the sample for 16-24 hours at 4-6 cycles/hr.

11.4.4. Allow the extract to cool after the extraction is complete.

11.5. Extract concentration

11.5.1. Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10 ml evaporation flask.

11.5.2. Transfer the extract to a K-D concentrator. Wash the extractor flask with three 5-10 mL portions of extraction solvent to complete the quantitative transfer.

Note: It may be necessary to dry the extract by passing it through a vacuum filtration funnel containing about 100g of anhydrous sodium sulfate during the transfer. This is most often needed for tissue samples.

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11.5.3. Add a few clean boiling chips to the flask and attach a three ball Snyder column. Prewet the Snyder column by adding about 1 ml if Dichloromethane to the top of the column. Place the K-D apparatus on a hot water bath (15-20°C above the boiling point of the solvent) 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 10-20 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 5-10 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Record the temperature of the water bath on the benchsheet.

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11.5.4. If a solvent exchange is required, momentarily remove the Snyder column, add approximately 10 ml of the exchange solvent and a new boiling chip, and reattach the Snyder column. Re-concentrate the extract as described above, raising the temperature of the water bath, if necessary, to maintain proper distillation. When the apparent volume again reaches 2-5 ml, remove the K-D apparatus from the water batch and allow it to drain and cool for at least 10 minutes. (Note: For some analytes such as pesticides, it is preferable to allow the volume to go down to only 5-10 ml.)

Determinative Method	Exchange Solvent Required for Analysis	Exchange Solvent Required for Cleanup
8081	Hexane	Hexane
8082	Hexane	Hexane
8141	MTBE	Hexane

11.5.5. Alternatively, remove the solvent collection device from the top of the Snyder column and add solvent down the Snyder column without removing from the bath. To use this technique, this must be set up in a hood.

11.5.6. Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 ml of Dichloromethane or exchange solvent. The extract may be further concentrated by using the technique described below, or adjusted to 10.0 ml with the solvent last used.

11.5.7. If further concentration is required, nitrogen blowdown technique is used to adjust the extract to the final volume.

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11.5.7.1. Place the concentrator tube in a warm water bath (approximately 35°C) and evaporate the solvent volume to the required level using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

CAUTION: Do not use plasticized tubing between the carbon trap and the sample.

11.5.7.2. The internal wall of the tube must be rinsed down several times with the appropriate solvent during the operation. During evaporation, the solvent level in the tube must be positioned to prevent water from condensing into the sample (i.e., the solvent level should be below the level of the water bath). The volume of extract in the tube must be monitored during blowdown to avoid loss of more volatile analytes. Under normal operating conditions, the extract should not be allowed to become dry.

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CAUTION: When the volume of solvent is reduced below 1 ml, semivolatile analytes may be lost.

11.5.7.3. Adjust the extract final volume to the prescribed volume with the solvent last used. Measure the final extract volume using an appropriate sized graduated pipet (7.15).

11.5.8. During the final volume step, transfer the extracts to an appropriate storage or autosampler vial. Label the vial with the sample or QC identification and store in the extract storage area. The extracts obtained may now be analyzed for the target analytes using the appropriate determinative technique. The extract holding time is 40 days from sample preparation to analysis.

12. QUALITY CONTROL

12.1. Refer to the SOP for the determinative method and SOP for *Analytical Batches and Analytical Sequences* for minimum QC requirements. Project-specific batching protocols may also be required.

12.2. The QC solutions required by the method must be added as described in the analytical method. The amount and identification of QC solutions added must be documented on the benchsheet. Any reagent blanks, laboratory control samples, or matrix spike samples should be subjected to exactly the same analytical procedures as those used on actual samples.

13. DATA REPORTING AND REVIEW

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13.1. Preparation of all samples must be documented on a benchsheet. All information regarding the sample(s) extracted, aliquoting, QC spiking, extraction steps, date and time of extracdtion, etc. must be documented by the person(s) performing the extraction.

13.2. The benchsheet must be reviewed by the extraction lead, supervisor, or instrument lab analyst. The instrument lab analyst signs off on the benchsheet, thus accepting custody of the extracts.

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14. CORRECTIVE ACTION

14.1. Refer to the SOP for *Nonconformity and Corrective Action (ADM_CA)* for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2. Documentation of a nonconformity must be done using a Nonconformity and Corrective Action Report (NCAR) when: a) corrective action is not taken or not possible b) corrective action fails to correct an out-of-control problem on a laboratory QC or calibration analysis c) reanalysis corrects the nonconformity but is not a procedurally compliant analysis.

15. POLLUTION PREVENTION AND WASTE MANAGEMENT

It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when recycled or disposed of properly.

16. WASTE MANAGEMENT

16.1. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the laboratory Safety Manual, Sections 6 and 7.

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16.2. This method uses Dichloromethane and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and recycled off site.

16.3. This method uses non-halogenated solvents and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and disposed of in accordance with Federal and State regulations.

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17. METHOD PERFORMANCE

Available method performance data is given in the reference method. In addition, this procedure was validated through single laboratory studies of accuracy and precision as specified in the determinative procedures.

18. TRAINING

18.1. Training outline

18.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

18.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

18.1.3. Perform initial demonstration of capability by spiking into 'clean matrix' as described in the determinative methods SOP. Summaries of the initial demonstration of capability results are reviewed and signed by the technical director. A copy is forwarded to QA for documentation and filing in the employee's training file.

18.2. Training is documented following the SOP *for Documentation of Technical Personnel Training*.

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

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19. REFERENCES

19.1. EPA SW-846, Test Methods For Evaluating Solid Waste, Third Edition, Update III, December 1996, Method 3540C, Revision 3

19.2. EPA SW-846, Test Methods For Evaluating Solid Waste, Third Edition, Update III, December 1996, Method 3500B, Revision 2

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20. CHANGES SINCE THE LAST REVISION

20.1. Replaced methylene chloride with dichlolormethane.
20.2. Sec 11.3 Corrected sop reference.
20.3. Sec 18.1.3 Revised IDC language to reflect solid matrix.

Table 1

APPLICABLE DETERMINATIVE SOPs

CONGENER-SPECIFIC DETERMINATION OF PCBS BY GG/ECD	SOC-8082C
DETERMINATION OF NITROGEN OR PHOSPHORUS CONTAINING PESTICIDES	SOC-8141
ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY: CAPILLARY COLUMN TECHNIQUE	SOC-8081
PCBS AS AROCLORS	SOC-8082A

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Attachments

Test-Specific Benchsheets

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Additional Prep Information For Pest/PCB Low Level Soils

Service Request #_____ Work Group # Pest:_____

PCB:_____

Solvents/Reagents used:

DCM Lot #_____ Hexane Lot #_____ Sulfate Lot #_____

Acetone Lot # _____

Soxhlet Start (Time/Date/Initial):_____

Soxhlet Stop (Time/Date/Initial):_____

Solvent Exchanged To Hexane (Date/Initial) ☐ yes:_____ S-Evap Temp:_____

Cleanups:

UNCONTROLLED

GPC Clean-up (3640):_____ ☐ all samples

Sulfuric Acid Clean-up (3665):_____ (PCB aliquots only)

Clean-up #3:_____ ☐ all samples ☐ some samples:_____

Clean-up #4:_____ ☐ all samples ☐ some samples:_____

Pest Vial:_____ Vial Storage:_____

PCB Vial:_____ Vial Storage:_____

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Archived Extract Storage:_____

Comments/Observations:_____

_____

_____

_____

_____

Bench Sheet Review Check List
☐ Hold Times Met (if no, Reason: _____)
☐ Prep date, dept, method, product code correct in stealth
☐ Spike Information correct
☐ Weights/Volumes and units correct on raw and final bench sheets
☐ Sample IDs have been checked—Bottle numbers appended if required
☐ Names present for: Started by, Completed by, relinquished by, and witnessed by.
☐ Training has been circled
☐ Extract Storage recorded
☐ Additional Prep Sheet completely filled out (NA or line out Blanks)
☐ All clean-ups have been noted on additional prep sheet
☐ Signed service request with Form V, if applicable, has been attached

STANDARD OPERATING PROCEDURE FOR EXTRACTION OF ORGANOCHLORINE PESTICIDES AND PCBs IN SOIL AND SEDIMENT

EPA 8081A/8082-AR by 3540/Low-Level Method

1. **Mix the sample thoroughly. Weigh 20 grams ± 2.0g dry weight into a beaker. Record the weight to the nearest 0.01 gram. Use the same amount of sodium sulfate for method blank and sand for laboratory control sample.**

1. Mix the sample with a sufficient amount of muffled sodium sulfate to completely dry the sample.

2. Add 200uL of 8081/8082 surrogate to all samples and add 100uL of 8081 matrix spike and 100uL of 8082 matrix spike to appropriate QC samples.

3. Place 300 mL of **4:1 DCM: Acetone** into a 500 mL Erlenmeyer flask with five to ten clean boiling chips. Attach the flask to a soxhlet trumpet. Pour the sample mixture onto the top of a plug of glass wool in the bottom of the trumpet.
4. Add surrogates and analyte spikes as necessary.

5. Extract the sample for 16-24 hours on setting #4, and record the start and stop times on the bench sheet.

6. Decant the extract into a prerinsed K-D apparatus. If necessary, decant the extract through a modified funnel with small amount of muffled anhydrous sodium sulfate (add more sodium sulfate, if necessary) atop a glass wool plug. Rinse the flask three times (and pass through the sulfate, rinsing the sulfate if applicable), adding rinses to the rest of the extract.

7. Evaporate the extract down on an S-Evap. keeping the temperature 80-85°C. Adjust the bath temperature as necessary. **DO NOT LET THE SAMPLE GO DRY.**

8. Evaporate to about 2 mL on an N-Evap, keeping the temperature <39°C. Take extract to 10 mL intermediate volume in DCM.

9. Do GPC cleanup (3640) on the entire 10 mL of extract. Refer to CAS SOP.

10. Evaporate on the S-Evap, keeping temperature 70-75°C. When extracts are at about 10 mL, squirt about 5 mL of hexane down top of Snyder column, keeping collector in the hot water of the bath. If the extract is allowed to cool, it will be necessary to add another boiling chip to the extract. Let the extracts solvent exchange on the S-Evap (75°C) into hexane for until solvent stops dripping. **Do not let the samples go dry.**

11. Concentrate the extract to about 1 mL on an N-evap, keeping the temperature <39°C.

12. Take the extract to 2 mL final volume in hexane and split for 8082-AR if necessary. The 8082-AR portion will then receive a concentrated sulfuric acid clean up (3665), followed by centrifugation.

13. Place 8082-AR portion in a green vial and the Pesticide portion in a yellow vial for analyses. Mark meniscuses on vials.

Approved By / Date
EE 10/13/04
Extractions Manager
QIA 10/14/04
Instrument Lab Manager
10/19-14-04
QA Coordinator

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Additional Prep Information For Pest/PCB Soils

Service Request #_____ Work Group # Pest:_____

PCB:_____

Solvents/Reagents used:

DCM Lot #_____ Hexane Lot #_____ Sulfate Lot #_____

Acetone Lot # _____

Soxhlet Start (Time/Date/Initial):_____

Soxhlet Stop (Time/Date/Initial):_____

Solvent Exchanged To Hexane (Date/Initial) ☐ yes:_____ S-Evap Temp:_____

Cleanups:

UNCONTROLLED

GPC Clean-up (3640):_____ ☐ all samples

Sulfuric Acid Clean-up (3665):_____ (PCB aliquots only)

Clean-up #3:_____ ☐ all samples ☐ some samples:_____

Clean-up #4:_____ ☐ all samples ☐ some samples:_____

Pest Vial:_____ Vial Storage:_____

PCB Vial:_____ Vial Storage:_____

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Archived Extract Storage:_____

Comments/Observations:_____

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Bench Sheet Review Check List
☐ Hold Times Met (if no, Reason: _____)
☐ Prep date, dept, method, product code correct in stealth
☐ Spike Information correct
☐ Weights/Volumes and units correct on raw and final bench sheets
☐ Sample IDs have been checked—Bottle numbers appended if required
☐ Names present for: Started by, Completed by, relinquished by, and witnessed by.
☐ Training has been circled
☐ Extract Storage recorded
☐ Additional Prep Sheet completely filled out (NA or line out Blanks)
☐ All clean-ups have been noted on additional prep sheet
☐ Signed service request with Form V, if applicable, has been attached

ORGANOCHLORINE PESTICIDES AND PCBs IN SOIL AND SEDIMENT

Methods 8081/8082-AR/3540

1. Mix sample thoroughly and weigh approximately 15 grams into a 400 ml beaker. Record the weight to the nearest 0.01 gram. Use the same amount of sodium sulfate for method blank and sand for laboratory control sample.

2. Mix soil with approximately the same weight of muffled anhydrous sodium sulfate.

3. Add 500uL of 8081/8082 surrogate to all samples and add 250uL of 8081 matrix spike and 250uL of 8082 matrix spike to all appropriate QC samples.

4. Place 300 ml of 4:1 methylene chloride:acetone, into a 500 ml Erlenmeyer flask with two or three clean boiling chips. Attach the flask to the extractor and extract the sample for 16-24 hours on setting #4, and record start and stop times on bench sheet.

5. Decant the extract into a prerinsed K-D apparatus via funnel with small amount of muffled anhydrous sodium sulfate (add more sodium sulfate, if necessary). Rinse flask three times with extraction solvent using a squirt bottle and pass through sulfate, rinse sulfate with extraction solvent.

6. Evaporate the extract down on the S-Evap. keeping the temperature 80-85°C. Adjust bath temperature as necessary. **DO NOT LET THE SAMPLE GO DRY.**

7. Evaporate to approx. 2 ml on the N-Evap, temperature <39°C. Take samples to 10 ml intermediate volume in DCM.

8. Do GPC cleanup. Refer to CAS SOP.

9. After GPC, concentrate the sample extract on the S-evap, keeping the temp between 60-75 °C. Concentrate to approximately 5 ml, remove from S-evap, cool, add a new boiling chip, and approximately 10 ml of hexane. Return sample extract to S-evap and concentrate until solvent stops dripping.

10. Concentrate sample to approximately 1 ml on N-evap at 35°C.

11. Take to 2 ml final volume in hexane and split for 8082-AR if needed. 8082-AR will then receive a Sulfuric acid clean up.

12. Place PCB portion in green vials and the Pesticides in yellow vials for analysis. Mark level on vials.

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Approved By / Date

SS 19/13/04

Extractions Manager

9-14-4

Instrument Lab Manager

LW 9-14-04

QA Coordinator

Additional Prep Information For OP Pest Soil

Service Request #_____ Work Group #OP Pest:_____

Solvents/Reagents used:

DCM Lot #_____ Powder Sulfate Lot # _____
Acetone Lot # _____

Start time _____
Stop Time _____

Extract solvent exchanged to MTBE _____ MTBE Lot # _____
S-Evap Temp. _____

Cleanups:
GPC Clean-up (3640): _____ ☐ all samples

Additional Clean-up #2 _____ ☐ all samples ☐ some samples _____

Additional Clean-up #3 _____ ☐ all samples ☐ some samples_____

Vial:_____ Vial Storage:_____

Archived Extract Storage:_____

Comments/Observations:

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Bench Sheet Review Check List
☐ Hold Times Met (if no, Reason: _____)
☐ Prep date, dept, method, product code correct in stealth
☐ Spike Information correct
☐ Weights/Volumes and units correct on raw and final bench sheets
☐ Sample IDs have been checked—Bottle numbers appended if required
☐ Names present for: Started by, Completed by, relinquished by, and witnessed by.
☐ Training has been circled
☐ Extract Storage recorded
☐ Additional Prep Sheet completely filled out (NA or line out Blanks)
☐ All clean-ups have been noted on additional prep sheet
☐ Signed service request with Form V, if applicable, has been attached

EXTRACTION STANDARD OPERATING PROCEDURE FOR

Organophosphorus Pesticides in Soil

Methods 8141/3540

1. Mix sample thoroughly and weigh approximately 15 grams into a 400 ml beaker. Record weight to the nearest 0.01 gram. Use the same amount of sodium sulfate for method blank and sand for laboratory control sample.

2. Mix soil with approximately the same weight of muffled anhydrous sodium sulfate.

3. Add 500uL of 8141surrogate to all samples and add 500uL of mix A and mix B matrix spikes to all appropriate QC samples.

4. Place 300 ml of 4:1 DCM:acetone, into a 500 ml Erlenmeyer flask with two or three clean boiling chips. Attach the flask to the extractor and extract the sample for 16-24 hours on setting #5, and record start and stop times on bench sheet.

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5. Decant the extract into a pre-rinsed K-D apparatus via funnel with small amount of muffled anhydrous sodium sulfate (add more sodium sulfate, if necessary). Rinse flask three times with extraction solvent using a squirt bottle and pass through sulfate, rinse sulfate with extraction solvent.

6. Evaporate the extract down on the S-Evap. to approx. 5mL keeping the temperature 70-75°C. Adjust bath temperature as necessary. **DO NOT LET THE SAMPLE GO DRY.**

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7. Solvent exchange into MTBE unless the extract needs a GPC cleanup. When extracts are at about 10 mL, squirt about 10 mL of MTBE down top of Snyder column, keeping collector in the hot water of the bath. If the extract is allowed to cool, it will be necessary to add another boiling chip to the extract. Let the extracts solvent exchange on the S-Evap (75°C) into MTBE until extract is less than 10 mL; do not let the extracts go dry. ***Special Note: MTBE evaporates at a temperature close to DCM, so watch it closely.**

8. Bring to 1mL final volume in MTBE, place into a 2mL amber vial, and mark level on the vial.

9. **Note: Organophosphorus pesticides are light sensitive. Cover all glassware if extract is to be stored overnight.**

Approved by/Date
EE 1 9/13/04
Extractions Manager
[illegible] 1 9/14/04
Instrument Lab Manager
LW/1 9-14-04
QA Coordinator

Additional Prep Information For Low Level Pest/PCB-Con in Soil

Service Request #_____ Work Group # Pest:_____

CON:_____

Solvents/Reagents used: Acetone Lot # _____

DCM Lot #_____ Hexane Lot #_____ Sulfate Lot #_____

Soxhlet Start (Time/Date/Initial):_____

Soxhlet Stop (Time/Date/Initial):_____

Solvent Exchanged To Hexane (Date/Initial) ☐ yes:_____ S-Evap Temp:_____

Cleanups:

GPC Clean-up (3640):_____ ☐ all samples

Florisil Clean-up (3620):_____ ☐ all samples Florisil Lot # _____

Copper Clean-up (3660):_____ ☐ all samples

Sulfuric Acid Clean-up (3665):_____ (CON aliquots only)

Clean-up #3:_____ ☐ all samples ☐ some samples:_____

Clean-up #4:_____ ☐ all samples ☐ some samples:_____

Pest Vial:_____ Vial Storage:_____

CON Vial:_____ Vial Storage:_____

Archived Extract Storage:_____

Comments/Observations:_____

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Bench Sheet Review Check List
☐ Hold Times Met (if no, Reason: _____)
☐ Prep date, dept, method, product code correct in stealth
☐ Spike Information correct
☐ Weights/Volumes and units correct on raw and final bench sheets
☐ Sample IDs have been checked—Bottle numbers appended if required
☐ Names present for: Started by, Completed by, relinquished by, and witnessed by.
☐ Training has been circled
☐ Extract Storage recorded
☐ Additional Prep Sheet completely filled out (NA or line out Blanks)
☐ All clean-ups have been noted on additional prep sheet
☐ Signed service request with Form V, if applicable, has been attached

ORGANOCHLORINE PESTICIDES AND PCBs CONGENERS IN SOIL AND SEDIMENT

EPA 8081A/8082-CON by 3540/Low-Level Method

1. Mix the sample thoroughly. Weigh 20 grams dry weight into a beaker. Record the weight to the nearest 0.01 gram. Use the same amount of sodium sulfate for method blank and sand for laboratory control sample.

2. Mix the sample with a sufficient amount of muffled sodium sulfate to completely dry the sample.

3. Add 200uL of Congener surrogate to all samples and add 200uL of congener matrix spike and 100uL of 8081 spike to appropriate QC samples. **Be sure to use the congener surrogate.**

4. Place 300 mL of **4:1 DCM: Acetone** into a 500 mL Erlenmeyer flask with five to ten clean boiling chips. Attach the flask to a soxhlet trumpet. Pour the sample mixture onto the top of a plug of glass wool in the bottom of the trumpet.

5. Extract the sample for 16-24 hours on setting #4, and record the start and stop times on the benchsheet.

6. Decant the extract into a prerinsed K-D apparatus. If necessary, decant the extract through a modified funnel with small amount of muffled anhydrous sodium sulfate (add more sodium sulfate, if necessary) atop a glass wool plug. Rinse the flask three times (and pass through the sulfate, rinsing the sulfate if applicable), adding rinses to the rest of the extract.

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7. Evaporate the extract down on an S-Evap. keeping the temperature 80-85°C. Adjust the bath temperature as necessary. **DO NOT LET THE SAMPLE GO DRY.**

8. Evaporate to about 2 mL on an N-Evap, keeping the temperature <39°C. Take extract to 10 mL intermediate volume in DCM.

9. Do GPC cleanup (3640) on the entire 10 mL of extract. Refer to CAS SOP for GPC clean-up.

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10. Evaporate on the S-Evap, keeping temperature 70-75°C. When extracts are at about 10 mL, squirt about 5 mL of hexane down top of Snyder column, keeping collector in the hot water of the bath. If the extract is allowed to cool, it will be necessary to add another boiling chip to the extract. Let the extracts solvent exchange on the S-Evap (75°C) into hexane for until solvent stops dripping. **Do not let the samples go dry.**

11. Concentrate the extract to about 1 mL on an N-evap, keeping the temperature <39°C.

12. Do Florisil (EPA 3620) cartridge clean-up on entire extract. Refer to CAS SOP on florisil clean-ups.

13. Evaporate extracts on N-evap at <39°C to <2mL and bring to 2mL final volume (Note: True final volume will be 4mL)

14. Do a Copper clean-up to remove sulfur. Refer to CAS SOP on sulfur removal. Be sure not to add any solvent since the extract was taken to its final volume in step 13.

15. Split extact ½ for pesticides and ½ congeners. Put pesticide portion in a yellow vial.

16. The congener portion will then receive a concentrated sulfuric acid clean up (EPA 3665), followed by centrifugation.

17. Place exactly 0.5mL in a green vial for congeners. Mark meniscus on vials.

Approved By / Date
EE 1 9/13/04
Extractions Manager
9/14/04
Instrument Lab Manager
LW 9-14-04
QA Coordinator

Additional Prep Information For Anthraquinone in Paper by 3540

Service Request #_____ Work Group # _____

Solvents/Reagents used:

Ethanol Lot #_____ DCM Lot #_____ Hexane Lot #_____

Silica Gel Lot #_____

Soxhlet Start (Time/Date/Initial):_____

Soxhlet Stop (Time/Date/Initial):_____

Solvent Exchanged To Hexane ☐ yes:_____ S-Evap Temp:_____

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Cleanups:

Silica Gel Clean-up (3630):_____

Clean-up Standard ID:_____

Solvent Exchanged To Hexane ☐ yes:_____ S-Evap Temp:_____

Internal Standard ID:_____

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Vial Storage:_____

Archived Extract Storage:_____

Date Completed:_____

Comments/Observations:_____

_____

_____

_____

Bench Sheet Review Check List
☐ Hold Times Met (if no, Reason: _____)
☐ Prep date, dept, method, product code correct in stealth
☐ Spike Information correct
☐ Weights/Volumes and units correct on raw and final bench sheets
☐ Sample IDs have been checked—Bottle numbers appended if required
☐ Names present for: Started by, Completed by, relinquished by, and witnessed by.
☐ Training has been circled
☐ Extract Storage recorded
☐ Additional Prep Sheet completely filled out (NA or line out Blanks)
☐ All clean-ups have been noted on additional prep sheet
☐ Signed service request with Form V, if applicable, has been attached

STANDARD OPERATING PROCEDURE FOR EXTRACTION OF

Anthraquinone in Paper

(NCASI Method AQ-S108.01)

Soxhlet Extraction

1. Weigh 5 grams of pre-cut paper (1x1 cm) into a 250 mL beaker.
2. Add 300 mL of Ethanol and boiling chips to pre-cycled and Dichloromethane (DCM) rinsed Erlenmeyer flasks.
3. Transfer the sample to the soxhlet.
4. Add the surrogate (50 uL of ***2-chloroanthraquinone***) to each sample and the matrix spike (50 uL of ***anthraquinone spike***) to the **MS/DMS/LCS** samples. Only use glass wool for the LCS/MB.
5. Cycle for 12-24 hours on a heating mantle seating of 5-5.5. Adjust as needed to achieve 4-5 wash cycles per hour.

Sample Concentration

6. Concentrate the extracts while they are still on the soxhlet heating mantles. After extracting for a minimum of 12 hours, begin concentrating the extracts by dumping off the recondensed Ethanol collecting in the soxhlet trumpet. Concentrate until the extract is just above the tops of the boiling chips (15-20 mLs). **The flasks will be VERY HOT, so use an oven mitt when handling flasks.**
7. Pour the remaining 15-20 mLs into a DCM rinsed KD. Rinse the flask 2X with Ethanol and 2X with Hexane and pour into the KD. Add a couple boiling chips and evaporate the extracts further on the S-Evap keeping the temperature between 95-98°C. The KDs and Snyder columns might need to be wrapped with aluminum foil to aid in concentration.
8. Once the extracts have concentrated to about 5 mLs, solvent exchange the extracts into Hexane by adding 10 mLs of Hexane into the KD. Repeat this 2 more times. **Once the Hexane is added, the samples will concentrate quickly. Do not let the samples go dry.**
9. Remove the KDs after the third Hexane exchange concentrates to about 5 mLs. Let the KD and Snyder column cool before dismantling.
10. Further concentrate the extracts on the N-Evap to about 2 mLs, keeping the temperature <35°C.

Packing Silica Gel Columns

11. Add a plug of glass wool to the bottom of a large 19 mm glass chromatography clean-up column with a stopcock.
12. Rinse the column 3 times with DCM.
13. Add ½ inch of matrix sand to the top of the glass wool.
14. Weigh out 10g of **5% deactivated silica gel** (rinsed, baked, deactivated, tumbled overnight) into a small beaker.
15. Wet the silica gel with a small amount of Hexane to form a slurry.
16. With the stopcock closed, pour the mixed slurry of silica gel into the top of the column.
17. As the silica gel settles, rinse the walls of the column with additional Hexane.
18. Open the stopcock and drain the Hexane until approximately 2 cm of hexane remains above the silica gel.
19. Close the stopcock and add ½ inch of matrix sand to the top of the column.
20. Condition the column with 40 mL of additional Hexane. Close the stopcock when the solvent reaches the sand.

Silica Gel Column Clean-up

21. Add 25 µL of the AQ clean-up recovery standard ***(d_{10}-benzophenone)*** to each sample extract.
22. Using a pipet, mix the extract and transfer to the column.
23. Rinse the collector with 1-2 mLs of Hexane and transfer to the column. Repeat rinse.
24. Open the stopcock and allow the solvent to go to waste.
25. Just as the solvent reaches the top of the sand bed, add 20 mL of Hexane and allow the solvent to go to waste.
26. Just as the solvent reaches the top of the sand bed, add 10 mL of DCM and allow the solvent to go to waste.
27. Close the stopcock just as the DCM reaches the top of the sand bed.
28. Place a rinsed KD or small flask under the stopcock.
29. Add 50 mLs of DCM to the column and elute the entire 50 mLs into the KD or flask.

Final Volume

30. Concentrate the 50 mLs of DCM extract on the S-Evap, keeping the temperature between 70-75° C.
31. Concentrate to about 5 mLs and solvent exchange into Hexane by adding 5-10 mLs of Hexane into the KD.
32. N-Evap extracts and bring all too approximately 0.5 mL, keeping the temperature <35°C.
33. Add 25 µL of the AQ internal standard ***(d_{10}-anthracene)*** to each sample extract.
34. Take extracts to a 1 ml Final Volume (FV) in Hexane and transfer to red vials marked with "AQ". If a precipitate forms, dilute the sample with enough Ethanol to get the precipitate back into solution. Then take a small aliquot from the diluted sample, solvent exchange into Hexane, and take to a 1 mL FV. Note the new true FV.

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STANDARD OPERATING PROCEDURE

REMOVAL OF SULFUR USING COPPER

SOP No.: EXT-3660
Revision: 4
July 10, 2007

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Approved by: Adam Bradbury — 7-10-07
Supervisor — Date

7-10-07
QA Manager — Date

7/10/07
Laboratory Manager — Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL
NUMBER: _____
Initials: _____ Date: _____

REMOVAL OF SULFUR USING COPPER

1. SCOPE AND APPLICATION

1.1. This procedure is used to perform cleanup of sample extracts prior to analysis for pesticides and/or polychlorinated biphenyls(PCBs), and is based on EPA Method 3660B. This procedure may be used when a sample matrix giving a complex chromatogram (most often in sediment samples, marine algae, and some industrial wastes) prevents accurate quantitation of target analytes. Sulfur will be evident in gas chromatograms obtained from electron capture detectors and flame photometric detectors operated in the sulfur or phosphorous mode. In some cases, the sulfur interference can completely mask significant regions of the chromatogram. The cleanup is applicable to extracts from all sample matrices.

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1.2. This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2. SUMMARY OF METHOD

2.1. The extract (in hexane) is treated with powdered or granular copper. The sample to undergo cleanup is mixed with cleaned copper and is shaken. The extract is removed from the copper. Blanks and replicate analysis samples must be subjected to the same cleanup as the samples associated with them. If not in hexane already, it is important that all the extracts be exchanged to hexane before initiating the following treatments.

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2.2. When performing sulfur cleanups using Method 3660B, Mercury may not be used. EPA Method 3660, with the update to 3660B, no longer allows the use of Mercury.

3. INTERFERENCES

The copper technique requires that the copper powder be very reactive, as evidenced by a bright shiny appearance (see Section 4 for the preparation of this reagent). However, care must be taken to remove all traces of the acid used to prepare the copper, in order to avoid degradation of some analytes.

4. REAGENTS

4.1. Pesticide grade solvents should be used in all steps of the procedure. Other grades may be used, provided it is shown that the reagent is of sufficient purity to permit its use without adversely impacting the accuracy of the determination.

4.2. 10% Nitric acid, HNO_3.

4.3. Acetone, CH_3COCH_3 - Pesticide grade or equivalent.

4.4. Hexane, C_6H_{14} - Pesticide grade or equivalent.

4.5. Copper powder – Remove oxides by treating with 10% nitric acid, rinse with a profuse amount of organic-free reagent water to remove all traces of acid, rinse with acetone and dry under a stream of nitrogen. (Copper, fine granular Mallinckrodt, or equivalent).

5. APPARATUS

5.1. Pastuer pipets.

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5.2. Vials - 1, 2 and 10 mL, glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

5.3. Vortex mixer.

6. SAFETY

6.1. The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. A reference file of material safety data sheets is available to all personnel involved in these analyses. CAS also maintains a file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

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6.2. Sample extracts and reagents should be handled in accordance with CAS safety policies.

7. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Refer to the determinative method or SOP.

8. RESPONSIBILITIES

8.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

8.2. It is the responsibility or the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the determinative method, is also the responsibility of the department supervisor/manager.

9. PROCEDURE

9.1. Removal of sulfur in extracts

9.1.1. Concentrate the sample to exactly 1.0 mL or other known volume. Perform concentration using the techniques described in the appropriate 3500 series method.

CAUTION: When the volume of solvent is reduced below 1 mL, semivolatile analytes may be lost.

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9.1.2. If the sulfur concentration is such that crystallization occurs, centrifuge to settle the crystals, and carefully draw off the sample extract with a disposable pipet leaving the excess sulfur in the concentration vessel. Transfer 1.0 mL of the extract to a centrifuge tube.

9.1.3. Add approximately 2 g of cleaned copper powder to the centrifuge tube. Vigorously mix the extract and the copper powder for at least 15 minutes on the vortex mixer.

9.1.4. Allow the phases to separate. Separate the extract from the copper by drawing off the extract with a disposable pipet and transfer to a clean vial. The volume remaining still represents 1.0 mL of extract.

COPY

NOTE: This separation is necessary to prevent further degradation of the pesticides.

9.2. Removal of sulfur in soils/sediments

9.2.1. If sulfur is suspected to be present in a sample, the excess sulfur may be removed by mixing 1g of cleaned copper for each 1g of soil. Weigh the sample into a beaker. After the soil has been dried, mix the cleaned copper with the sample. Proceed with the appropriate extraction method.

9.2.2. Alternatively, when extracting by method 3545 (ASE) the sulfur can be removed from the extract by adding copper to the extract collection vessel.

9.2.3. This procedure has been verified for 8082 PCB analysis only.

10. QUALITY CONTROL

Refer to the determinative method or SOP.

11. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

12. WASTE MANAGEMENT

The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the laboratory Safety Manual, Sections 6 and 7.

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13. REFERENCES

Sulfur Cleanup, Method 3660B, U.S. EPA Test Methods for Evaluating Solid Waste, SW-846, Update III, December, 1996.

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STANDARD OPERATING PROCEDURE

SULFURIC ACID CLEANUP

EXT-3665

Revision 2

April 30, 2007

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Approved by: _____ Supervisor — 5/1/07 Date

_____ QA Manager — 5-1-07 Date

_____ Laboratory Manager — 5/1/07 Date

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SULFURIC ACID CLEANUP

1. SCOPE AND APPLICATION

1.1. This procedure is used to perform cleanup of sample extracts prior to analysis for polychlorinated biphenyls (PCBs), using EPA Method 3665A. This procedure may be used when a sample matrix giving a complex chromatogram prevents accurate quantitation of PCBs. The cleanup is applicable to extracts from all sample matrices. This procedure should not be applied to cleanup of extracts for other target analytes, as it will destroy most organic chemicals including the most organochlorine pesticides.

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1.2. This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2. SUMMARY OF METHOD

The extract (in hexane) is sequentially treated with portions of concentrated sulfuric acid. Appropriate caution must be taken when using concentrated sulfuric acid. Blanks and replicate analysis samples must be subjected to the same cleanup as the samples associated with them. If not in hexane already, it is important that all the extracts be exchanged to hexane before initiating the following treatments.

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3. INTERFERENCES

This technique will not destroy chlorinated benzenes, chlorinated naphthalenes (Halowaxes), and some chlorinated pesticides. Care must be taken when interpreting chromatograms where these components may be present.

4. SAFETY

4.1. The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. A reference file of material safety data sheets is available to all personnel involved in these analyses. CAS also maintains a file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

4.2. Sample extracts and reagents should be handled in accordance with CAS safety policies. An acid-proof apron, gloves, and eye protection must be worn. Also, see the **CAUTION**s included for some steps.

5. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

Refer to the determinative method or SOP.

6. REAGENTS

6.1. Pesticide grade solvents should be used in all steps of the procedure. Other grades may be used, provided it is shown that the reagent is of sufficient purity to permit its use without adversely impacting the accuracy of the determination.

6.2. Concentrated Sulfuric acid.

6.3. Hexane, C_6H_{14} - Pesticide grade or equivalent.

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7. APPARATUS

7.1. Syringes, Pastuer pipets, or Class A volumetric pipets, 1.0, 2.0 and 5.0 mL sizes.

7.2. Vials - 1, 2 and 10 mL, glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

7.3. Vortex mixer.

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8. RESPONSIBILITIES

8.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

8.2. It is the responsibility or the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the determinative method, is also the responsibility of the department supervisor/manager.

9. PROCEDURE

9.1. After the extract is brought to the desired final volume, transfer 1.0 or 2.0 mL of the hexane extract to a 10 mL vial and carefully add an equivalent volume of concentrated sulfuric acid. This should be carried out in a fume hood. The volume of hexane extract used depends on the requirements of the GC autosampler used for the analysis.

CAUTION: Make sure that there is no exothermic reaction nor evolution of gas prior to proceeding.

9.2. Cap the vial tightly and vortex for one minute. A vortex must be visible in the vial.

CAUTION: Stop the vortexing immediately if the vial leaks. AVOID SKIN CONTACT, SULFURIC ACID BURNS.

9.3. Allow the phases to separate for at least 1 minute. All extracts should be centrifuged a minimum of 1 minute to separate phases, and centrifuged longer to if an emulsion exists. Examine the top (hexane) layer; it should not be highly colored nor should it have a visible emulsion or cloudiness.

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9.3.1. If a clean phase separation is achieved, proceed to Sec.9.4.

9.3.2. If the hexane layer is colored or the emulsion persists after the first acid cleanup, transfer the extract and let sit on 0.5g Florisil overnight. Dispose of the acid layer properly.

9.4. Transfer the hexane layer to a clean 2 mL autosampler vial and cap. Take care not to include any of the acid layer in this clean vial, as it can cause damage to the instrumentation.

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10. QUALITY CONTROL

Refer to the determinative method or SOP.

11. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

12. WASTE MANAGEMENT

The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the laboratory Safety Manual, Sections 6 and 7.

13. REFERENCES

U.S. EPA Test Methods for Evaluating Solid Waste, SW-846, Update II, September 1994, Method 3665, Revision 0.

U.S. EPA Test Methods for Evaluating Solid Waste, SW-846, Update III, December 1996, Method 3665A, Revision 1.

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STANDARD OPERATING PROCEDURE

CARBON COLUMN CLEANUP
EXT-CARCU

Revision 1

September 1, 2009

UNCONTROLLED COPY

Approved By: _____ Supervisor — 9/1/09 Date

_____ QA Manager — 9/1/09 Date

_____ Laboratory Manager — 9/1/09 Date

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Standard Operating Procedure

for

CARBON COLUMN CLEANUP

1. SCOPE AND APPLICATION

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This Standard Operating Procedure (SOP) describes the procedure used for the clean up of Organochlorine Pesticides, Organophosphorus Pesticides and Polychlorinated Biphenyls (PCB) extracts using carbon cartridges. Carbon cleanup is a convenient way to eliminate compounds that interfere with gas chromatographic analysis of certain target analytes. The correct cleanup method will depend upon the nature of the interfering compounds.

This cleanup may be done on extracts of water, soil, sediment, or tissue samples. This cleanup is applicable only to method 608, 8081, 8141 and 8082-Aroclor extracts.

2. METHOD SUMMARY

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This procedure is based on Restek Applications Note 59103. Refer to Attachment A.

The extracts to be analyzed are passed through a SPE cartridge packed with carbon using a positive pressure or vacuum manifold. The cartridges are rinsed then the sample eluted with DCM:Hexane (20:80), leaving the interferences on the carbon.

3. DEFINITIONS

Method Blank (MB) - The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

4. INTERFERENCES

Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and or elevated baselines causing misinterpretation of chromatograms. All of these materials must be demonstrated to be free from interferences by analyzing method blanks or reagent blanks.

5. SAFETY

5.1. Follow all CAS safety practices as described in the CAS Safety Manual.

5.2. Each chemical compound or reagent should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level. A reference file of material safety data sheets is available to all personnel involved in these analyses. CAS also maintains a file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

Refer to the applicable section in the analytical method or SOP for collection, preservation, and holding times.

7. REAGENTS AND STANDARDS

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7.1. Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination.

7.2. Solvents- Dichloromethane and Hexane, pesticide grade or equilivant.

8. EQUIPMENT AND SUPPLIES

8.1. Solid Phase Extraction

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8.1.1. Resprep CarboPrep 90 250mg/3mL or equivalent. New suppliers must be tested before using.

8.1.2. Vacuum or positive pressure manifold with flow regulator for solid phase extraction cartridges.

8.2. Disposable pipets.

8.3. Vials - 1, 2 and 10 mL, glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

9. PREVENTIVE MAINTENANCE

Routine cleaning of the apparatus used is necessary, especially parts potentially exposed to contact with samples.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are

STANDARD OPERATING PROCEDURE

TOTAL SOLIDS
GEN-160.3
Revision 11
April 10, 2007

UNCONTROLLED

Approved By: _____ Supervisor — 4/12/07 Date

_____ QA Manager — 4-13-07 Date

_____ Laboratory Manager — 4/13/07 Date

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TOTAL SOLIDS

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine total solids using Standard Methods 2540B and EPA method 160.3 and is applicable to drinking, surface, and saline waters, domestic and industrial wastes. This procedure can also be used to determine percent dry solids in soil, sediment, and solids. Total solids for Puget Sound Estuary Program analyses can also be determined using this procedure.

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1.2. The practical range of the determination is from 5 mg/L to 20,000 mg/L in water. The Method Detection Limit (MDL) is 5 mg/L using 200 mL of sample.

1.3. For other sample matrices treated on a weight/weight basis, the working range can be as low as 2 - 100% dry solids. However, samples with less than 5-10 % solids are generally treated as water samples.

2. SUMMARY OF METHOD

A well mixed aliquot of the sample is quantitatively transferred to a pre-weighed evaporating dish and evaporated to dryness at 103-105°C. Results are reported in mg/L for water. For non-aqueous samples, a nominal 10g portion of the sample is used. Results are reported in Percent (%) solids for soils, sediments, or solids.

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3. DEFINITIONS

3.1. Total residue is defined as the sum of the homogenous suspended and dissolved materials in a sample.

3.2. Dry solids is defined as the amount of solid remaining after evaporating off all liquid contained within the sample.

4. INTERFERENCES

4.1. For water samples, non-representative particulates such as leaves, sticks, fish and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result. However, for other matrices, these particles may be included if they are considered representative of the material undergoing other associated analyses.

4.2. Samples containing HF will react with the porcelain crucibles causing a significant weight

weight loss and artificially high results. When analyzing samples containing HF special teflon crucibles should be used and samples should be evaporated in a ventilation hood to dryness.

4.3. Floating oil and grease, if present, should be included in the sample and dispersed by a blender device before aliquoting.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

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5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. Since this is often the first test performed on solid samples, the analyst should use caution in opening containers. For non-routine matrices, based on the appearance of the sample, the analyst should immediately determine if a potential hazard exists.

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6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Sample bottles should be plastic and must be thoroughly cleaned and rinsed prior to use. Soil samples may be collected in glass jars, sleeves, or other suitable container.

6.2. A minimum of 100 mL of sample should be collected for water samples. For soil samples, a minimum of 10g is required. Collecting 8 oz. jars of soil improves subsampling homogeneity.

6.3. Samples must be stored refrigerated at 4 $\pm$2°C and analyzed within 7 days from date of sample collection for water samples. There is no holding time established for soils.

7. APPARATUS

7.1. Evaporating dishes: dishes of 100 ml capacity made of porcelain.

7.2. Evaporating pans, aluminum

7.3. Desiccators, containing desiccant.

7.4. Drying oven, for operation at 103-105°C, and thermometer. The thermometer is immersed in sand, or other suitable solid material, in a flask in the oven.

7.5. Analytical balance capable of weighing to 0.1 mg.

7.6. Glass cylinders.

7.7. Balance calibration verification weights, ASTM Class 1; 1g, 10g, 100g.

8. RESPONSIBILITIES

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8.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

8.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency is also the responsibility of the department supervisor/manager.

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9. PREVENTIVE MAINTENANCE

9.1. Oven thermometer calibration is performed quarterly and multi-point balance calibration checks are performed daily for each day analyses are performed. The results of these checks are recorded in the appropriate QA metrological logbook.

9.2. Periodic balance service is performed by an outside service provider and is coordinated by the QA section.

9.3. Analytical balances should be kept clean and free of debris. The balance bubble indicator should be checked to ensure the unit is level.

9.4. Desiccant should be changed as needed.

10. PROCEDURE

10.1. Water samples

10.1.1. Heat the clean evaporating dish to 103-105°C for a minimum of one hour. Cool, and desiccate. Weigh and store in desiccator until ready for use.

10.1.2. Transfer a measured aliquot of sample to the pre-weighed dish and evaporate to dryness on a steam bath or in a drying oven.

10.1.3. Choose an aliquot of sample sufficient to contain a residue of at least 25 mg. To obtain a measurable residue, successive aliquots of sample may be added to the same dish.

10.1.4. If evaporation is performed in a drying oven, the temperature should be lowered to approximately 98°C to prevent boiling and splattering of the sample.

10.1.5. Dry the evaporated sample for at least 1 hour at 103-105°C. Cool in a desiccator and weigh. Repeat the cycle of drying at 103-105°C, cooling, desiccating and weighing until a constant weight is obtained or until loss of weight is less than 4% of the previous weight, or 0.5 mg, whichever is less. Report the results of this weighing.

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10.2. Soils, sediments, and solids

10.2.1. Pre-dry the aluminum pans prior to use by heating at 103-105°C for one hour. Allow to cool. Label the pans with numbers corresponding to the sample I.D.s analyzed.

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10.2.2. After opening the sample jar, mix the sample to achieve a homogenous sample.

10.2.3. Determine the tare (dry pan) weight, then weigh 10 grams of sample into the pan.

10.2.4. Place in a drying oven overnight at 103-105°C.

10.2.5. Remove from the oven and let cool. Since percent level determinations are being made on solid samples, cooling in a desiccator is not necessary. Weigh and record the dry (pan+sample) weight.

11. QA/QC REQUIREMENTS

11.1. Prior to, and after each analysis batch, balance calibration verification is performed using weights bracketing the sample weights. Balance calibration verification measured weights must be ± 0.5% of the true value.

11.2. QC Samples Required

11.2.1. For water samples, run a laboratory control sample (LCS) per batch of 20 (or fewer) samples. For the LCS, a certified quality control standard is purchased from APG as a solid material. Add the standard to DI water in a 1L volumetric flack and dilute to volume (for APG, add all of the material provided). The LCS will be approximately 1000 mg/L, with exact values specified by lot number. Analyze as described above. The LCS recovery criteria is 92-106% of the true value. This statistically derived acceptance limit is subject to change as limits are updated.

11.2.2. Run one duplicate per batch of ten samples. The RPD should be $\leq$ 10%. This statistically derived acceptance limit is subject to change as limits are updated. For Puget Sound Estuary Program protocols, perform a triplicate analysis per batch of 10 samples. The RSD should be $\leq$20%.

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11.2.3. For water samples, run a method blank every twenty samples or one per desiccator.

12. DATA REDUCTION, REVIEW, AND REPORTING

12.1. For water samples, calculate total residue as follows:

$$\textit{Total residue, mg / L} = \frac{(A - B) \times 1{,}000}{C}$$

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where:

A = weight of sample + dish in mg
B = weight of dish in mg
C = volume of sample in ml

12.2. For soils, sediments, and solids, calculate % solids as follows:

(tare + dry weight) - tare = dry weight

dry weight ÷ wet weight x 100 = % solids

12.3. Record all measurements on a benchsheet or spreadsheet. For soils, sediments, and solids, a spreadsheet is available to calculate % solids. This is used in conjunction with a system where the analytical balance is interfaced with a desktop computer. Use the Print button on the balance to transfer each reading to the PC. From this, a spreadsheet is used to calculate % solids.

12.4. Results are reported directly from benchsheets or spreadsheets. Use the appropriate form template in R:\WET or R:\TSOLIDS directories.

12.5. Report water results in mg/L total solids using whole numbers. The Method Reporting Limit is 5 mg/L. Report non-aqueous samples as % Solids by 160.3Modified. Use up to a maximum of three significant figures.

12.6. Data review

12.6.1. It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. All data will be initialed, dated and attached to required data quality worksheet.

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12.6.2. The data packet for the sequence is submitted for review by supervisor or designee.

12.6.3. Refer to the SOP for *Laboratory Data Review Process* for general instructions for data review.

13. CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the SOP for *Nonconformity and Corrective Action* for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for *Report Generation* or in project-specific requirements.

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14. METHOD PERFORMANCE

14.1. Refer to the reference method for additional method performance data available.

14.2. The method detection limit (MDL) is established using the procedure described in the SOP for *The Determination of Method Detection Limits and Limits of Detection* (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

15. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when recycled or disposed of properly.

16. WASTE MANAGEMENT

The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17. TRAINING

17.1. Refer to the SOP for *Documentation of Training*. The SOP describes the training outline and necessary documentation.

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17.2. Review literature (see References section). Review the SOP. Also review safety procedures. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

17.3. The next training step is to assist in the procedure under the guidance of an experienced analyst for a period of 1-2 months. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

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17.4. Independently perform Initial Demonstration of Proficiency studies and QC analyses. The data must be reviewed by a supervisor or trained analyst. Documentation is forwarded to the employee's training file.

18. REFERENCES

18.1. Total solids dried at 103-105°C, Method 2540B in Standard Methods for the Examination of Water and Wastewater, 20th Ed., 1998.

18.2. Residue, Total, Method 160.3 EPA 600/4-79-020, Revised March 1983.

APPENDIX A

Benchsheet

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Work Order #.: _____ Method: EPA 160.3

Analysis: Total Solids

Sample Number							
Crucible Number							
Conductivity							
Sample Volume (mL)							
Wt. Cru. + Dry Sample (1)							
Wt. Crucible. (g)							
Wt. Dry Sample (g)							
Total Solids (mg/L)							

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Sample Number							
Crucible Number							
Conductivity							
Sample Volume (mL)							
Wt. Cru. + Dry Sample (1)							
Wt. Crucible. (g)							
Wt. Dry Sample (g)							
Total Solids (mg/L)							

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Calculation: Dissolved Solids (mg/L) = Wt. Dry Sample (g) x 1000 x 1000 / Volume (ml)

APG #: Lot #: ID#: T.V. = % Rec =

Comments:

Mean = RPD =

Time Start	
Stop	
Temp Start	
Stop	

Time Start	
Stop	
Temp Start	
Stop	

Balance #

Analyzed By:	Date Prepared:
Reviewed By:	Date Reviewed:

COLUMBIA ANALYTICAL SERVICES, INC.

Service Request #: _____ Method: EPA METHOD 160.3 MODIFIED

Analysis For: _____

Pan ID	Sample Name	Wet Weight (g)	Tare (g)	Tare - Dry Wt.(g)	Dry Weight (g)	% Total Solids

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Oven Temp : _____ Time In : _____ Time Out : _____

Comments:

$\bar{x}$ RPD =

$\bar{x}$ RPD =

$\bar{x}$ RPD =

Analyst:	Date:
Reviewed By:	Date:

TSGEN.SPD

STANDARD OPERATING PROCEDURE

CARBON, TOTAL ORGANIC IN SOIL

GEN-ASTM
Revision 6
October 18, 2007

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Approved By: _____ Supervisor — 10/18/07 Date

_____ QA Manager — 10-18-07 Date

_____ Laboratory Manager — 10/18/07 Date

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CARBON, TOTAL ORGANIC IN SOIL

1. SCOPE AND APPLICATION

1.1. This procedure is applicable to the determination of Total Organic Carbon (TOC) using ASTM method D4129-82, modified for soil and sediment matrices (Puget Sound Estuary Program and Lloyd Kahn). Total organic carbon is a measure of the total amount nonvolatile, partially volatile and particulate organic compounds in a sample. Sample should be treated to remove inorganic carbon (carbonates, bicarbonates, free CO_2 etc.), prior to analysis, as these compounds will interfere with true readings.

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1.2. This method is applicable to all soils and sediments and most matrices that can be dried and shatter-boxed to a fine powder.

1.3. Results are reported as percent (%) carbon, and the applicable range is the MDL - 100%. The Method Reporting Limit (MRL) for TOC on soils is 0.05%, dry weight basis. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The Method Detection Limit (MDL) has been determined at 0.02%.

2. METHOD SUMMARY

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2.1. Samples are combusted in an oxygen atmosphere to convert organic and inorganic forms of carbon to CO_2. The combustion temperature is selected to completely oxidize all carbon forms. The combustion product gases are swept through a barium chromate catalyst/scrubber to ensure that all of the carbon is oxidized to CO_2. Other potentially interfering product gases such as SO_2, SO_3, HX, and NO_x are removed from the gas stream in a series of chemical scrubbers. The CO_2 is then swept to the coulometer where it is detected by automatic, coulometric titration, with coulometric end point indication.

2.2. The coulometer cell is filled with a partially aqueous medium containing ethanolamine and a colorimetric indicator. When a gas stream passes through the solution, CO_2 is quantitatively absorbed. CO_2 reacts with the ethanolamine to form a strong titratable acid which caused the indicator to fade. The titration current automatically turns on and electrically generates base to return the solution to its original color.

3. DEFINITIONS

3.1. Analysis Batch - Samples are analyzed in a set referred to as an analysis batch. The batch begins with calibration/standardization followed by QC analyses and samples. The batch ends when the QC analyses and set of samples has been completed.

3.2. Method Blank - The method blank is an artificial sample (empty boat) designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.3. Laboratory Control Sample (LCS) - A standard of known TOC concentration which is used to ensure that the analysis produces an accurate measurement of TOC in samples analyzed in the batch.

4. INTERFERENCES

4.1. Acidic and other gases, including SO_2, SO_3, H_2S, HCl, HBr, HI, Cl_2, and NO_x can be effectively removed using scrubbers such as KI, Ag_2SO_4, $AgNo_3$, and MnO_2.

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4.2. Volatile organics may be lost in the decarbonization process.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

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5.3. Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

5.4. Disconnect teflon tubing from furnace at check valve whenever system is not in use or when O_2 flow is turned off or furnace temperature is reduced. If the carbon cathode solution should be siphoned through a failed check valve into the magnesium perchlorate scrubber potentially explosive DMSO-perchlorate could be formed.

5.5. Do not attempt to combust large samples of organic or other materials that will react with pure oxygen. Such samples can cause the pyrolysis tube to explode.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

Samples can be collected in glass or plastic containers. Samples are preserved by storage at 4±2°C. Samples are analyzed within 28 days of collection.

7. APPARATUS AND EQUIPMENT

7.1. Induction furnace, Coulometrics Incorporated.

7.2. Analytical balance, 0.1mg accuracy.

7.3. Desiccator.

7.4. Quartz combustion boats.

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7.5. Sample scoop.

7.6. Porcelain dishes.

7.7. Glass ladles and miscellaneous laboratory glassware,

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Standards

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8.1.1. Urea - 20% carbon. Use 10 μg.

8.1.2. Nutrients in Soil, purchased standard with a known TOC value (typically ERA #542). Use 50 mg for LCS.

8.2. Reagents

8.2.1. Hydrochloric acid, 50% and 10%.

8.2.2. Carbon Cathode Solution. Dimethyl Sulfoxide; DMSO. Purchased from Coulometrics Inc. as a prepared solution. Used for coulometer solution.

8.2.3. Anode Solution. Dimethyl Sulfoxide and potassium iodide. Purchased from Coulometrics Inc. as prepared solution.

8.2.4. Manganese dioxide. Gas scrubber solution.

8.2.5. Potassium Hydroxide. Gas scrubber solution.

8.2.6. Potassium Iodide. Anode chemical.

8.2.7. Magnesium Perchlorate desiccant

9. RESPONSIBILITIES

9.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

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9.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the *SOP for Documentation of Training*, is also the responsibility of the department supervisor/manager.

10. PREVENTIVE MAINTENANCE

Maintenance is performed as follows:

Maintenance Item	Frequency
Cell	Clean daily with methanol and water to clean frit
Mg Perchlorate Scrubber	change daily
KOH Scrubber	change monthly
NOX scrubber	change as needed
Repack Precombustion Column	as needed
Repack Combustion Column	as needed

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11. PROCEDURE

11.1. Sample Preparation.

11.1.1. Turn furnace on to #5 (≈1000°C). Allow furnace to warm-up for about 1/2 hours. Turn on oxygen to ≈ 5 psi and 75 to 125 ml/min at flowmeter.

11.1.2. Clean quartz boats. Scrape out old sample and rinse boats with DI water. Place boats in crucible and muffle for at least 10-15 minutes. Remove boats and place in desiccator until ready for use.

11.1.3. Samples should be dried at 70°C and homogenized prior to analysis.

11.1.4. As a rule, the darker (or closer to black) a sample is, the more carbon it contains. Place a small portion of sample on a watch glass. Add 1 drop of 10% HCl. Watch for effervescence or bubbling. If bubbles are present, the sample contains inorganic carbon (CO_3). If sample bubbles, reduce sample size to prevent sample from bubbling out of boat. If sample is dark, wood product or sludge reduce sample volume to 5 → 10mg. Normal sample volume = 50mg. After boats are loaded with sample add 1 to 2 drops 10% HCl. Place boats in 70°C oven to dry. If samples bubbled when acid was added, add 1 to 2 drops more acid and dry at 70°C. Continue acidifying and drying until samples no longer bubble. Place samples in desiccator until ready for analysis.

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11.2. Apparatus Preparation.

11.2.1. Fill cell with carbon cathode solution to 100 → 125 ml, drop in stir bar. Place cell top on snug.

11.2.2. Cover bottom of anode cell with KI. About 2 small scoops.

11.2.3. Add carbon anode solution to cell such that when anode is inserted in the anode cell, the anode solution level is the same as the cathode solution level.

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11.2.4. Place cell in coulometer cell holder.

11.2.5. Turn on detector lamp and stir plate. (Power on)

11.2.6. Turn adjust knob to 122 (all the way to the right) then turn back down to 100. Rotate cell until maximum transmittance is obtained.

11.2.7. With oxygen bubbling to cell and maximum transmittance obtained, turn on the current to the anode and cathode. The carbon cathode solution will begin to titrate to a blue color.

11.2.8. Change Magnesium Perchlorate desiccant daily.

11.2.9. The instrument is now ready to run.

11.3. Calibration and Standardization.

11.3.1. Burn both ladles for five minutes each to remove any residual TOC.

11.3.2. Establish baseline.

11.3.2.1. After placing ladles in sample inlet, allow system to purge for 1 minute.

11.3.2.2. Burn three boats empty five minutes each. The average of the three runs is the baseline.

11.4. Analysis.

11.4.1. Place one platinum or quartz boat in a ladle. Place the ladle in the sample inlet and purge for 1 minute. Simultaneously insert the sample into the furnace, press the reset button on the coulometer and start the timer for five minutes.

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11.4.2. After five minutes, obtain a reading from the instrument. Remove the ladle from the furnace. (Occasionally, a high sample may require longer than 5 minutes to complete the titration).

11.4.3. Load the other ladle with the next platinum (or quartz) boat. Remove the ladle in use from the inlet port and insert the next ladle.

11.4.4. Repeat steps 10.4.1 through 10.4.3 until all samples are analyzed.

12. QA/QC REQUIREMENTS

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12.1. Initial Precision and Recovery Validation

The precision and accuracy of the procedure must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made. To do this, four LCS's are prepared and analyzed. The RSD should be <20% and average recovery must be 85-115%.

12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Analyze a minimum of seven spiked blank replicates at a level near the MRL. Follow the procedures starting in Section 11 to analyze the samples. Refer to the CAS *SOP for The Determination of Method Detection Limits and Limits of Detection*.

12.2.2. Calculate the average concentration found (x) in the *sample concentration*, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study should be done annually.

12.3. Ongoing QC Samples required are described below and in the *SOP for Sample Batches*. Project-specific batching protocols may also be required.

12.3.1. LCS - An LCS must be analyzed with each batch of 20 or fewer samples. Analyze 50mg of the purchased standard (see 8.1.2) is used. The acceptance criteria for this LCS is ± 15% of the true value.

12.3.2. Method Blank - Burn one empty boat per batch of 20 or fewer samples. Method Blank must be <0.05% carbon.

12.3.3. CCV (Continuing Calibration Verification) - A CCV must be analyzed every tenth analysis. Analyze ~10mg urea. The CCV must be 18.0% - 22.0% carbon.

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12.3.4. CCB (Continuing Calibration Blank) - A CCB must be analyzed following every CCV.

12.3.5. Sample duplicate - One sample per batch of 20 or fewer samples must be analyzed in duplicate. Duplicates should be 20% RPD, if > five times the MRL.

12.3.6. Matrix Spike - One spike must be analyzed with each batch of 20 or fewer samples. The acidified sample will be spiked with a known amount of urea.

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12.3.7. See Table 1 for a summary of acceptance criteria and corrective actions.

13. DATA REDUCTION AND REPORTING

13.1. Calculate % carbon as follows:

$$\%Carbon = \frac{(Gross\ reading\ - baseline\ \mu g)(0.1)}{mg\ sample\ analyzed}$$

13.2. For duplicate analyses, calculate relative percent difference as follows:

$$RPD = \frac{S_1 - S_2}{Avg} * 100$$

where S1 = Sample with higher value
S2 = Sample with lower value
Avg = Average of the two sample values

13.3. Calculate percent recovery as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

13.4. It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified above. Average, RPD, spike level and spike recovery are entered on spreadsheet for corresponding samples. All data will be initialed, dated and attached to required data quality worksheet.

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13.5. The data packet for the sequence is submitted for review by supervisor or designee. The results are transferred to the appropriate report form located in the CAS network directory R:\WET\WIP. These forms are made from templates located in R:\WET\FORMS.

13.6. Refer to the *SOP for Laboratory Data Review Process* for general guidelines for data review.

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13.7. Reporting

13.7.1. Total organic carbon is reported as % carbon, normally on a dry weight basis. Results may be reported on an as received basis.

13.7.2. The Method Reporting Limit is 0.05% carbon, on a dry weight basis.

13.7.3. Report all results to three significant figures.

13.7.4. Bench sheets are labeled "Total Organic Carbon, TOC". These benchsheets, located in Appendix I, should be in use at all times during TOC analysis.

14. CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the SOP for Nonconformity and Corrective Action for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for Report Generation or in project-specific requirements.

15. METHOD PERFORMANCE

15.1. This method is validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2. The method detection limit (MDL) is established using the procedure described in the SOP for The Determination of Method Detection Limits (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16. POLLUTION PREVENTION

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It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

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17.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the CAS EH&S Manual for details.

17.3. This method uses a base. Waste base is hazardous to the sewer system and to the environment. All waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the CAS EH&S Manual for details.

18. REFERENCES

18.1. Coulometrics Inc. Instruction Manual, Model 5020.

18.2. EPA Method Modified 415.1.

18.3. Total Organic Carbon, Method 9060, EPASW846, Test Methods For Evaluating Solid Waste, Third Edition, September 1986, Revision 0.

18.4. Total Organic Carbon(TOC), Conventional Sediment Variables, Puget Sound Estuary Program, March 1986.

18.5. Determination of Total Organic Carbon in Sediment, Lloyd and Kahn, U.S.E.P.A Region II, July 1998.

18.6. ASTM Method D4129-88

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TABLE 1

Summary of Corrective Actions

Method Reference	Analysis	Control Item	Acceptance Criteria	Corrective Action
ASTM Method D4129-82	TOC (Soil)	Urea	±10%	Re-analyze all samples affected.
		Benzoic Acid	± 15%	Re-Analyze.
		Method Blank	<0.05%	Re-analyze. If still high, clean boats and start over.
		Sample Duplicate	20% RPD	Analyze a triplicate. Homogenize again and reanalyze.
		Sample Spike	75-125%	Re-analyze.

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APPENDIX I

BENCHSHEETS

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Columbia Analytical Services, Inc.

Service Request #: _____ Method: ASTM D4129-82 (Combustion/Coulometric)

Analysis For: Total Organic Carbon (TOC) Matrix: Soil / Dry Weight Basis

Sample Number	mg Sample Injected	Sample Reading, µg C	Baseline Reading, µg C	Net µg C	% Carbon

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Acid Purge Time: 1 minute **Reading Time:** 5 minutes **TOC % =** $\frac{\text{(Net Reading)(µg 0.1)}}{\text{mg Sample Injected}}$

CCV : Urea Fisher (lot #0953513) ID#: TOCS/1-10-B TV = 20.0%C

CCV1 =97 CCV2 =

LCS: ERA Cat#: 542 Lot#: 1102 ID#: TOCS/1-10-D TV = 1.16%C REC%=

Comments :

X= RSD= SPK%REC=

Analyzed By:	Date:
Reveiwed By:	Date:

TOC.XLT

Columbia Analytical Services, Inc.

Service Request #: _____ Method: ASTM D4129-82 (Combustion/Coulometric)

Analysis For: Total Organic Carbon (TOC) Matrix: Soil / Dry Weight Basis

Sample Number	mg Sample Injected	Sample Reading, µg C	Baseline Reading, µg C	Net µg C	% Carbon

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Acid Purge Time: 1 minute **Reading Time:** 5 minutes **TOC % =** $\frac{\text{(Net Reading)(µg 0.1)}}{\text{mg Sample Injected}}$

CCV : Urea Fisher (lot #0953513) ID#: TOCS/1-10-B TV = 20.0%C

LCS: ERA Cat#: 542 Lot#: 1102 ID#: TOCS/1-10-D TV = 1.16%C % Rec =

Comments :

Analyzed By:	Date:
Reveiwed By:	Date:

TOC Soil Benchsheet

Sample #	mg Sample	Reading	Date Baked	Baseline
				AVG.

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TOC Soil Benchsheet

Sample #	mg Sample	Reading	Date Baked	Baseline
CCV-1				
CCB-1				
LCS				
MB				Avg
CCV-2				
CCB-2				
CCV-3				
CCB-3				
CCV-4				
CCB-4				
CCV-5				
CCB-5				

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Columbia Analytical Services, Inc.

Service Request #: K7112 / K7040 / K7669 Method: 9060s / PSEP (Combustion/Coulometric)

Analysis For: Total Organic Carbon (TOC) Matrix: Soil / Dry Weight Basis

Sample Number	mg Sample Injected	Sample Reading, µg C	Baseline Reading, µg C	Net µg C	% Carbon
CCV-1	6.8	1328.3	7.3	1321.0	19.4
CCB-1	50.0	6.8	7.3	-0.5	< 0.05
LCS-1	47.4	198.4	7.3	191.1	0.40
MB-1	50.0	7.2	7.3	-0.1	< 0.05
LCS-2	47.8	201.1	7.3	193.8	0.41
LCS-3	56.1	247.8	7.3	240.5	0.43
LCS-4	46.1	238.5	7.3	231.2	0.50
LCS-5	47.5	196.5	7.3	189.2	0.40
LCS-6	46.2	194.2	7.3	186.9	0.40
LCS-7	47.0	200.7	7.3	193.4	0.41
LCS-8	46.0	214.9	7.3	207.6	0.45
LCS-9	47.1	194.8	7.3	187.5	0.40
LCS-10	45.6	203.5	7.3	196.2	0.43
CCV2	10.7	2074.1	7.3	2066.8	19.3
CCB-2	50.0	9.3	7.3	2.0	< 0.05

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Acid Purge Time: 1 minute **Reading Time:** 5 minutes **TOC % =** (Net Reading)(µg 0.1) / mg Sample Injected

CCV : Urea Baker (lot #A17584) ID#: TOCS/1-10-J TV = 20.0%C

CCV1 = 97 CCV2 = 97

LCS: ERA Cat#: 542 Lot#: D060-542 ID#:PO3/2-26E TV = 0.34%C

Comments :

LCS 1 to 10 $\bar{x}$ = 0.42 RSD = 8%

ERA QC Performance Acceptance Limits for TOC are 436 to 6370 mg/kg

MATERIAL STANDARDIZED TO 0.42%. USE AS TV FOR THIS LOT CWF 8-27-08

Analyzed By:		Date: 8/26/08	Time: 0900
Reveiwed By:		Date: 8/27/08	

TOC.XLT

STANDARD OPERATING PROCEDURE

For

pH IN SOIL AND SOLIDS

SOP No.: GEN-PHS

Revision: 10
August 25, 2009

Approved by: _____ Supervisor — 8/31/09 Date

_____ QA Manager — 8/31/09 Date

_____ Laboratory Manager — 8/31/09 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

pH IN SOIL AND SOLIDS

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine pH in soil, solid, and certain waste samples using EPA Method 9045D

1.2. When used to determine pH in multiphase wastes, the procedure is applicable if the aqueous phase constitutes less than 20% of the total volume of the waste.

2. METHOD SUMMARY

2.1. The pH is determined by potentiometric measurement of a soil slurry or aqueous solution using a standard combination glass pH electrode and pH meter.

2.2. The procedure uses methodology described in EPA Method 9045D, WDOE Test Method, and Oregon State Soil Methods.

3. DEFINITIONS

3.1. Analysis Batch - A sequence of samples, which are analyzed within a 24-hour period and include no more than 20 field samples.

3.2. Sample Duplicate - two aliquots of the same sample that are treated exactly the same throughout laboratory analytical procedures. The purpose is to verify the precision associated with the laboratory procedures.

3.3. Sample Triplicate – three aliquots of the same sample that are treated exactly the same throughout laboratory analytical procedures. The purpose is to verify the precision associated with the laboratory procedures.

4. INTERFERENCES

4.1. Samples with very low or very high pH may give incorrect readings on the meter. Samples with a high sodium concentration and pH > 10 can cause error. Reduce this error by using a "low sodium error" electrode. Strong acid solutions with pH < 1 may give incorrect high pH readings.

4.2. Samples containing oil may coat the electrode and cause a sluggish response or inaccurate reading. If an electrode becomes coated with a material which cannot be rinsed off, the electrode can be cleaned with an ultrasonic bath, be washed with detergent and rinsed then

placed in 1:1 HCl so that the lower third of the electrode is submerged, then rinsed thoroughly with water.

4.3. Temperature fluctuations will cause instrument errors.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Sample bottles can either be glass or plastic and must be thoroughly cleaned and rinsed prior to use.

6.2. Samples must be stored refrigerated at 4°C (± 2°C). Although there is no holding time established for soils, samples should be analyzed as soon as possible.

7. APPARATUS AND EQUIPMENT

7.1 Fisher Accumet pH meter, model 25.

7.2 Combination electrode for pH with temperature probe.

7.3 Conductivity jars, 50 ml.

7.4 Analytical balance capable of weighing 0.1 g.

7.5 Paint filters.

7.6 Erlenmeyer flasks, 250 ml.

7.7 Water bath capable of maintaining a constant temperature of 25°C. One large for all samples and buffers and one smaller bath for analyzing samples at 25°C ± 1°C.

7.8 Standard stir plate and submersible stir plate and stir bars.

7.9 Eight or 16 ounce juice bottles and caps.

8 STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1 pH buffers: 1.00, 4.00, 7.00, 10.00, 12.45, (true value of buffers at 25° C).

8.2 Commercially available solutions should be validated and traceable to NIST standards and are recommended for routine use.

9 PREVENTIVE MAINTENANCE

9.1 The probe should contain filling solution past the coils to ensure accurate readings. Filling solution should be a non-AgCl containing solution.

9.2 Clean the electrode as described in section 3.2 or as per the manufacturer's instructions.

10 RESPONSIBILITIES

It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

11 PROCEDURE

11.1 Calibration

11.1.1 All buffers are placed in conductivity jars, capped and stored in the large 25° C water bath. All readings need to be within 1°C of the buffer temperatures.

11.1.2 Perform calibration daily. Record calibration; buffer checks and buffer temperatures in instrument logbook or benchsheet with date and analyst's initials.

11.1.3 Calibration (Fisher Accumet pH Meter 25)

11.1.3.1 Push "standardize"

11.1.3.2 Select 2

11.1.3.3 Push "standardize".

11.1.3.4 Select 1

11.1.3.5 Enter the first buffer value, 4.00. Push "enter".

11.1.3.6 Place enough buffer solution in a conductivity jar so that the electrode is sufficiently submersed without coming into contact with the stir bar.

11.1.3.7 Rinse electrode

11.1.3.8 Immerse the electrode in the solution. Allow time to stabilize.

11.1.3.9 Push "enter".

11.1.3.10 Record the buffer value to 0.01 pH units and the buffer temperature to the nearest °C. (4.00 and 25°C).

11.1.3.11 Repeat steps 11.2.2.1 through 11.2.2.9 for buffers 7.00 and 10.00. Also use the 12.45 and 1.00 buffers if needed.

11.1.4 Calibration (Fisher Accumet pH Meter AR25)

Calibrate according to the manufacturer's specifications.

Note: Initial calibration is performed using the 4.00, 7.00, and 10.00 buffers. If any subsequent sample pH is outside the calibration range (greater than 10.00 or less than 4.00), the 1.00 and/or 12.45 buffers are added to the calibration and the applicable samples are reanalyzed.

11.2 Soil samples preparation for EPA Method 9045D.

11.2.1 Weigh out 20g of soil into a beaker. Add 20mL of reagent water, cover, and stir the suspension continuously for 5 minutes. Additional dilutions may be performed if working with hygroscopic soils and salts, or other problematic matrices.

11.2.2 Let the soil suspension stand for 1 hour to allow for settling. Alternatively, filter or centrifuge off the aqueous phase for pH determination.

11.2.3 Setup electrodes in clamps so that when the electrode is lowered into the beaker, the electrode will be immersed just deep enough in the supernatant solution to establish a good electrical contact through the ground-glass joint or fiber capillary hole. Immerse the electrode in samples in this manner.

11.3 Waste material preparation for EPA Method 9045D.

11.3.1 Wastes may be solids, sludges, or non-aqueous liquids. For multi-phase wastes by method 9045D, a determination of the percentage of the sample that is non-

aqueous must be made. This can be calculated from a % solids determination. If the non-aqueous phase is > 20%, continue with this section. If the non-aqueous phase is < 20%, analyze the sample by EPA Method 9040B see also SOP GEN-pHW.

11.3.2 Weigh out 20g of waste sample into a beaker. Add 20mL of reagent water, cover, and stir the suspension continuously for 5 minutes. Additional dilutions may be performed if working with hygroscopic soils and salts, or other problematic matrices.

11.3.3 Let the waste suspension stand for 15 minutes to allow for settling. Alternatively, filter or centrifuge off the aqueous phase for pH determination.

Note: If the waste absorbs all the reagent water, begin the test again with 20g waste and 40mL of water.

Note: If the supernatant is multi-phasic, decant the oily phase and perform the pH determination on the aqueous phase.

11.3.4 Setup electrodes in clamps so that when the electrode is lowered into the beaker, the electrode will be immersed just deep enough in the supernatant solution to establish a good electrical contact through the ground-glass joint or fiber capillary hole. Immerse the electrode in samples in this manner.

11.4 Sample preparation for Washington DOE Test Method.

11.4.1 Weigh three, 50.0g aliquots of each sample into either 3, 8-ounce or 3, 16-ounce juices bottles and add 50mL of D.I. water to each and cap tightly. Each sample is analyzed in triplicate.

11.4.2 Place all bottles on the wrist action shaker. The speed of the shaker should be adjusted so that the sample and water have maximum contact time however the shaking action should not be so vigorous as to cause absorption of CO_2 into the sample.

11.4.3 Filter the liquid through a paint filter into a clean conductivity jar for analysis.

11.5 Oregon State Soil Methods sample preparation

11.5.1 Weigh 20.0g of soil into a beaker and add 40mL of D.I. water.

11.5.2 Stir the suspension 2-3 times over a 30-minute period.

11.5.3 Analyze the supernatant.

11.6 Sample Analysis

11.7.1 Rinse and blot electrode, then immerse into the sample. Press pH and record the pH when stabilized, record the temperature to the nearest °C. Remove electrodes from sample after each measurement and rinse 3 times with D.I. water.

11.7.2 Regardless of the method employed, all pH readings must be within 2°C of the temperature of the buffer solutions.

11.7.3 If the pH of the sample is ≥11.00 control the temperature of the samples to 25°C±1°C.

12 QA/QC REQUIREMENTS

12.1 A buffer check is analyzed after every 10 readings. For buffer checks, use either pH 4.00 or 10.00, choosing whichever standard brackets the majority of the previous samples with pH 7.00. The buffer check should be within 0.05 pH units of the true value.

12.2 A laboratory control sample (LCS) is analyzed at a frequency of one per 20 samples, with acceptance criteria of 85-115% of the true value. Analyze the LCS prior to the sample set. The LCS is prepared identically to associated samples and documented on the benchsheet. If the LCS is outside of these limits, recalibrate.

12.3 A duplicate sample is analyzed at a frequency of 10% of the samples, with acceptance criteria of 10% RPD between the two readings. If the duplicate is outside of these limits, the sample is reanalyzed. Duplicates are documented on the benchsheet. For duplicate analyses, calculate relative percent difference as follows:

$$RPD = \frac{S_1 - S_2}{Avg} * 100$$

Where S1 = Sample with higher value
S2 = Sample with lower value
Avg. = Average of the two sample values

12.4 For DOE/pH, all samples are analyzed in triplicate and the logarithmic average is reported.

12.4.1 Sum the antilog of the three pH readings obtained in section 11.5, divide by 3 then take the log.

Example:
Three pH readings obtained: 1.5 1.6 2.5

antilog(1.5) + antilog(1.6) + antilog(2.5) =
31.62 + 39.81 + 316.23 = 387.66
387.66 ÷ 3 = 129.22
log(129.22) = 2.11
pH(average) = 2.11

13 DATA REDUCTION, REVIEW, AND REPORTING

13.1 All data and corrective actions must be recorded, dated, and signed or initialed by the analyst. It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified above. Average, RPD, and buffer level are entered on the benchsheet for corresponding samples. All data will be initialed, dated and attached to required data quality worksheet.

13.2 The data packet for the sequence is submitted for review by supervisor or designee. The results are transferred to the appropriate report form located in the CAS network directory R:\WET\WIP. These forms are made from templates located in R:\WET\FORMS.

13.3 Refer to the SOP for Laboratory Data Review Process for general guidelines for data review.

13.4 Reporting

13.4.1 The pH is reported as pH units. Values are reported to 0.01 pH units.

13.4.2 The benchsheets, located in Appendix A, should be in use at all times during pH analysis.

14 CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the SOP for *Nonconformity and Corrective Action* for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for *Report Generation* or in project-specific requirements.

15 METHOD PERFORMANCE

This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

16 POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

17 WASTE MANAGEMENT

The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

18 TRAINING

18.1 Training Outline

18.1.1 Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

18.1.2 The next training step is to assist in the procedure under the guidance of an experienced analyst for a period of one week. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

18.1.3 Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

18.2 Training is documented following the SOP for Documentation of Technical Personnel Training.

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

19 CHANGES SINCE THE LAST REVISION

19.1 Sec 1.1 removed 9045c and replaced with 9045D.

19.2 Sec 18 Updated Training Documentation Requirements.

20 REFERENCES

20.1 EPASW846, Test Methods For Evaluating Solid Waste, Third Edition, Update IIIB, August 2002, Method 9045D, Revision 4.

20.2 Method 83-13, State of Washington, Department of Ecology.

20.3 Oregon State University, Methods of Soil Analysis Used in the Soil Testing Laboratory at Oregon State University.

APPENDIX A

Benchsheet

COLUMBIA ANALYTICAL SERVICES, INC.

Service Request #: _____ Method: 9045C

Analysis: pH Corrosivity Matrix: Soil/Solid

Calibration						
Buffer	1.00	4.00	7.00	10.00	12.45	Value of Buffers at 25 °C
Reading						
Temp °C						

Sample Number	Sample Wt. (g)	Vol of Ext Sol (mLs)	pH Reading	% Solids	Temp °C
Buffer Check	**Buffer Value:**				
LCS	--	--			
Buffer Check	**Buffer Value:**				
Buffer Check	**Buffer Value:**				

LCS: APG 4063 **Lot #:** **ID #:** **T. V. =** **% REC. =**

pH 4.00 buffer Cond/1-75-_____ pH 7.00 buffer Cond/1-77-_____ pH 10.00 buffer Cond/1-79-_____

ph 1.00 buffer Cond/1-73-_____pH 12.45 buffer Cond/1-81-_____ Extraction Solution: _____

Comments: _____

Revision: 1a ; 2009

Analyst:	Date:
Reviewed By:	Date:

COLUMBIA ANALYTICAL SERVICES, INC.

Service Request #: Method: 9045C

Analysis: pH Corrosivity Matrix: Soil /Solid

Sample Number	Sample Wt. (g)	Vol of Ext Sol (mLs)	pH	% Solids	Temp °C
Buffer Check	**Buffer Value:**				
Buffer Check	**Buffer Value:**				
Buffer Check	**Buffer Value:**				

Comments:

Revision: 1a ; 2009

Analyst:	Date:
Reviewed By:	Date:

SOILPH

STANDARD OPERATING PROCEDURE

PARTICLE SIZE DETERMINATION – ASTM PROCEDURE
GEN-PSASTM
Revision 1
November 20, 2007

UNCONTROLLED COPY

Approved By: _____ Supervisor — 11/20/07 Date

_____ QA Manager — 11-20-07 Date

_____ Laboratory Manager — 11/20/07 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL
NUMBER: _____
Initials: _____ Date: _____

PARTICLE SIZE DETERMINATION – ASTM PROCEDURE

1. SCOPE AND APPLICATION

1.1. This procedure covers the quantitative determination of the distribution of particle sizes in soils as described in ASTM D 422-63. The distribution of particle sizes larger than 75 μm is determined by sieving, while the distribution of particle sizes smaller than 75 μm is determined by a sedimentation process, using a hydrometer to obtain the necessary data.

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1.2. Detection limits are determined from accuracy of analytical balances. Samples are weighed to the nearest 0.01g and results are reported to the nearest 0.01 percent.

1.3. The pre-preparation of the samples employs ASTM Method D421-85.

2. METHOD SUMMARY

Particle size is used to characterize the physical characteristics of sediments. Because particle size influences both chemical and biological variables, it can be used to normalize chemical concentrations according to sediment characteristics and to account for some of the variability found in biological assemblages. Particle size is also an important variable for marine engineering purposes.

COPY

3. DEFINITIONS

3.1. Particle size – The size of various solid components making up a sediment, as named in the applicable method reference (gravel, sand, silt, clay, etc.).

3.2. Dispersing agent – A solution introduced into the soil suspension to disperse the small sediment fractions and reduce flocculation, allowing more accurate determination of particle sizes using a hydrometer.

4. INTERFERENCES

Depending on the required particle size distribution, organic material can be an interference.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Samples can be collected in glass or plastic containers. A minimum sample size of 300-2000g is recommended. If unrepresentative material is to be removed from the sample, it should be removed in the field under the supervision of the chief scientist and noted on the field log sheet.

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6.2. Samples should be stored at 4 ± 2 °C, and can be held for up to 6 months before analysis. Samples must not be frozen or dried prior to analysis, as either process may change the particle size distribution.

7. APPARATUS AND EQUIPMENT

7.1. Sieve shaker - Ro-Tap or equivalent

7.2. Drying oven

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7.3. Mortar and rubber covered pestle

7.4. Thermometer accurate to 0.5 °C

7.5. Analytical balance - 0.1mg accuracy

7.6. Desiccator

7.7. Clock - with second hand

7.8. Standard sieves - Appropriate mesh sizes, sieve pan and top, sieve brush.

7.9. Funnel

7.10. Graduated cylinders

7.11. 250-mL beakers

7.12. Hydrometer – An ASTM hydrometer, graduated to read specific gravity and conforming to the requirements for hydrometers 151H or 152H in Specifications E 100.

7.13. Stirring apparatus as described in ASTM D422-63 Section 3.2.1.

7.14. Water pique or squirt bottle

7.15. Glossy paper

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

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8.1. Dispersant - 4 percent sodium hexamethaphosphate. To prepare, weigh 40.0g sodium hexamethaphosphate and dilute to 1.0L in DIW.

8.2. Distilled water

9. PREVENTIVE MAINTENANCE

9.1. No specific maintenance steps are needed for sieves other than normal cleaning and inspection.

9.2. Balance calibration checks are performed daily.

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9.3. Color-indicating desiccant is recommended so that spent desiccant can be detected easily. Also, the seal on the desiccator should be checked periodically, and, if necessary, the ground glass rims should be greased or the "0" rings should be replaced.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Training and proficiency is documented in accordance with the *SOP for Documentation of Training* (ADM-TRANDOC).

11. PROCEDURE

11.1. Sample Preparation (from ASTM D421-85)

11.1.1. As received samples shall be air-dried at room temperature until thoroughly dried.

11.1.2. A portion of the as received sample shall be used to determine the specific gravity of the sample for later use on the report.

11.1.3. Air-dried samples shall be broken up into the individual aggregates using a mortar and rubber covered pestle. Analyst will take care not to reduce the size of individual particles, but only de-aggregate clumps of dried material.

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11.1.4. After de-aggregation, select approximately 10-15g for hygroscopic moisture content. Moisture content is to be measured by drying the sub sample at 110 ± 5 °C.

11.2. Analysis

11.2.1. Select a portion of the air-dried sample for the purpose of testing. Separate the test sample by sieving with a No. 10 sieve. Grind the fraction retained on the No. 10 sieve with a mortar and rubber covered pestle until the aggregations of soil particles are broken up into separate grains and re-sieve that portion.

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11.2.2. Wash the fraction retained on the No. 10 sieve free of all fine material, dry at 110 ± 5 °C, and weigh. Record this mass as the mass of course material.

11.2.3. Separate the portion retained on the No. 10 sieve into a series of fractions using the 3/4, 3/8 in, No. 4 (4.75 mm) and No. 10 (2.00 mm) sieves and a Ro-Tap shaker.

11.2.4. Determine the mass of each fraction using a balance accurate to 0.1mg and record on the benchsheet.

11.2.5. Thoroughly mix together the fractions passing the No. 10 sieve in both sieving operations, and by the method of quartering, select a portion weighing approximately 115g for sandy soils and 65g for silt and clay soils.

11.2.6. Take 10 to 15g of the sample passing the No. 10 sieve, dry it at 110 ± 5 °C, and record the weight. Enter these weights on the benchsheet under hygroscopic moisture.

11.2.7. Weight a portion of the remaining sample passing the No. 10 sieve. Use approximately 50g when the sample is mostly silt and/or clay and approximately 100g when the sample is mostly sand. Place this portion in a 250 mL beaker and cover with 125 mL of sodium hexametaphosphate solution and stir until thoroughly wetted. Allow to soak for at least 16 hours.

11.2.8. After the 16 hour soaking period, move wetted sample into a stirring apparatus as described in ASTM D422-63 Section 3.2.1. Stir sample for a period of one minute and transfer slurry into a sedimentation cylinder. Bring the final volume up to 1 L.

11.2.9. Determine the correction factor for each hydrometer by placing 125 mL sodium hexametaphosphate solution in a sedimentation cylinder, and diluting to 1 L with de-ionized water. Place the hydrometers in the cylinder and take a reading. This number will be subtracted from each reading taken. It is important to note that the readings should be taken at the top of the meniscus as it is nearly impossible to see the bottom of the meniscus when soil particles are present in the suspension.

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11.2.10. Cap the sedimentation cylinder with Parafilm and turn the cylinder upside down and back for a period of one minute. At the end of the one minute, set the cylinder on the counter, uncap, and take hydrometer readings of the suspension at the following intervals: 2, 5, 15, 30, 60, 250, and1440 minutes. When a hydrometer reading is taken, carefully insert the hydrometer into the suspension 20 to 25 seconds before the desired reading to allow the hydrometer to settle. After the reading is taken, carefully remove the hydrometer from the suspension and place it with a spinning motion in a graduate of clean de-ionized water. Record the temperature of the suspension after each hydrometer reading.

11.2.11. After the last hydrometer reading is taken, transfer the suspension to a No. 200 (75 μm) sieve and wash with tap water until the wash water is clear. Transfer the material to a suitable weighed container and dry to a constant weight in an oven at110 ± 5 °C, record dried sample weight, and make a sieve analysis of the portion retained using the No. 20, No. 40, No. 60, No. 140, and No. 200 sieves and a pan.

12. QA/QC REQUIREMENTS

For ASTM D422-63 one duplicate analyses shall be conducted on one of every 20 samples, or one sample per batch if less than 20 samples are analyzed.

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. The data is entered into a spreadsheet and results determined using the appropriate equations. Refer to Appendix A.

13.2. Reporting and review

13.2.1. The weight of each sediment fraction should be reported to the nearest 0.0001g dry weight. The laboratory should report the results of all samples analyzed (including QA replicates) and should note any problems that may have influenced data quality.

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13.2.2. It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified for samples (above). These results are then used to calculate QC determinations

13.2.3. The results are entered directly onto the appropriate EDD forms located in the CAS network directory R:\WET\WIP. Refer to Appendix A. Once the results are transferred, the data and report are reviewed.

13.2.4. Refer to the SOP for Laboratory Data Review Process for general instructions for data review.

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14. CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the SOP for *Nonconformity and Corrective Action* for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for *Report Generation* or in project-specific requirements.

15. METHOD PERFORMANCE

Refer to the reference method for method performance data available.

16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the

volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

18. TRAINING

18.1. Training outline

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18.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

18.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

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18.1.3. Independently perform the analyses. For Initial Demonstration of Capability the data must be reviewed by a supervisor and the supervisor must document that the analyst is trained.

18.2. Training is documented following the *SOP for Documentation of Training*.

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

19. REFERENCES

ASTM Procedure D422.

Appendix A

Benchsheets and Spreadsheets

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COPY

Columbia Analytical Services, Inc.

Sample Name :	sample 1	Service Request:	0
Lab Code:	-001	Method:	D421 / D422
Client:	0	Date Collected:	01/00/00
Project:	0	Date Received:	01/00/00
Sample Matrix:	0	Date Analyzed:	01/00/00
Time Started:	0000		

Sample Preparation (ASTM D421-85)

(1) Mass of total test sample. (g) (6.1) _____

(2) Coarse Material (6.2)

Coarse material + Tare. (g) _____

Tare weight. (g) _____

(3) Mass retained on the No. 4 sieve. (g) (6.2) _____

(4) Selected portion of fine material. (g) (7.1) _____

Particle Analysis (ASTM D422-63)

Sieve Analysis Of Coarse Material. (6.1)

Gravel 19.0 mm	3/4"	
Gravel 9.50 mm	3/8"	
Gravel 4.75 mm	4	
Gravel 2.00 mm	10	

Sieve Analysis Of Fine Material. (11.1)

Gravel 0.850 mm	20	
Gravel 0.425 mm	40	
Gravel 0.250 mm	60	
Gravel 0.106 mm	140	
Gravel 0.075 mm	200	
Pan	-	

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Analysis of fine material.

Hydrometer corrections. (7.3)

	Hydro 1	Hydro 2
Reading:		

Hygroscopic moisture. (8.1)

Air dried (g)	
Tare (g)	
Oven dried + Tare	

Weight of Fine material

Dry wt.+Tare (g)	
Tare (g)	
Fine material (g)	

Determination of Silt/Clay Fraction. (10.2) (10.3) (10.4)

(10.2) T (Min)	(10.2) Hydro Reading	(7.3) Corr. Fact.	(10.4) Temp °C	Specific Gravity		Table 1 a		Table 3 K		
2										
5										
15										
30										
60										
250										
1440										

Analyst : _____ Date : _____

Reviewed by : _____ Date : _____

Client:	0	**Service Request:**	
Project:	0	**Date Collected:**	
Sample Matrix:	0	**Date Received:**	
		Date Analyzed:	

ASTM Method D422 Particle Size

Sample Name: sample 1 **Lab Code:** -001

Initial Weight of air-dried sample. **(6.1)**	
Mass retained on the No. 4 sieve. **(6.2)**	
Air-dried portion of fine material. **(7.1)**	

Course Sieving ASTM D421 (6.2)

Mass of coarse material (g)	
Percent Recovered	

Coarse sieving (6.1)		**Sieve #**	**Weight (g)**	**% Passing**	**Mass Passing**
Gravel	19.0 mm	3/4"			
Gravel	9.50 mm	3/8"			
Medium Gravel	4.75 mm	4			
Fine Gravel	2.00 mm	10			
Fine sieving (11.1)					
V.C. Sand	0.850 mm	20			
C. Sand	0.425 mm	40			
M. Sand	0.250 mm	60			
F. Sand	0.106 mm	140			
V.F. Sand	0.075 mm	200			**(16.2)**

Calculated amount retained on the # 10 sieve. **(16.1)**	

Total Recovered (g)	
Total Recovered (%)	

Hygroscopic Moisture (8.1)

Air-dried sample (g)	
Oven dried sample (g)	
Hygroscopic Moisture **(13.1)**	

(W) **(14.2)**	

Oven-Dried Sample used in hydrometer analysis. (g) **(14.1)**	

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Determination of Silt/Clay Fraction

(10.2) T (Min)	**(10.2)** Hydro Reading	**(7.3)** Corr. Fact.	**(10.4)** Temp °C	**Table 1** Specific Gravity	H (Net)	**(14.3)** a	**Table 2** L Eff. Depth	**Table 3** K	**(14.3)** % Passing = H/Wa * 100	**(15.1)** Dia-meter
2										
5										
15										
30										
60										
250										
1440										

Comments:

Analyzed By:	Date:
Reviewed By:	Date:

Sieve	X arithmetic Percent Passing (%)	Y logarithmic Particle Diameter (mm)	Convert Y mm to nm (nm)	Value of Y Log form (log)
3/4"		19.0	19000000	7.279
3/8"		9.5	9500000	6.978
4		4.75	4750000	6.677
10		2.00	2000000	6.301
20		0.850	850000	5.929
40		0.425	425000	5.628
60		0.250	250000	5.398
140		0.106	106000	5.025
200		0.0750	75000	4.875
2			#VALUE!	#VALUE!
5			#VALUE!	#VALUE!
15			#VALUE!	#VALUE!
30			#VALUE!	#VALUE!
60			#VALUE!	#VALUE!
250			#VALUE!	#VALUE!
1440			#VALUE!	#VALUE!

determined hydrometer mm	mm to nm	log hyd x	% Passing
0.074	74000	4.87	#VALUE!
0.005	5000	3.70	#VALUE!
0.001	1000	3.00	#VALUE!

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COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client: 0
Project: 0
Sample Matrix: 0

Service Request: 0
Date Collected:
Date Received:
Date Analyzed:

Particle Size Determination
ASTM Method D 422

Sample Name: sample 1
Lab Code: -001

Gravel and Sand
(Sieve Analysis)

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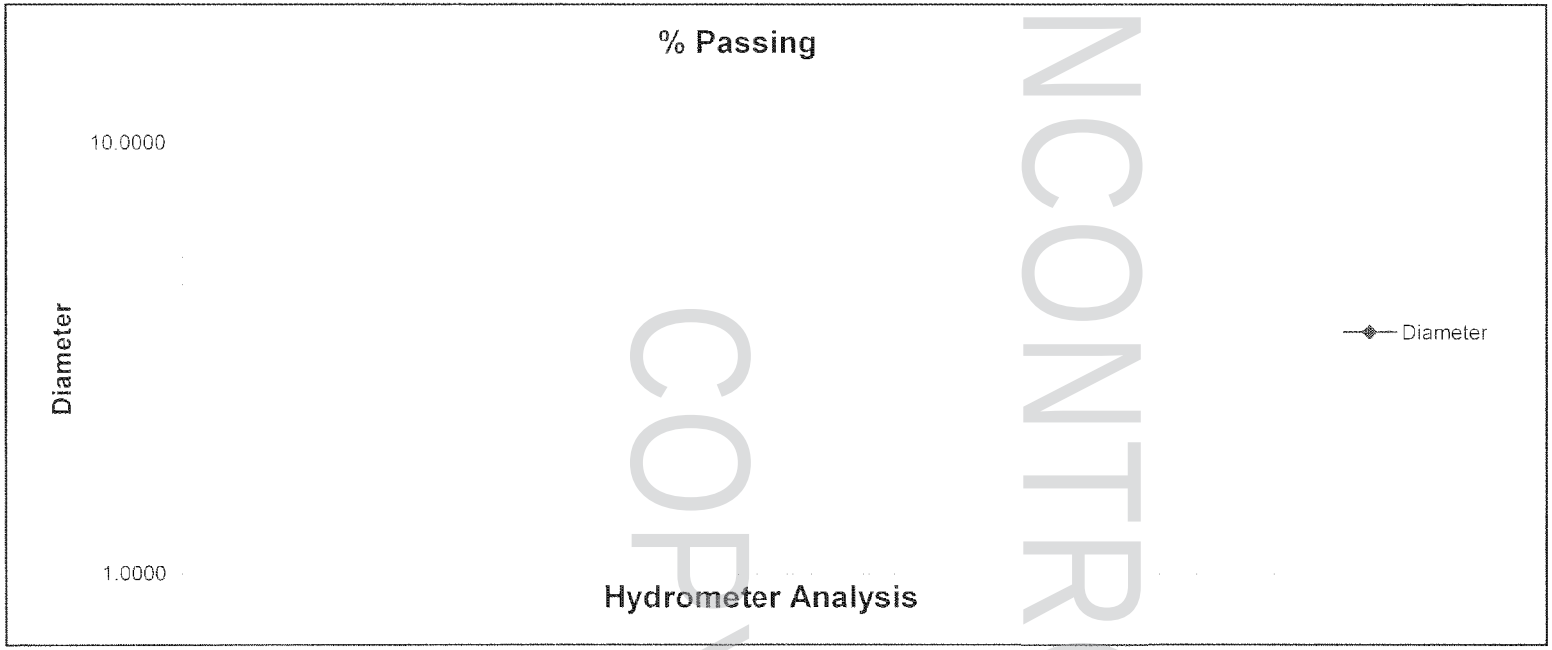
Description	Sieve Size	Weight (g)	Percent Passing
Gravel	No.3/4"(19.0 mm)		
Gravel	No.3/8"(9.50 mm)		
Gravel, Medium	No.4 (4.75 mm)		
Gravel, Fine	No.10 (2.00 mm)		
Sand, Very Coarse	No.20 (0.850 mm)		
Sand, Coarse	No.40 (0.425 mm)		
Sand, Medium	No.60 (0.250 mm)		
Sand, Fine	No.140 (0.106 mm)		
Sand, Very Fine	No.200 (0.0750 mm)		

Silt and Clay
(Hydrometer Analysis)

Particle Diameter	Percent Passing
0.074 mm	#VALUE!
0.005 mm	#VALUE!
0.001 mm	#VALUE!

Approved By: _____ Date: _____
1A/102094

Sample Name: sample 1
Lab Code: -001



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STANDARD OPERATING PROCEDURE

PARTICLE SIZE DETERMINATION
GEN-PSP
Revision 6
July 23, 2009

UNCONTROLLED COPY

Approved By: _____ Supervisor _____ 7/24/09 Date

_____ QA Manager _____ 7/24/09 Date

_____ Laboratory Manager _____ 7/27/09 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL
NUMBER: _____
Initials: _____ Date: _____

PARTICLE SIZE DETERMINATION

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine the fraction of pre-determined sizes of particles in sediments. The procedures are based on Puget Sound Protocols, Plumb (1981) and ASTM D422 methodology. Particle size can be characterized in a wide range of detail. The grossest divisions that generally are considered useful for characterizing particle size distributions are percentages of gravel, sand, silt, and clay. However, each of these size fractions can be subdivided further so that additional characteristics of the size distribution (e.g., mean diameter, skewness, kurtosis) can be determined. Additionally, this procedure describes wet sieving protocols used when trace metals analysis are to be performed on the sieved fractions.

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1.2. Detection limits are determined from accuracy of analytical balances. Samples are weighed to the nearest 0.01g and results are reported to the nearest 0.01 percent.

2. METHOD SUMMARY

2.1. Particle size is used to characterize the physical characteristics of sediments. Because particle size influences both chemical and biological variables, it can be used to normalize chemical concentrations according to sediment characteristics and to account for some of the variability found in biological assemblages. Particle size is also an important variable for marine engineering purposes. In addition to Plumb (1981), other references discuss the uses and measurement of particle size (e.g., Krumbein and Pettijohn 1938; Folk 1968; Buchanan 1984).

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2.2. Particle size determinations can either include or exclude organic material. If organic material is removed prior to analysis, the "true" (i.e., primarily inorganic) particle size distribution is determined. If organic material is included in the analysis, the "apparent" (i.e., organic plus inorganic) particle size distribution is determined. Because true and apparent distributions may differ, detailed comparisons between samples analyzed by these different methods are questionable. It is therefore desirable that all samples within each study (at a minimum) and among different studies (if possible) be analyzed using only one of these two methods.

3. DEFINITIONS

3.1. Particle size – The size of various solid components making up a sediment, as named in the applicable method reference (gravel, sand, silt, clay, etc.).

3.2. True particle size – The particle size determined when organic material is removed prior to analysis.

3.3. Apparent particle size – The particle size determined when organic material is not removed prior to analysis.

4. INTERFERENCES

Depending on the required particle size distribution, organic material can be an interference.

5. SAFETY

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5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

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6.1. Samples can be collected in glass or plastic containers. A minimum sample size of 100-150g is recommended. If unrepresentative material is to be removed from the sample, it should be removed in the field under the supervision of the chief scientist and noted on the field log sheet.

6.2. Samples should be stored at 4 ± 2 °C, and can be held for up to 6 months before analysis. Samples must not be frozen or dried prior to analysis, as either process may change the particle size distribution.

7. APPARATUS AND EQUIPMENT

7.1. Sieve shaker - Ro-Tap or equivalent

7.2. Drying oven

7.3. Constant temperature bath

7.4. Analytical balance - 0.1mg accuracy

7.5. Desiccator

7.6. Clock - with second hand

7.7. Standard sieves - Appropriate mesh sizes, sieve pan and top, sieve brush.

7.8. Funnel

7.9. Graduated cylinders

7.10. 250-mL beakers

7.11. 20-mL pipettes

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7.12. Water pique or squirt bottle

7.13. Glossy paper

7.14. 1000 mL Polycarbonate Centrifuge Bottles

7.15. Centrifuge - With buckets appropriate for 1000 mL bottles

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8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Dispersant - 1 percent sodium hexamethaphosphate = 1 percent commercially available Calgon. To prepare, weigh 10.0g sodium hexamethaphosphate and dilute to 1.0L in DIW.

8.2. Distilled water

9. PREVENTIVE MAINTENANCE

9.1. No specific maintenance steps are needed for sieves other than normal cleaning and inspection.

9.2. Balance calibration checks are performed daily.

9.3. Color-indicating desiccant is recommended so that spent desiccant can be detected easily. Also, the seal on the desiccator should be checked periodically, and, if necessary, the ground glass rims should be greased or the "0" rings should be replaced.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Training and proficiency is documented in accordance with the SOP for Documentation of Training (ADM-TRANDOC).

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11. PROCEDURE (Particle Size Fractionation)

11.1. Sample Preparation

11.1.1. Unless specifically asked for, organic oxidation is not performed and the apparent particle size distribution is determined.

11.1.2. It is critical that each sample be homogenized thoroughly in the laboratory before a subsample is taken for analysis. Laboratory homogenization should be conducted even if samples were homogenized in the field.

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11.1.3. The total amount of sample used for analysis should be 25-100g. Refer to section 12.1 for a discussion of use of alternate sample sizes.

11.2. Analysis

11.2.1. Particle size determination is determined by four similar methods using this SOP. They include Puget Sound Estuary Protocols; condensed version and expanded version, and ASTM D422 Modified; condensed version and expanded version. The differences between these methods are explained below:

11.2.1.1. Puget Sound Estuary Protocol (Condensed Version): Course particle size fractions are determined by using the following standard sieves: 10, 18, 35, 60, 120, and 230. The silt/clay portion of the sample is determined by pipette extraction using the withdrawal times given in Table 1.

11.2.1.2. Puget Sound Estuary Protocol (Expanded Version): Course particle size fractions are determined by using the following standard sieves: 10, 18, 35, 60, 120, and 230. The silt/clay portions of the sample are broken down into subdivisions and are determined by pipette extraction using the withdrawal times given in Table 2.

11.2.1.3. ASTM D422 Modified (Condensed Version): Course particle size fractions are determined by using the following standard sieves: 4, 10, 20, 40, 60, 140, and 200. The silt/clay portion of the sample is determined by pipette extraction using the withdrawal times given in Table 1.

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11.2.1.4. ASTM D422 Modified (Expanded Version): Course particle size fractions are determined by using the following standard sieves: 4, 10, 20, 40, 60, 140, and 200. The silt/clay portions of the sample are broken down into subdivisions and are determined by pipette extraction using the withdrawal times given in Table 2.

11.2.2. Weigh sample as received and perform total solids at the same time.

11.2.3. Wet sieve through the appropriate (200 or 230) sieve, washing what goes through into a 1000mL graduated cylinder.

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11.2.4. Add 10mL of a 1% sodium hexametaphosphate solution to cylinder.

11.2.5. Retain what was left on the 200 (or 230) sieve (gravel/sand fraction). Wash into a preweighed 250-ml beaker, and dry in 105°C oven to dryness. Dry sieve through the applicable sieves (see 11.2.1). After dry-sieving a sample, all material must be removed from the sieve. This can be accomplished by tapping the rim of the sieve evenly on a hard surface and by brushing the screen.

11.2.6. Silt-clay fraction:

Note: Before pipette extractions can be made, the sample must be homogenized thoroughly within the settling cylinder. Once the pipette analysis begins, the settling cylinders must not be disturbed, as this will alter particle settling velocities. Care must be taken to disturb the sample as little as possible when pipette extractions are made.

11.2.6.1. After sample is brought to 1L and mixed, it is left on the counter and the temperature is tracked with a thermometer in a graduated cylinder with DI water. The temperature does not usually vary by more than ± 2°C.

11.2.6.2. Withdrawal times for pipette analysis as a function of particle size and water temperature are given in Tables 1 and 2. Shake for 1 minute and take a 20mL aliquot at 20cm deep at 20 seconds.

11.2.6.3. Take 20mL aliquots at 10cm at differing times according to temperature of the suspension (see the attached sheets for specific times).

11.2.6.4. If the condensed analysis is wanted (silt & clay) only use 4 and 8 phi/times (Table 1). If the expanded analysis is wanted, refer to Table 2 for phi sizes and times.

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11.2.6.5. After a pipette extract has been transferred to a drying beaker, any sample adhering to the inside of the pipette must be removed. This can be accomplished by drawing 20mL of distilled water into the pipette and adding this rinse water to the drying beaker.

11.2.7. Dried samples should be cooled in a desiccator and held there until they are weighed. If a desiccator is not used, the sediment will accumulate ambient moisture and the sample weight will be overestimated. A color-indicating desiccant is recommended so that spent desiccant can be detected easily.

12. PROCEDURE (Preparation for trace metals analysis)

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12.1. Special Considerations

12.1.1. Metal sieves must not be used when preparing samples for trace metals analysis. Polymer mesh and pans are to be utilized.

12.1.2. Site water related to the samples being prepared is recommended for the wet sieving procedure. However, if site water is not available laboratory dionized water can be used.

12.1.3. Decontamination of all equipment that comes in contact with the sample must be performed prior to initiating the sieving. Rinse with 25% hydrochloric acid, followed by a minimum DIW rinses.

12.2. Analysis (Dry Sieve for Fractions >63 um)

12.2.1. Follow protocol described in Section 11, but include considerations listed in Section 12.1.

12.3. Analysis (Wet Sieve for ≤ 63 um Fraction)

12.3.1. Place an aliquot of sample onto the appropriate size sieve (sieve sized to be determined on a project specific basis).

12.3.2. Using a squirt bottle filled with the specified rinse solution (i.e. site or DI water) wash the sample through the sieve. Use a plastic funnel to capture the target sample fraction in a 1000 mL polycarbonate centrifuge bottle.

12.3.3. When sufficient sample has been captured, centrifuge the polycarbonate bottle then pour off and discard the supernatant. Transfer the remaining sample to an appropriate sized glass jar (i.e. 8 oz., 16 oz., etc.) clearly labeled with the sample I.D. and particle size fraction information. Store at 4±2°C.

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12.3.4. The remaining sample is now ready for trace metals determination. (Note: The variety and combinations of trace metals analyses that can be performed vary widely, so subsequent analytical procedures will be defined on a project specific basis.) If further particle size reduction in needed an aliquot is retained for analysis. The remaining sample is then processed once more as described above using the next smaller sieve size as designated in the project plan.

12.3.5. So the final results can be reported on a dry weight basis perform a total solids determination on each fraction being analyzed for trace metals.

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13. QA/QC REQUIREMENTS

13.1. The total amount of fine-grained material used for pipette analysis should be 5-25g. If more material is used, particles may interfere with each other during settling and the possibility of flocculation may be enhanced. If less material is used, the experimental error in weighing becomes large relative to the sample size.

13.2. It is recommended for Puget Sound Protocols that triplicate analyses be conducted on one of every 20 samples, or one sample per batch if less than 20 samples are analyzed. For ASTM Modified, it is recommended that duplicate analyses be conducted on one of every 20 samples, or one sample per batch is less than 20 samples are analyzed.

14. DATA REDUCTION, REVIEW, AND REPORTING

14.1. Calculations

14.1.1. The total weight of a phi-size interval in the 1-L graduated cylinder is determined as follows:

Phi weight (g dry weight) = 50[(A-C)-(B-C)]

Where: A = weight (g) of residue in a 20-mL aliquot for a given phi-size boundary
B = weight (g) residue in a 20-mL aliquot for the next larger phi-size boundary
C = mean weight (g) of dispersant in a 20-mL aliquot.

14.2. The data is entered into a spreadsheet and results determined using the appropriate equations. Refer to Appendix A.

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14.3. Reporting and review

14.3.1. The weight of each sediment fraction should be reported to the nearest 0.0001g dry weight. The laboratory should report the results of all samples analyzed (including QA replicates) and should note any problems that may have influenced data quality.

14.3.2. It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified for samples (above). These results are then used to calculate QC determinations

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14.3.3. The results are entered directly onto the appropriate EDD forms located in the CAS network directory R:\WET\WIP. Refer to Appendix A. Once the results are transferred, the data and report are reviewed.

14.3.4. Refer to the SOP for Laboratory Data Review Process for general instructions for data review.

15. TRAINING

15.1. Training outline

15.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

15.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a

role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

15.1.3. Independently perform the analyses. For Initial Demonstration of Capability the data must be reviewed by a supervisor and the supervisor must document that the analyst is trained.

15.2. Training is documented following the *SOP for Documentation of Training*.

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

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16. CHANGES SINCE THE LAST REVISION

16.1. Sec 1.1 removed the word Estuary.
16.2. Sec. 1.1 added Plumb 1981 reference
Sec 12.2 Added dry sieve fraction size.
Sec 12.3 Added wet sieve fraction size
Sec 12.3.3 Added storage and container transfer instructions.
Sec 12.3.4 Added note for trace metal analysis.

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17. REFERENCES

17.1. Conventional Sediment Variables - Particle Size, March 1986, *Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound*, January, 1996.

17.2. *Procedures for Handling and Chemical Analysis of Sediment and Water Samples*, R.H. Plumb, prepared for USEPA and Army Corps of Engineers, May, 1981.

17.3. ASTM Procedure D422.

Table 1

Withdrawal Times for Pipette Analysis as a Function of Particle Size and Water Temperature Silt & Clay Fraction – Condensed Analysis

Micron µm	Diameter finer than (phi)	Diameter finer than (µm)	Withdrawal depth (cm)	Elapsed time for withdrawal of sample in hours (h), minutes (m), and seconds(s).						
				18°C	19°C	20°C	21°C	22°C	23°C	24°C
62.5	4.0	62.5 - 3.9	20	20s	20s	20s	20s	20s	20s	20s
3.9	8.0[a]	3.9	10	2h8m	2h5m	2h2m	1h59m	1h56m	1h53m	1h51m

a) Breakpoint between silt and clay.

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Table 2

Withdrawal Times for Pipette Analysis as a Function of Particle Size and Water Temperature Silt & Clay Fraction – Expanded Analysis

Micron µm	Diameter finer than (phi)	Diameter finer than (µm)	Withdrawal depth (cm)	Elapsed time for withdrawal of sample in hours (h), minutes (m), and seconds(s).						
				18°C	19°C	20°C	21°C	22°C	23°C	24°C
62.5	4.0	62.5	20	20s	20s	20s	20s	20s	20s	20s
31.2	5.0	31.2	10	2m0s	1m57s	1m54s	1m51s	1m49s	1m46s	1m44s
15.6	6.0	15.6	10	8m0s	7m48s	7m36s	7m25s	7m15s	7m5s	6m55s
7.8	7.0	7.8	10	31m59s	31m11s	30m26s	29m41s	28m59s	28m18s	27m39s
3.9	8.0[a]	3.9	10	2h8m	2h5m	2h2m	1h59m	1h56m	1h53m	1h51m
1.95	9.0	1.95	10	8h32m	8h18m	8h6m	7h56m	7h44m	7h32m	7h22m
0.98	10.0	0.98	10	34h6m	33h16m	32h28m	31h40m	30h56m	30h12m	29h30m

a) Breakpoint between silt and clay.

Appendix A

Benchsheets and Spreadsheets

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1317 South 13th Avenue
Kelso, Wa 98626

Method: PSEP Particle Size
Puget Sound Protocol

Service Request #: 0
Sample #: 0

Client:	0
Project:	0
Sample Matrix:	0

Sample Name:	0
Date Collected:	1/0/1900
Date Received:	1/0/1900
Date Analyzed:	1/0/1900

I. Sieving Operation	Sieve #	Weight (g)	As Rec'd (g)
Gravel 2.00 mm (g)	10		#DIV/0!
V.C. Sand, 1.00 mm (g)	18		#DIV/0!
C. Sand, 0.500 mm (g)	35		#DIV/0!
M. Sand, 0.250 mm (g)	60		#DIV/0!
F. Sand, 0.125 mm (g)	120		#DIV/0!
V.F. Sand, 0.0625 mm (g)	230		#DIV/0!
S/C <0.0625 mm (g)	Pan		#DIV/0!

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Total (g) Recov'd	0.0000
Total (%) Recov'd	#DIV/0!

II. Dry Sieving of Gravel/Sand

Grams Gravel/Sand & Beaker	
Grams Beaker (Tare)	
GramsGravel/Sand	0.0000

I. Sample Preparation

Grams As Received Sample	
Percent (%) Solids	
Grams Oven Dried Sample	0.0000

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III. Determination of Silt/Clay Fraction

Temperature: 0 Time Start: 12:00 AM Time Finish: 12:00 AM

	Silt/Clay Fraction	Clay Fraction
Total Volume of Sample (mls)	1000	1000
Amount of Dispersant	10	10
Volume of Aliquot (mls)	20	20
Grams Sample, Dispersant & Tare		
Grams of Tare		
Grams of Dispersant Correction	0.0000	0.0000
Grams of Sample	0.0000	0.0000
Total Grams Sample X 50	0.0000	0.0000

Analyst: 0 Date: 1/0/1900
Reviewed by: Date:

COLUMBIA ANALYTICAL SERVICES, INC.

1317 South 13th Avenue
Kelso, Wa 98626

Method: PSEP Particle Size
Puget Sound Protocol

Service Request #: 0
Sample #: 0

Client:	0
Project:	0
Sample Matrix:	0

Sample Name:	0
Date Collected:	1/0/1900
Date Received:	1/0/1900
Date Analyzed:	1/0/1900

I. Sieving Operation	Sieve #	Weight (g)	As Rec'd (g)
Gravel 2.00 mm (g)	10		N/A
V.C. Sand, 1.00 mm (g)	18		N/A
C. Sand, 0.500 mm (g)	35		N/A
M. Sand, 0.250 mm (g)	60		N/A
F. Sand, 0.125 mm (g)	120		N/A
V.F. Sand, 0.0625 mm (g)	230		N/A
S/C <0.0625 mm (g)	Pan		N/A

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Total (g) Recov'd	0.0000
Total (%) Recov'd	#DIV/0!

II. Dry Sieving of Gravel/Sand

Grams Gravel/Sand & Beaker	
Grams Beaker (Tare)	
GramsGravel/Sand	0.0000

I. Sample Preparation

Grams As Received Sample	
Percent (%) Solids	
Grams Oven Dried Sample	0.0000

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III. Determination of Silt/Clay Fraction

Temperature: _____

	4	5	6	7	8	9	10
Total Volume of Sample (mls)	1000	1000	1000	1000	1000	1000	1000
Amount of Dispersant	10	10	10	10	10	10	10
Volume of Aliquot (mls)	20	20	20	20	20	20	20
Grams Sample, Dispersant & Tare							
Grams of Tare							
Grams of Dispersant Correction		0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
Grams of Sample	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
Total Grams Sample X 50	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000

Analyst: _____ Date: _____
Reviewed by: _____ Date: _____

STANDARD OPERATING PROCEDURE

for

METALS DIGESTION

SOP No.: MET-3050

Revision: 10

July 12, 2007

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Approved by: _____ Supervisor — 7/12/07 Date

_____ QA Manager — 7-12-07 Date

_____ Laboratory Manager — 7/12/07 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: 7/10/08
Initials: _____ Date: 6/16/09
Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

METALS DIGESTION

1. SCOPE AND APPLICATION

This procedure uses techniques described in method 3050B for acid digestion of sediments, sludges, and soil samples designated for "Total Metals" analysis. One technique is designed for the preparation of samples for analysis by flame AA (Methods 7420-Pb, 7742-Se, and 7062-As) or ICP-OES (methods 6010B and 200.7). Another technique is given for the preparation of samples for analysis by GFAA (see SOP MET-GFAA for methods) or ICP-MS (methods 6020 and 200.8). This procedure is not a *total digestion* technique, but extracts "environmentally available" elements from the sample of interest.

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2. METHOD SUMMARY

One-gram equivalent dry weight sediment, sludge, or soil samples are digested with repeated additions of nitric acid (HNO_3) and hydrogen peroxide (H_2O_2). For GFAA and ICP-MS analysis the resultant digestate is reduced in volume while heating and then diluted to a final volume of 100 mL. For ICP-OES and flame AA analysis, hydrochloric acid (HCl) is added to the initial digestate and the sample is refluxed prior to dilution to a final volume of 100 mL.

3. DEFINITIONS

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3.1. Laboratory Control Sample (LCS): A laboratory blank that has been fortified with target analyte and used to determine that the analysis is in control.

3.2. Matrix Spike (MS) Analysis - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The percent recovery is calculated. The MS is used to evaluate the effects of the sample matrix on the method used for the analysis. The concentration of the spike should be at three to five times the sample result or at levels specified by a project analysis plan.

3.3. Duplicate Sample (DUP) - A laboratory duplicate. The duplicate sample is a separate field sample aliquot that is processed in an identical manner as the sample proper. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.4. Method Blank (MB) - The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

4. INTERFERENCES

Refer to the determinative method for a discussion of interferences.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

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6. SAMPLE COLLECTION, PRESERVATION AND STORAGE

Samples may be collected in plastic or glass jars. Non-aqueous samples are refrigerated at 4 ± 2°C from receipt until analysis.

7. APPARATUS AND EQUIPMENT

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7.1. Borosilicate glass beakers, 150 mL

7.2. Borosilicate watch glasses, ribbed

7.3. Hot Plates: "Thermolyne Cimerac 3", calibrated to maintain 90-95°C

7.4. Polypropylene graduated cylinders, 100 mL

7.5. Laboratory balance, top-loader capable of readings .001g (3-place)

7.6. Evergreen disposable tubes, 50 mL. Check tubes for accuracy on a per batch basis by filling a tube to the 50 mL mark and measuring the water's mass. The measured mass must be accurate to ± 3%, if not obtain a new lot of tubes and retest. Refer to the SOP for Checking Pipet Calibration (ADM-CPIP), section 8.1 for detailed instructions on performing the accuracy test.

7.7. USS # 10 sieve.

8. STANDARDS AND REAGENTS

8.1. Reagent water: ASTM Type I water (resistivity ≥18 MΩ-cm, conductivity ≤0.056 uS/cm).

8.2. Concentrated Nitric Acid: J.T. Baker "Instra-analyzed", Trace Metals Grade

8.3. Concentrated Hydrochloric Acid: EMD GR ACS

8.4. Hydrogen Peroxide (30%): EMD GR ACS

8.5. Metals spiking solutions: Three spiking solutions are needed to prepare the matrix spike sample; SS1, SS5 and SS6. Follow the formulations laid out on the "Metals Spike Form" (see Attachments for example). These solutions are prepared in acid rinsed Class A volumetric flasks using purchased custom mixed standards or 1000 ppm single analyte standards. Aliquots are made using acid rinsed Class A volumetric pipettes of the appropriate size.

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8.5.1. SS1 (Al, Ag, Ba, Be, Cd, Co, Cr, Cu, Fe, Pb, Mn, Ni, Sb, V, and Zn): Fill a 1000 mL volumetric flask approximately half full with reagent water, add 50 mL of nitric acid and mix. Next add 100 mL of the custom mixed standard (CAS-CAL-14) purchased from "Inorganic Ventures". In addition add 50 mL of 1000 ppm Antimony. Dilute to volume with reagent water, mix thoroughly and transfer to a 1000 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

8.5.2. SS4 (GFAA As, Cd, Cu, Pb, Se, Tl): Fill a 500 mL volumetric flask approximately half full with reagent water, add 25 mL of nitric acid and mix. Next add 2.0 mL each of 1000 ppm Arsenic, Cadmium, Copper, Lead, Selenium, and Thallium. Dilute to volume with reagent water, mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

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8.5.3. SS5 (As, Se, and Tl): Fill a 500 mL volumetric flask approximately half full with reagent water, add 25 mL of nitric acid and mix. Next add 50 mL each of 1000 ppm Arsenic, Selenium, and Thallium. Dilute to volume with reagent water, mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

8.5.4. SS6 (B, Mo): Fill a 500 mL volumetric flask approximately half full with reagent water, add 25 mL of nitric acid and mix. Next add 50 mL each of 1000 ppm Boron and Molybdenum. Dilute to volume with reagent water, mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution's expiration date is determined by the earliest expiration date of any single component in the solution.

8.6. Metals reference material (ERA Priority PollutnT/CLP Inorganic Soil) for use as the laboratory control sample. The expiration date is assigned by the manufacturer.

8.7. Teflon beads, Teflon boiling chips, or other suitable blank material.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook. Pertinent information must be in the logbook. Maintenance entries should include date, symptom of problem, corrective actions, and description of maintenance, date, and name. The log should contain a reference to return to analytical control.

9.2. Maintenance for this procedure is generally limited to glassware cleaning, pipet monitoring, hot plate calibration. Procedures for glassware washing are described in the SOP for Metals Laboratory Glassware Cleaning (MET-GC). Procedures for pipet monitoring are given in the SOP for Checking Pipet Calibration (ADM-CPIP).

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9.3. Each hotplate is uniquely identified and temperature calibration performed monthly. To perform the calibration, a certified thermometer is placed in a 150 mL beaker half filled with mineral oil, which is then placed in the center of the hotplate. A clamp is used to ensure the thermometer does not touch the bottom of the beaker. The hot plate is turned to the 95°C setting and the mineral oil is allowed to come to temperature. The analyst will verify that the hotplate gives a temperature of 95°C ±2°C. If not, the thermostat is adjusted until the thermometer reads and maintains 95°C ±2°C. The thermostat is then marked to clearly indicate the correct setting to be used during sample digestion. The thermostat position and thermometer reading are recorded for each hotplate in a bound logbook.

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10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training.

11. PROCEDURE

11.1. Record all digestion and sample information on the applicable benchsheet. To assist the analyst, a brief description of the procedure is given on the backside of the benchsheet (see Attachments).

11.2. Mix the sample thoroughly to achieve homogeneity. Sieve if necessary using a USS #10 sieve.

11.3. It can be difficult to obtain a representative sample with wet or damp materials. As per Method 3050B, wet samples may be dried, crushed, and ground to reduce subsample variability, however, drying is not recommended since drying may affect the extraction of the analytes of interest in the sample.

11.4. Weigh approximately 1g of sample into a 150ml beaker and record the weight to the nearest 0.01g. For sludges and sediments that have high moisture content, use more sample. The objective is to use about 1g of dry weight sample. For analysis of Lead by Flame AA, use about 2.5g of dry wt. sample and change the final dilution volume to 50ml. This will achieve a lower detection limit needed for most projects. At this point add the appropriate spiking solutions directly onto the designated spike sample prior to addition of reagents.

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11.5. Add 5ml reagent water and 5ml concentrated HNO3. Cover and reflux (without boiling) at 95°C for 10 to 15 minutes. Allow the sample to cool, then add 5ml of concentrated HNO3, cover and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO3, repeat the addition of 5ml of HNO3 and reflux over and over until no brown fumes are given off. Reduce the digestate volume to approximately 5 mL without boiling or digest for two hours maintaining a covering of solution over the bottom of the beaker at all times. If this occurs discard the digestate and begin with a new sample aliquot.

Note: When digesting USACE HTRW project samples, the 95°C hotplate temperature must be monitored and documented on a per-batch basis.

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11.6. Cool the sample and add 2 mL of water followed by 3 mL of 30% H_2O_2. Cover and heat to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessive effervescence. Heat until effervescence subsides and cool the beaker.

11.7. Continue to add 30% H2O2 in 1ml aliquots with warming until the effervescence is minimal, or until the general sample appearance is unchanged. Do not add more than 10ml of 30% H2O2. When the peroxide additions are complete cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate until the volume has been reduced to approximately 5 mL or heat at 95°± 5°C without boiling for two hours. Maintain a covering of solution over the bottom of the vessel at all times.

11.8. If the sample is being prepared for analysis by ICP-OES or Flame AA, add 10 mL of concentrated HCl. If the sample is being prepared for ICP-MS or GFAA analysis no HCl is added and the sample may be diluted to 100 mL. (Note: for ICP-MS digestates the tubes must be pre-rinsed with 25% HCl.)

Note: For method 7062 and 7742 samples, the 3050B soil digestion is modified as follows: After the final peroxide addition (i.e. before the final reduction stage) add 5.0mL

of concentrated hydrochloric acid and reduce the digestate volume to less than 5.0mL, but not to dryness. After cooling, dilute the digestate to 100mL with reagent water.

11.9. Cover and reflux the Flame AA and ICP samples for 15 minutes at 95°C. After cooling, the samples may be diluted to 100ml for ICP analysis, or 50ml for Flame AA analysis.

11.10. Particulates in the digestates that may clog the nebulizer are allowed to settle overnight, or the digestates may be centrifuged.

11.11. To improve solubilities for Antimony, Barium, Lead and Silver, the following modification of the digestion procedure may be used as directed by the client or project chemist.

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11.11.1. Weigh (to the nearest 0.01g) 1.00 g of sample into a 150ml beaker. For sludges and sediments that have a high moisture content, use more sample. The objective is to use about 1g of dry weight sample.

11.11.2. Add 2.5mL HNO3 and 10mL HCl and cover with a watch glass. Reflux for 15 minutes.

11.11.3. Filter the digestate through Whatman No. 41or equivalent filter paper and collect in a 100mL volumetric flask. Wash the filter paper, while still in the funnel, with no more than 5mL of hot (95°) HCl, and then with 20mL of hot (95°) reagent water. Collect washing in the same volumetric flask.

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11.11.4. Remove the filter and residue from the funnel, and place them back in the beaker. Add 5mL HCl , cover and heat at 95° ± 5° until the filter paper dissolves. Remove from the heat and wash the cover and sides with reagent water.

11.11.5. Filter the residue and collect the filtrate in the same 100mL flask. Allow to cool, then dilute to volume.

11.11.6. If precipitation occurs in the flask upon cooling, do not dilute to volume. Instead, add up to 10mL of HCl to dissolved the precipitate. After precipitate is dissolved, dilute to volume with water.

12. QA/QC REQUIREMENTS

12.1. Monitor hotplates on a monthly basis. Report all deficiencies to the Lab Manager. Corrective action must be taken.

12.2. Digest one laboratory control sample with each batch. Weigh 1.00 g of the current lot of Environmental Resource Associates PriorityPollutnT/CLP Inorganic Soil prepared reference material into a 150 mL beaker and digest as per the procedure.

12.3. Digest one preparation blank (method blank) per digestion batch, or per 20 samples whichever is more frequent. For the method blank, use Teflon beads, Teflon boiling chips, or other suitable solid blank material and follow the digestion procedures.

12.4. Digest one duplicate and one spiked sample with each sample matrix. Prepare one duplicate and spike sample per each digestion batch, or per twenty samples whichever is more frequent. At times, specific samples will be assigned as duplicates of spikes depending on client requirements.

12.5. Soil spikes are prepared by adding 2.0 mL of spike solution #1, and 1 mL each of spike solutions 5 and 6 directly to the sample aliquot, prior to the addition of any water or acid. Fill out a spiking data sheet and keep it with the digestion data sheets.

For ICP and ICP-MS digestions 2.0 mL of SS1 and 1.0 mL of SS5 and SS6 are added to the sample aliquot designated as the matrix spike sample. For GFAA digestions 2.0 mL of SS4 is added to the sample aliquot designated as the matrix spike sample. The matrix spike sample is then digested as per the procedure.

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13. REPORTING

13.1. Digestion data sheets including weights and volumes used and reagents/acids are completed and a prep run number or batch lot number is assigned and attached to the data sheet. The lot numbers for the reagents used are added to the digestion data sheet (see Attachments).

13.2. Spiking sheets are completed including all spike data and volumes of spiking solutions used (See Attachments).

14. CORRECTIVE ACTION

14.1. Refer to the SOP for *Nonconformity and Corrective Action* for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2. Documentation of a nonconformity must be done using a Nonconformity and Corrective Action Report (NCAR) when: a) corrective action is not taken or not

possible b) corrective action fails to correct an out-of-control problem on a laboratory QC or calibration analysis c) reanalysis corrects the nonconformity but is not a procedurally compliant analysis.

15. METHOD PERFORMANCE

Available method performance data is given in the reference method. In addition, this procedure was validated through single laboratory studies of accuracy and precision as in the determinative procedure. The method detection limit(s) and method reporting limit(s) are established for the determinative procedure.

16. POLLUTION PREVENTION

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It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

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17.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the CAS EH&S Manual for details.

18. TRAINING

18.1. Training outline

18.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

18.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

18.1.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

18.2. Training is documented following the *SOP for Documentation of Training*.

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

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19. REFERENCES

Test Methods For Evaluating Solid Waste, Physical/Chemical Methods. EPA SW-846, 3rd Edition, Final Update III, Method 3050B, December 1996.

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Attachments

Benchsheet
Metals Spiking Form

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Columbia Analytical Services
Metals Digestion Sheet

Service Request Number(s) :
Star Lims Run No.:

Method : CLP 3010A 3020A
3005A 3050B CLP Hot Block Other-_____

Analysis for : ICP ICP-MS GFAA
Flame AA Other _____

Sample	Initial Weight (g)	Dry	Wet	Initial Volume(ml)	Final Volume (ml)	Matrix

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Time Digestion Started:_____

Lot # Acids Used: HNO3 _____ **HCl** _____ **H2O2** _____

LCSS (circle appropriate) ERA CLP Soil Lot # D045540 Other:_____

GFAA LCSW = GFLCSW, MET1-52-W, _____ mls. added
ICP LCSW = CLP-CV-1, MET1-52-A, _____ mls. added
QCP CICV-2, MET1-51-T, _____ mls. added
QCP CICV-3, MET1-51-U, _____mls. added
SS6, MET1-52-Y, _____mls. added

TCLP Spike/LCSE
TSS1 MET1-47-C _____mls. added

SPIKE INFO
SS1-MET1-52-Z,_____mls added SS4-MET1-48-A,_____mls added
SS5-MET1-54-G,_____mls added 200.8 1000ppb Stock (MS9-85-A)_____mls added
SS6-MET1-54-E,_____mls added Ag 1000ppb Stock (MS10-10-A) _____ mls added

Additional spikes:_____

Comments: _____

Analyst _____ **Date** _____

Reviewer_____ **Date** _____

metdig.xls

Digestion Procedures

Method #	GFAA	ICP-OES
CLP Water	Shake sample, Aliquot 50 mls into beaker Add 0.5 mls. HNO3 Add 2 ML 30% H2O2 Cover with ribbed watchglass Heat without boiling for 2 hrs (volume = 0.5x orginal Vol.) Cool sample, Dilute to 50 mls. with DI water	same same Add 2.5 mls Concentrated HCl instead of H2O2 Cover with ribbed watchglass same same
EPA 3010A SW-846 TCLP Ext. USACOE		Shake sample and measure 50 ml into beaker Add 1.5 ml of HNO3, cover with ribbed watchglass Evaporate to 5 ml. Do Not let go DRY Cool, Add 1.5 ml of HNO3, Cover and reflux until digestate is light color or color has stabilized Reduce volume to 5 mls, Cool Add 2.5 mls Concentrated HCl. Cover and reflux for 15 mi Dilute to 50 mls with DI water.
EPA 3020A SW-846 TCLP Ext. USACOE	Shake sample and measure 50 ml into beaker Add 1.5 ml of HNO3, cover with ribbed watchglass Evaporate to 5 ml. Do Not let go DRY Cool, Add 1.5 ml of HNO3, Cover and reflux until digestate is light color or color has stabilized Reduce volume to 5 mls, Cool Add 10 ml DI water, reflux 10-15 min. Dilute to 50 mls with DI water.	
EPA 3050B	Mix sample , weigh out 1.00 g dry weight. * Add spike solution to sample. Add 5 ml DI water and 5 ml HNO3. Reflux 15 min. Cool. Add 5 ml HNO3. Cover. Reflux 30 min. NOTE: *If brown fumes are* generated, repeat the *addition of 5 mls of HNO3 after 30 min of reflux.* Reflux for 2 hrs. or to vol of 5 mls. Cool sample and add 2 mls H20 & 3 mls 30%H2O2 Cover and heat until effervescence subsides Repeat H2O2 adds twice until effervescence is minimal. Don't add > 10 mls. H2O2 **Heat to vol. of 5 mls.** Dilute to 100mls with DI water.	same same same same same same same same same same Add 10 ml of Concentrated HCl, Reflux for 15 min. Dilute to 100 mls. (to 50 mls for Flame)

GFAA NOTE: For SW-846 analysis prepare a digestate from Method 3020B for Pb & Tl analysis & a digestate from the CLFAA for As & Se.

*** Suggested Sample Aliquot Weights**

% Solids	Aliquot Wet Weight Range
>80 %	1.00 g
70-80 %	1.20 g
60-70 %	1.40 g
50-60 %	1.80 g
40-50 %	2.20 g
30-40 %	2.70 g

Spike Solution Chart

GFAA soil - 2mls SS # 4
ICP-OES soil - 2mls SS# 1 & 1 ml SS # 5 & 6
ICP-MS soil - 2mls SS# 1, & 1 ml SS # 5 & 6
ICP-OES water- 0.5mls SS# 1,5,6
GFAA water - 0.5 ml SS # 4
ICP-MS water - 1ml 200.8 stock sol'n & 1 ml Ag sol'n

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Columbia Analytical Services
Metals Digestion Sheet

Service Request Number(s) :
Star Lims Run No.:

Method : CLP 3010A 3020A
3005A 3050B CLP Hot Block Other-_____

Analysis for : ICP ICP-MS GFAA
Flame AA Other _____

Sample	Initial Weight (g)	Dry	Wet	Initial Volume(ml)	Final Volume (ml)	Matrix

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Time Digestion Started:_____

Lot # Acids Used: HNO3 _____ **HCl** _____ **H2O2** _____

LCSS (circle appropriate) ERA CLP Soil Lot # D045540 Other:_____

GFAA LCSW = GFLCSW, MET1-52-W, _____ mls. added
ICP LCSW = CLP-CV-1, MET1-52-A, _____ mls. added
QCP CICV-2, MET1-51-T, _____ mls. added
QCP CICV-3, MET1-51-U, _____mls. added
SS6, MET1-52-Y, _____mls. added

TCLP Spike/LCSE
TSS1 MET1-47-C _____mls. added

SPIKE INFO
SS1-MET1-52-Z,_____mls added SS4-MET1-48-A,_____mls added
SS5-MET1-54-G,_____mls added 200.8 1000ppb Stock (MS9-85-A)_____mls added
SS6-MET1-54-E,_____mls added Ag 1000ppb Stock (MS10-10-A) _____ mls added

Additional spikes:_____

Comments: _____

Analyst _____ **Date** _____

Reviewer_____ **Date** _____

metdig.xls

Digestion Procedures

Method #	GFAA	ICP-OES
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EPA 3050B	Mix sample , weigh out 1.00 g dry weight. * Add spike solution to sample. Add 5 ml DI water and 5 ml HNO3. Reflux 15 min. Cool. Add 5 ml HNO3. Cover. Reflux 30 min. NOTE: *If brown fumes are* generated, repeat the *addition of 5 mls of HNO3 after 30 min of reflux.* Reflux for 2 hrs. or to vol of 5 mls. Cool sample and add 2 mls H20 & 3 mls 30%H2O2 Cover and heat until effervescence subsides Repeat H2O2 adds twice until effervescence is minimal. Don't add > 10 mls. H2O2 **Heat to vol. of 5 mls.** Dilute to 100mls with DI water.	same same same same same same same same same same Add 10 ml of Concentrated HCl, Reflux for 15 min. Dilute to 100 mls. (to 50 mls for Flame)

GFAA NOTE: For SW-846 analysis prepare a digestate from Method 3020B for Pb & Tl analysis & a digestate from the CLFAA for As & Se.

*** Suggested Sample Aliquot Weights**

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50-60 %	1.80 g
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Spike Solution Chart

GFAA soil - 2mls SS # 4
ICP-OES soil - 2mls SS# 1 & 1 ml SS # 5 & 6
ICP-MS soil - 2mls SS# 1, & 1 ml SS # 5 & 6
ICP-OES water- 0.5mls SS# 1,5,6
GFAA water - 0.5 ml SS # 4
ICP-MS water - 1ml 200.8 stock sol'n & 1 ml Ag sol'n

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STANDARD OPERATING PROCEDURE

for

DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY (ICP)

SOP No.: MET-ICP

UNCONTROLLED

Revision: 21

June 26, 2009

Approved by: _____ Supervisor — 7/6/09 Date

_____ QA Manager — 7/6/09 Date

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_____ Laboratory Manager — 7/6/09 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY (ICP)

1 SCOPE AND APPLICATION

1.1 This procedure describes the steps taken for the analysis of soil, sludge surface water and drinking water digestates using EPA ICP methods 6010B/C, 200.7, and CLP ILM04.0 for a variety of elements. The digested samples and QC standards are all diluted in a similar acid matrix. A procedure is also given for calculation of hardness by Standard Methods 2340B.

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1.2 The Method Reporting Limits (MRLs) for common elements are listed in Table 1. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The reported MRL may be adjusted if required for specific project requirements, however, the capability of achieving other reported MRLs must be demonstrated. The Method Detection Limits (MDLs) that have been achieved are listed in Table 1. The MDL and MRL may change as annual studies are performed.

1.3 This procedure is commonly applied to DoD projects. In addition to this SOP, the user should refer to the *SOP for Department of Defense Projects Laboratory Practices and Project Management* (ADM-DOD), the DoD QSM and any applicable project QAPP.

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2 METHOD SUMMARY

2.1 A representative aliquot of sample is prepared as described in the applicable digestion SOP. The digestate is analyzed for the elements of interest using ICP spectrometry. The instrument measures characteristic emission spectra by optical spectrometry. The intensity of emission lines are monitored.

2.2 Final results are calculated using the digestion information and the results from the ICP analysis. Data is reported using standard CAS procedures and formats, or following project specific reporting specifications.

2.3 Deviations from the reference method(s): This SOP contains no deviations from the reference methods.

3 DEFINITIONS

3.1 **Analysis Sequence** - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample digestates interspersed with calibration standards.

3.2 **Independent Calibration Verification** (ICV) - ICV solutions are made from stock solutions different from the stock used to prepare calibration standards and are used to verify the validity of the standardization.

3.3 **Laboratory Control Sample** (LCS): A laboratory blank that has been fortified with target analyte and used to determine that the analysis is in control. For solids, a reference material may be used unless prohibited by project protocols.

3.4 **Matrix Spike** (MS) - In the matrix spike analysis, predetermined quantities of standard solutions of certain analytes are added to a sample matrix prior to sample digestion and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recoveries are calculated for each of the analytes detected.

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3.5 **Matrix Spike Duplicate** (MSD) - In the matrix spike duplicate analysis, predetermined quantities of standard solutions of certain analytes are added to a sample matrix prior to sample digestion and analysis. The purpose of the matrix spike duplicate is to evaluate the effects of the sample matrix on the methods used for the analyses. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the matrix spikes is calculated and used to assess analytical precision.

3.6 **Duplicate Sample** (DUP) - A laboratory duplicate is a separate field sample aliquot that is processed in an identical manner as the sample proper. The relative percent difference between the samples is calculated and used to assess analytical precision.

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3.7 **Method Blank** - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.8 **Continuing Calibration Verification Standard (CCV)** - A standard analyzed at specified intervals and used to verify the ongoing validity of the instrument calibration.

3.9 **Instrument Blank (CCB)** - The instrument blank (also called continuing calibration blank) is a volume of blank reagent of composition identical to the digestates. The purpose of the CCB is to determine the levels of contamination associated with the instrumental analysis.

3.10 **Laboratory fortified Blank (LFB)**- A laboratory blank that has been fortified with target analyte at the method reporting limit and used to determine if the laboratory can detect contaminates at the method reporting limit.

4 INTERFERENCES

4.1 Interferences from contaminated reagents must be eliminated. The purity of acids must be established by the laboratory as being high enough to eliminate the introduction of contamination above the MRL (or above ½ the RL for DoD work).

4.2 Background emission and stray light can be compensated by background correction.

4.3 Spectral overlaps resulting in interelement contributions can be corrected for by using interelement correction factors. Interelement correction factors are established for each instrument and are maintained by the analyst at the workstation.

5 SAFETY

5.1 Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

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5.2 Hydrochloric, Nitric and Hydrofluoric Acids are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. Safety glasses, lab coat and gloves should be worn while working with the solutions.

5.3 High Voltage - The power unit supplies high voltage to the RF generator which is used to form the plasma. The unit should never be opened. Exposure to high voltage can cause injury or death.

5.4 UV Light -The plasma when lit is a very intense light, and must not be viewed with the naked eye. Protective lenses are in place on the instrument. Glasses with special protective lenses are available.

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6 SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1 Samples are prepared using methods 3005A, 3010A, 3050, or CLPILM04.0 (CAS SOPs MET-3005A, MET-3010A, MET-3050, and MET-DIG). Samples are received in the ICP lab as completed digestates. Samples are stored in 50 mL plastic centrifuge tubes, 100 mL digestion vessels or in 100 mL volumetric flasks.

6.2 Water samples analyzed by EPA method 200.7 are preserved after arrival at the laboratory. These samples are held for a minimum of 16 hours and the pH verified to be <2 prior to digestion.

6.3 Soil samples are diluted prior to instrumental analysis by a factor of 2. This allows the method to meet the required 1 g of sample to 200 mL dilution during digestion.

6.4 Following analysis, digestates are stored until two weeks after all results have been reviewed and then brought to 3< pH<10 and disposed of through the sewer system.

7 APPARATUS & EQUIPMENT

7.1 Inductively Coupled Plasma Atomic Emission Spectrometer

7.1.1 Thermo Jarrell Ash ICAP-61.

7.1.2 Thermo Jarrell Ash IRIS.

7.1.3 Thermo Scientific ICAP 6500

7.2 Cross-Flow Nebulizer (TJA-61), Concentric nebulizer (IRIS & ICAP 6500).

7.3 Torches and injector tips for each ICP.

7.4 Cyclonic and/or baffled cylindrical spray chambers for each instrument.

7.5 Water coolers for each ICP (internal on the IRIS.)

7.6 Argon Humidifiers for the IRIS and ICAP 6500.

7.7 Peristaltic Pumps for each Spectrometer.

7.8 ASX-520 autosamplers for the IRIS and ICAP 6500.

7.9 RF Generators for each ICP (internal on the IRIS and ICAP 6500).

7.10 Computer system interfaced to each ICP. A compatible Windows-based data system is used to acquire, store, and perform calculations on raw data.

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8 STANDARDS, REAGENTS, & CONSUMABLE MATERIALS

8.1 Standards Preparation

8.1.1 All standards are prepared from NIST traceable or certified standards as per SOP ADM-DATANTRY *Making Entries into Logbooks and onto Benchsheets.*. Manufacturer's expiration dates are used to determine the viability of standards.

8.1.2 Calibration Standards

Calibration standards are prepared from commercially purchased single element 1000 ppm or 10,000 ppm stock standards as well as pre-mixed multi element stock standards. All standards are aliquoted using Class A volumetric pipettes, or calibrated

fixed and adjustable volume autopipetters. All dilutions are made in Class A volumetric glassware.

The standard mixes for each ICP system vary based on the requirements of each instrument (e.g. the IRIS and ICAP 6500 line switch for multiple elements and require calibration at various levels while the TJA61 does not). For the TJA61, calibration standards are prepared as outlined in Table 3. The composition of the IRIS calibration standards are outlined in Table 4. The composition of the ICAP 6500 standards are outlined in Table 5.

8.1.3 Continuing Calibration Verification (CCV) Standards

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CCV standards are analyzed at the midpoint of the calibration. For the IRIS and the ICAP 6500 these standards are produced by making a two-fold dilution of each calibration standard. The CCV standards are then run in sequence during the analytical run. For the TJA61 the two-fold dilution is accomplished by mixing equal parts of each working calibration standard to produce a single CCV standard.

8.1.4 Initial Calibration Verification (ICV) Standards

The ICV working standards are produced by direct dilution of three certified mixed stock solutions (QCP-CICV1, QCP-CICV2, and QCP-CICV3) purchased from Inorganic Ventures or another qualified vendor and various single element stock solutions from sources different than the calibration standards. The composition of these standards is outlined in Table 6.

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8.1.5 Interference Check Solutions (ICSA & ICSAB)

The ICSA and ICSAB working standards are produced by direct dilution of certified mixed stock solutions (CLPP-ICS-A and CLPP-ICS-B.) Antimony is also added to the ICSAB solution from a 1000 ppm single element stock standard. The composition of these standards is outlined in Table 7.

8.1.6 CRI/Low Level Calibration Verification

The CRI, Low Level Initial Calibration Verification (LLICV), and Low Level Continuing Calibration Verification (LLCCV) are produced by diluting 1000 or 10000ppm single stock standards into a 100X intermediate standard and then diluted 1/100 to obtain the MRL level.

8.1.7 The solutions and materials used for the LCS and matrix spikes are described in the applicable digestion SOP.

8.1.8 Standard Log

The analyte, source, initial volume, final volume, final concentration and expiration date are recorded in a standard logbook kept in the ICP lab. The operator who prepares the standard must date and initial the entry in the standards logbook. The operator also places his initials and the date prepared on the standard container. In addition to working standards used in calibration, all other standards used in the analytical run such as ICVs, MRL standards, and other project or client specific standards shall be documented in the standard logbook.

8.2 High Purity Argon.

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8.3 Capillary, rinse and peristaltic pump tubing.

8.4 17 x 100mm polypropylene test tubes.

9 PREVENTIVE MAINTENANCE

9.1 All maintenance is documented in the instrument logbook; including torch, nebulizer, and spray chamber cleaning. All instrument filters are vacuumed monthly. Dirty ICP torches and mixing chambers are soaked in aqua regia overnight, rinsed and placed in a clean dry area. The nebulizer is back flushed with acid or DI water as needed.

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9.2 IRIS-specific procedures

9.2.1 Each time the nebulizer is changed, the instrument optimization procedure is performed. A 10 ppm As standard is aspirated and the intensity counts are maximized by adjusting the depth to which the nebulizer is inserted into the spray chamber.

10 RESPONSIBILITIES

10.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2 It is the responsibility of the department supervisor/manager to document analyst training. Training and proficiency is documented in accordance with the SOP ADM-TRANDOC.

11 PROCEDURE

11.1 Operating Parameters

11.1.1 For the Thermo Jarrell Ash ICAP-61, the operating parameters are defined in the operating system *Method* file. Default operating parameters are given in file EPACLP1. However, each unique set of operating parameters is saved as a new file and the analyst must select and use the correct *Method* file for the application. Refer to the method files on the workstation for a listing of parameters for each file. The interelement correction factors to be used are established for the ICAP-61 and are saved on the workstation also. Since these parameters change with method and correction factor updates, and due to the large amount of hardcopy printout for listing these parameters, it is not practical to include the parameters in this SOP.

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11.1.2 For the Thermo Jarrell Ash IRIS, the operating parameters are defined in the operating system *Method* file. Default operating parameters are given in file GALILEO. However, each unique set of operating parameters is saved as a new file and the analyst must select and use the correct *Method* file for the application. Refer to the method files on the workstation for a listing of parameters for each file. The interelement correction factors to be used are established for the IRIS and are saved on the workstation also. Since these parameters change with method and correction factor updates, and due to the large amount of hardcopy printout for listing these parameters, it is not practical to include the parameters in this SOP.

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11.1.3 For the Thermo Scientific ICAP 6500, the operating parameters are defined in the *Method* file. Default operating parameters are given in *Tools/Options/New Method Parameters*. However, each unique set of operating parameters is saved as a new file and the analyst must select and use the correct *Method* file for the application. Refer to the method files on the workstation for a listing of parameters for each file. The interelement correction factors to be used are established for the ICAP 6500 and are saved on the workstation also. Since these parameters change with method and correction factor updates, and due to the large amount of hardcopy printout for listing these parameters, it is not practical to include the parameters in this SOP.

11.2 Calibration/Standardization

11.2.1 61-E

11.2.1.1 Plasma is ignited and instrument is allowed to warm up for at least 1 hour.

11.2.1.2 Adjust Profile. Specific directions for profile procedures are displayed on the computer screen by selecting F5-Profile from any analytical method

11.2.1.3 Analyze the Cu/Mn ratio. A solution of 1 ppm Cu, Cd, and Mn, is aspirated into the nebulizer. The Cd intensity indicates torch optimization. The CuMn ratio is calculated by the computer and is displayed in the Mn column. This indicates the nebulizer optimization. The ratio is to remain within 20% of the value when IECs were determined. The CuMn ratio is checked daily and prior to any standardization. This data is recorded in the instrument logbook.

11.2.1.4 Standardize by running a Blank and a High Standard for each element in the analytical method. Analyst will initial and date the first page of the standardization.

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11.2.2 IRIS

11.2.2.1 Plasma is ignited and instrument is allowed to warm up for at least 30 minutes.

11.2.2.2 An internal standard is used for routine analyses on this instrument. Scandium is used as the internal standard. The internal standard solution is introduced into the analyzed solutions (standards, blanks, QC, samples, etc.) at 1ug/mL.

11.2.2.3 Standardize by running a Blank and a High Standard for each element in the analytical method. Analyst will initial and date the first page of the standardization.

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11.2.2.4 The Cu/Mn ratio is calculated from Standard A. Ratio is to remain within 20% of the value when IECs were determined. This data is recorded in the instrument logbook.

11.2.3 ICAP 6500

11.2.3.1 Plasma is ignited and instrument is allowed to warm up for at least 30 minutes.

11.2.3.2 An internal standard is used for routine analyses on this instrument. Yttrium is used as the internal standard. The internal standard solution is introduced into the analyzed solutions (standards, blanks, QC, samples, etc.) at 2.64 ug/mL.

11.2.3.3 Standardize by running a Blank and a High Standard for each element in the analytical method. Analyst will initial and date the first page of the standardization.

11.2.3.4 The Cu/Mn ratio is calculated from Standard A using Mn2576. Ratio is to remain within 20% of the value when IECs were determined.

11.2.4 Standardization is completed by analyzing an ICV for each analyte to be determined. For method 200.7 the result must be within ± 5% of the true value. For method 6010B/C the result must be within ± 10% of the true value. If the ICV analysis fails, either the calibration standards or the ICV must be prepared fresh and the instrument restandardized when running method 6010C.

11.2.5 Method 6010C also requires a LLICV be analyzed at the MRL level. The result must be within ± 30% of the true value.

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11.3 Analytical Run

11.3.1 Following standardization and ICV analysis, the remainder of the run is determined by what analytical method is being performed. These are listed below.

11.3.1.1 CLP ILM04.0: ICB, CCV, CCB, CRI, ICSA, ICSAB, CCV, CCB, routine samples. The CRI, ICSA, and ICSAB will be analyzed every 20 samples. They will be labeled with an F indicating Final. Each set will be numbered in increasing order, i.e. ICSAF1, ICSAF2.

11.3.1.2 Methods 200.7 and 6010B/C: ICB, CCV, CCB, CRI, ICSA, ICSAB, routine samples.

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11.3.2 Evaluate the initial QC using the following criteria:

11.3.2.1 For methods 200.7 and 6010B/C, the following criteria apply:

- The ICB and CCB results are evaluated using method specified requirements. The following guidelines should also be used to determine acceptability:
  - For 200.7, the result should be less than 3 times the standard deviation of the mean background signal.
  - For method 6010B, the result should be less than the Method Detection Limit (MDL). In cases where the associated sample results are being reported to the Method Reporting Limit (MRL) the result may be greater than the MDL if the result does not adversely impact data quality.
  - Where project specifications allow, the result may be over the MDL if the result does not adversely impact data quality.

- The CCV immediately following standardization must verify within ± 10% of the true values with a relative standard deviation of <5% from 2 replicate integrations. For 200.7, the first CCV must verify within ± 5% with a RSD of <3% from 4 replicates. Calculate %RSD as follows:

$$\%RSD = \frac{StdDev_{CCV}}{Average_{CCV}} x\,100$$

where: $StdDev_{ccv}$ = Standard deviation of the replicate integrations
$Average_{ccv}$ = Average of the replicate CCV integrations

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- The CRI is a low level standard with concentrations at the RL. For DoD projects, the CRI standard concentrations will be equal to the project RLs and results must verify within 20% of the true value. For Method 6010C the CRI results should be within 30% of the true value.

- The ICSA is run to check the validity of the Interelement Correction Factors (IECs).

Note: DoD QSM requires this to be run at the beginning of each analytical run.

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- The ICSAB must be within 20% of the expected value for the CLPP-ICS-B elements and Sb.

11.3.2.2 The ICV, LLICV, ICB, CCV, CCB, CRI, and ICSAB must meet the criteria listed. Reanalyze any elements that fail.

11.3.2.3 For CLP, refer to SOW ILM04.0 for acceptance criteria.

11.3.3 Continuing Calibration Verification

11.3.3.1 CCVs are analyzed after every 10 samples and at the end of the analytical run. They must verify within ±10% of the expected value with a RSD of <10%.

11.3.3.2 CCBs are analyzed after every 10 samples and at the end of the analytical run. CCBs are evaluated as in section 11.3.2.1.

11.3.3.3 ICS solutions are to be run at least every 8 hours. Results for the spiked analytes in the ICSAB solution must agree within ±20% of the expected value.

11.3.3.4 Method 6010C requires a LLCCV be analyzed at the end of each analysis batch. The LLCCV is at the MRL level and must verify within ± 30% of the true value. Reanalyze any elements to be reported at low levels that are bracketed by the LLCCV if the standard fails.

11.3.3.5 If the CCV, CCB or ICS solutions fail, reanalyze any elements to be reported.

12 QA/QC REQUIREMENTS

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12.1 Initial Precision and Recovery Validation

The accuracy and precision of the procedure must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made. To do this, four LCS aliquots are prepared and analyzed. The average percent recovery for each analyte must meet LCS criteria and the RSD< 30%.

12.2 Method Detection Limits

12.2.1 A Method Detection Limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates at a level near or below the MRL. Follow the procedures in Section 11 to analyze the samples. Refer to the CAS SOP for The Determination of Method Detection Limits (ADM-MDL.)

12.2.2 Calculate the average concentration found (x) and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. MDL's must be performed annually or whenever there is a significant change in the background or instrument response.

12.3 Lower Limit of Quantitation Check

For Method 6010C and drinking waters by method 200.7 a Lower Limit of Quantitation Check (LLQC) sample must be analyzed after establishing the MRL and as needed afterward to demonstrate the desired detection capability. The LLQC sample must be carried through the entire preparation and analytical procedure. Lower limits of quantitation are verified when all analytes are detected within 30% of their true value.

12.4 Linear Dynamic Range

The upper limit of the LDR must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing at least three succeeding higher standard concentrations of the analyte until the observed analyte concentration is no more than 10% above or below the stated concentration of the standard. Determined LDRs must be documented and kept on file. The LDR which may be used for the analysis of samples should be judged by the analyst from the resulting data. Sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified quarterly or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

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12.5 Instrument Detection Limit

On a quarterly basis, the instrument detection limits for all analytes are determined as per procedures outlined in ILM04.0 (Section E, paragraph 10, 12 resp.). IDLs are determined using blanks and this data is kept on file.

12.6 Interelement Correction Factors

Semi-annually, instrument interferences are calculated as per ILM04.0 (Section E, paragraph 11) and Method 6010B/C. During the course of routine work, other interferences may be found. They are verified by the operator during the analytical run and data is manually corrected. Copies of this data are kept on file.

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12.7 Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the *SOP for Sample Batches*. The Quality Assurance Manual (Section 12) provides guidance for interpretation of QC results. Ongoing QC samples include:

12.7.1 Each sample preparation batch must have a method blank associated with it. The method blank result should be < MRL. If the method blank is found to be contaminated, it may be reported if the concentration in the associated samples is at least 20 times the amount found in the method blank, otherwise the redigest the batch. For Method 6010C, the method blank may be reported if the concentration in the associated samples is at least 10 times the amount found in the method blank.

Note: DoD QSM requires contamination in the MB be <1/2 the RL or < 1/10 any sample amount.

12.7.2 A Laboratory Control Sample (LCS) is digested one per batch, or per 20 samples. For method 200.7, the LCS recovery criteria is 85-115% for water samples. For method 6010B/C, the control limits are derived from lab data and are listed in

Table 2. These limits should be no wider than 80-120%. For soil samples, the recovery must fall within the ranges specified for the reference material. For CLP, use the prescribed limits for the SOW in use. In all cases, project-specific QC limits may be required. If the LCS fails the acceptance criteria, redigest the batch of samples. For specifics on the preparation and composition of LCS samples refer to the appropriate digestion SOP.

12.7.3 A Duplicate sample is digested one per batch, or per 20 samples (i.e. 5%) for 6010B/C analysis, or per 10 samples (i.e. 10%) for 200.7 analysis. The default criteria of <20% RPD for water samples may be used if statistically generated criteria is broader or insufficient points are available for accurate statistical limits. Currently, statistically generated criteria are broader and the default is used for all elements but Manganese, for which the limit is 17% RPD. The RPD criteria is <30% for soil samples for methods 200.7 and 6010B. The RPD criteria is <20% for method 6010C. Criteria are subject to change as statistical data are generated. If the RPD is outside acceptance limits, either redigest the sample batch or flag the data appropriately, depending on the physical nature of the samples (e.g. non-homogenous).

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12.7.4 A Laboratory fortified Blank (LFB) at the MRL is digested and analyzed with every batch of drinking water samples (method 200.7). The default acceptance criteria of 50-150% are to be used until sufficient data points are acquired to calculate in-house control limits.

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12.7.5 A Matrix Spike sample is digested one per batch, or per 20 samples (i.e. 5%) for 6010B/C analysis, or per 10 samples (i.e. 10%) for 200.7 analysis. Where specified by project requirements, a matrix spike duplicate may be required. Matrix spike recovery criteria for method 200.7 is 70-130% for both water and soil samples. For 6010B, the control limits are derived from lab data and are listed in Table 2. For 6010C, Table 2 is used unless the control limits are broader than the default criteria of 75-125% in which case the default criteria are used. For CLP, use the prescribed limits for the SOW in use. In all cases, project-specific QC limits may be required. If the recovery is outside acceptance limits, either redigest the sample batch or flag the data appropriately, depending on the physical nature of the samples (e.g. non-homogenous). If the sample concentration is >4x the spike level, no action is required and data is flagged accordingly. For specifics on the preparation and composition of matrix spike solutions refer to the appropriate digestion SOP.

12.7.6 The analyst must run a dilution test (1:5 dilution) to test for matrix interference if there is a reason to suspect such an interference and for all tier III deliverables. For Method 6010C, a dilution test must be run on each preparation batch and agree within ± 10% of the original result.

12.7.7 If the analyte concentration is too low to permit a dilution test, then a post digestion spike may be performed. For Method 6010C, a post-spike is performed on all preparation batches. This spike should be recovered to within 80-120% of the true value.

Note: DoD QSM recovery acceptance limits are 75-125%.

12.7.8 Additional QC measures include control charting and compiling of QC data for generation of control limits.

12.8 CLP analyses are performed as per the QA/QC guidelines in the most current CLP SOW.

13 DATA REDUCTION, REVIEW, AND REPORTING

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13.1 Calculate sample results using the data system printouts and digestion information. The digestion and dilution information is entered into the data system. The data system then uses the calculations below to generate a sample result. The wavelengths used to quantify each metal are summarized in Table 8 for the TJA61, Table 9 for the IRIS and Table 10 for the ICAP6500.

Aqueous samples are reported in µg/L:

$$\mu g/L\,(Sample) = C^{*}\ x\ Digestion\,Dilution\,Factor\ x\ Post\ Digestion\,Dilution\,Factor \times 1000 \mu g/mg$$

Solid samples are reported in mg/Kg:

$$mg/Kg\,(Sample) = C^{*}\ x\ Post\,Digestion\,Dilution\,Factor\ x\ \frac{Digestion\,Vol\,(ml)}{Sample\,wt.\,(g)}\ x\ \frac{1L}{1000ml}\ x\ \frac{1000g}{1Kg}$$

C*= Concentration of analyte as measured at the instrument in mg/L (in digestate).

13.2 If total hardness is to be reported, use Calcium and Magnesium results to calculate as follows. For reporting calcium hardness, use only the calcium portion of the equation.

$$Hardness,\ mg\ equivalent\ CaCO_3/L = 2.497[Ca, mg/L] + 4.118[Mg, mg/L]$$

13.3 A daily run log of all samples analyzed is maintained. All CLP data should be printed and stored after operator has checked for evenness of burns. A copy of this document will go with each package of Tier III or higher data run that day.

13.4 Data Review and Reporting

13.4.1 It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified in section 12. The data is then placed in a work order file until complete. When the work order is complete, a report is generated. A final review is performed and the data is delivered to the project management department.

14 CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

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Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the *SOP for Corrective Action* for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the *SOP for Report Generation* or in project-specific requirements.

15 METHOD PERFORMANCE

This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional available method performance data.

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The method detection limit (MDL) is established using the procedure described in the SOP for *The Determination of Method Detection Limits and Limits of Detection* (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16 POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible and within method requirements. Standards are prepared in volumes consistent with the laboratory use in order to minimize the volume of expired standards to be disposed. The threat to the environment from reagents used in this method may be minimized when recycled or disposed of properly.

17 WASTE MANAGEMENT

17.1 The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2 This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 3-10 prior to disposal down the drain. The

neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the CAS EH&S Manual for details.

18 TRAINING

18.1 Refer to the *SOP for Documentation of Training* for standard procedures.

18.2 Training outline

18.2.1 Review literature (see references section). Review the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

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18.2.2 The next training step is to assist in the procedure under the guidance of an experienced analyst for a period of approximately two weeks. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

18.2.3 Perform initial precision and recovery (IPR) study as described in Section 12.1 for water samples. Summaries of the IPR are reviewed and signed by the supervisor.

18.3 Training and proficiency is documented in accordance with the SOP ADM-TRANDOC.

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19 CHANGES SINCE THE LAST REVISION

19.1 Sec 1.1 Added method 6010C
19.2 Sec 1.3 Removed AFCEE reference
19.3 Updated Sec 5.1
19.4 Sec 5.2 Added hydrofluoric acid
19.5 Sec 6.2 Added method 200.7 hold time for pH
19.6 Sec 6.4 Added disposal requirements
19.7 Sec 7.1.3 Added Thermo Scientific ICAP 6500
19.8 Sec 8.1.1 Updated SOP reference to ADM-DATANTRY
19.9 Sec 8.1.2 Updated calibration Tables 3-5
19.10 Sec 9.2.2 Removed
19.11 Sec 11.1.3 Added parameters for ICAP 6500
19.12 Sec 11.2 Re-wrote to reflect current practices
19.13 Sec 11.3 Re-wrote to reflect current practices
19.14 Sec 12.3 Added method 6010C LLQC requirement
19.15 Sec 12.7 Added method 6010C requirement
19.16 Updated table 8-10
19.17 Sec 13.2.2 Deleted
19.18 Sec 19 Included method 6010C in references
19.19 Sec 3.1 and 12.7.4 Added LFB for drinking waters.

20 REFERENCES

20.1 USEPA, Contract Laboratory Program, SOW #ILM04.0

20.2 Thermo Jarrell Ash ICAP61 Manual

20.3 USEPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Update III, Method 6010B, Revision 2, December 1996.

20.4 USEPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Update III, Method 6010C, Revision 3, February 2007.

20.5 USEPA, Methods for Determination of Metals in Environmental Samples, Supplement I, EPA/600/R-94/111, Method 200.7, Revision 4.4, May 1994.

20.6 *Hardness by Calculation, Method 2340B,* Standard Methods for the Examination of Water and Wastewater, 20th ed., 1998.

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TABLE 1

Selected Elements, Method Reporting Limits, and Method Detection Limits

Element	Matrix	Digestion	Standard MRL	MDL	Units	Method
Aluminum	Water	CLP	50	30	ug/L	6010 / 200.7
Antimony	Water	CLP	50	30	ug/L	6010 / 200.7
Arsenic	Water	CLP	100	20	ug/L	6010 / 200.7
Barium	Water	CLP	5	0.7	ug/L	6010 / 200.7
Beryllium	Water	CLP	5	0.4	ug/L	6010 / 200.7
Boron	Water	CLP	50	9	ug/L	6010 / 200.7
Cadmium	Water	CLP	5	3	ug/L	6010 / 200.7
Calcium	Water	CLP	50	30	ug/L	6010 / 200.7
Chromium	Water	CLP	5	3	ug/L	6010 / 200.7
Cobalt	Water	CLP	10	3	ug/L	6010 / 200.7
Copper	Water	CLP	10	7	ug/L	6010 / 200.7
Iron	Water	CLP	20	3	ug/L	6010 / 200.7
Lead	Water	CLP	50	20	ug/L	6010 / 200.7
Lithium	Water	CLP	10	6	ug/L	6010 / 200.7
Magnesium	Water	CLP	20	0.7	ug/L	6010 / 200.7
Manganese	Water	CLP	5	0.6	ug/L	6010 / 200.7
Molybdenum	Water	CLP	10	6	ug/L	6010 / 200.7
Nickel	Water	CLP	20	20	ug/L	6010 / 200.7
Phosphorus	Water	CLP	200	200	ug/L	6010 / 200.7
Potassium	Water	CLP	2000	900	ug/L	6010 / 200.7
Selenium	Water	CLP	100	40	ug/L	6010 / 200.7
Silicon	Water	CLP	400	40	ug/L	6010 / 200.7
Silver	Water	CLP	10	6	ug/L	6010 / 200.7
Sodium	Water	CLP	100	50	ug/L	6010 / 200.7
Strontium	Water	CLP	10	0.5	ug/L	6010 / 200.7
Thallium	Water	CLP	200	50	ug/L	6010 / 200.7
Tin	Water	CLP	50	20	ug/L	6010 / 200.7
Titanium	Water	CLP	10	3	ug/L	6010 / 200.7
Vanadium	Water	CLP	10	5	ug/L	6010 / 200.7
Zinc	Water	CLP	10	7	ug/L	6010 / 200.7

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TABLE 1 continued

Selected Elements, Method Reporting Limits, and Method Detection Limits

Element	Matrix	Digestion	Standard MRL	MDL	Units	Method
Aluminum	Water	3010A	50	50	ug/L	6010
Antimony	Water	3010A	50	20	ug/L	6010
Arsenic	Water	3010A	100	30	ug/L	6010
Barium	Water	3010A	5	2	ug/L	6010
Beryllium	Water	3010A	5	0.2	ug/L	6010
Boron	Water	3010A	50	30	ug/L	6010
Cadmium	Water	3010A	5	3	ug/L	6010
Calcium	Water	3010A	50	10	ug/L	6010
Chromium	Water	3010A	5	2	ug/L	6010
Cobalt	Water	3010A	10	4	ug/L	6010
Copper	Water	3010A	10	6	ug/L	6010
Iron	Water	3010A	20	3	ug/L	6010
Lead	Water	3010A	50	30	ug/L	6010
Lithium	Water	3010A	10	8	ug/L	6010
Magnesium	Water	3010A	20	0.5	ug/L	6010
Manganese	Water	3010A	5	2	ug/L	6010
Molybdenum	Water	3010A	10	4	ug/L	6010
Nickel	Water	3010A	20	20	ug/L	6010
Phosphorus	Water	3010A	200	90	ug/L	6010
Potassium	Water	3010A	2000	1000	ug/L	6010
Selenium	Water	3010A	100	40	ug/L	6010
Silicon	Water	3010A	400	200	ug/L	6010
Silver	Water	3010A	10	9	ug/L	6010
Sodium	Water	3010A	100	60	ug/L	6010
Strontium	Water	3010A	10	0.5	ug/L	6010
Thallium	Water	3010A	200	60	ug/L	6010
Tin	Water	3010A	50	20	ug/L	6010
Titanium	Water	3010A	10	2	ug/L	6010
Vanadium	Water	3010A	10	4	ug/L	6010
Zinc	Water	3010A	10	3	ug/L	6010

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TABLE 1 continued

Selected Elements, Method Reporting Limits, and Method Detection Limits

Element	Matrix	Digestion	Standard MRL	MDL	Units	Method
Aluminum	Soil	3050B	10	7	mg/Kg	6010 / 200.7
Antimony	Soil	3050B	10	8	mg/Kg	6010 / 200.7
Arsenic	Soil	3050B	20	5	mg/Kg	6010 / 200.7
Barium	Soil	3050B	2	0.2	mg/Kg	6010 / 200.7
Beryllium	Soil	3050B	1	0.03	mg/Kg	6010 / 200.7
Boron	Soil	3050B	10	2	mg/Kg	6010 / 200.7
Cadmium	Soil	3050B	1	1	mg/Kg	6010 / 200.7
Calcium	Soil	3050B	10	2	mg/Kg	6010 / 200.7
Chromium	Soil	3050B	2	0.7	mg/Kg	6010 / 200.7
Cobalt	Soil	3050B	2	0.6	mg/Kg	6010 / 200.7
Copper	Soil	3050B	2	0.7	mg/Kg	6010 / 200.7
Iron	Soil	3050B	4	0.6	mg/Kg	6010 / 200.7
Lead	Soil	3050B	20	7	mg/Kg	6010 / 200.7
Magnesium	Soil	3050B	4	2	mg/Kg	6010 / 200.7
Manganese	Soil	3050B	2	0.07	mg/Kg	6010 / 200.7
Molybdenum	Soil	3050B	2	0.9	mg/Kg	6010 / 200.7
Nickel	Soil	3050B	4	3	mg/Kg	6010 / 200.7
Phosphorus	Soil	3050B	40	20	mg/Kg	6010 / 200.7
Potassium	Soil	3050B	200	200	mg/Kg	6010 / 200.7
Selenium	Soil	3050B	40	6	mg/Kg	6010 / 200.7
Silver	Soil	3050B	2	2	mg/Kg	6010 / 200.7
Sodium	Soil	3050B	20	20	mg/Kg	6010 / 200.7
Strontium	Soil	3050B	2	2	mg/Kg	6010 / 200.7
Thallium	Soil	3050B	40	20	mg/Kg	6010 / 200.7
Tin	Soil	3050B	10	5	mg/Kg	6010 / 200.7
Titanium	Soil	3050B	2	0.5	mg/Kg	6010 / 200.7
Vanadium	Soil	3050B	2	2	mg/Kg	6010 / 200.7
Zinc	Soil	3050B	2	0.9	mg/Kg	6010 / 200.7

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TABLE 1 continued

Selected Elements, Method Reporting Limits, and Method Detection Limits

Element	Matrix	Digestion	Standard MRL (1)	MDL (1)	Units	Method
Aluminum	Tissue	PSEP	10	6	mg/Kg	6010
Antimony	Tissue	PSEP	5	3	mg/Kg	6010
Barium	Tissue	PSEP	1	0.2	mg/Kg	6010
Beryllium	Tissue	PSEP	1	0.02	mg/Kg	6010
Boron	Tissue	PSEP	5	0.6	mg/Kg	6010
Cadmium	Tissue	PSEP	0.5	0.3	mg/Kg	6010
Calcium	Tissue	PSEP	40	20	mg/Kg	6010
Chromium	Tissue	PSEP	1	0.6	mg/Kg	6010
Cobalt	Tissue	PSEP	1	0.4	mg/Kg	6010
Copper	Tissue	PSEP	1	0.5	mg/Kg	6010
Iron	Tissue	PSEP	4	4	mg/Kg	6010
Lead	Tissue	PSEP	5	3	mg/Kg	6010
Magnesium	Tissue	PSEP	4	3	mg/Kg	6010
Manganese	Tissue	PSEP	0.5	0.2	mg/Kg	6010
Molybdenum	Tissue	PSEP	1	0.7	mg/Kg	6010
Nickel	Tissue	PSEP	2	1	mg/Kg	6010
Phosphorus	Tissue	PSEP	20	10	mg/Kg	6010
Potassium	Tissue	PSEP	200	200	mg/Kg	6010
Sodium	Tissue	PSEP	10	8	mg/Kg	6010
Strontium	Tissue	PSEP	1	0.2	mg/Kg	6010
Tin	Tissue	PSEP	5	2	mg/Kg	6010
Vanadium	Tissue	PSEP	1	0.4	mg/Kg	6010
Zinc	Tissue	PSEP	1	0.4	mg/Kg	6010

(1) Dry Weight Basis

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TABLE 2

QC Limits

Element	Matrix	Digestion	Method	6010		200.7		RPD
				LCS Limit	MS Limit	LCS Limit	MS Limit	Limit
Aluminum	Water	CLP	6010 / 200.7	93-111	83-119	85-115	70-130	20
Antimony	Water	CLP	6010 / 200.7	95-111	82-119	85-115	70-130	20
Arsenic	Water	CLP	6010 / 200.7	94-113	84-116	85-115	70-130	20
Barium	Water	CLP	6010 / 200.7	93-114	76-127	85-115	70-130	20
Beryllium	Water	CLP	6010 / 200.7	93-112	82-116	85-115	70-130	20
Boron	Water	CLP	6010 / 200.7	89-112	76-132	85-115	70-130	20
Cadmium	Water	CLP	6010 / 200.7	92-116	71-145	85-115	70-130	20
Calcium	Water	CLP	6010 / 200.7	94-111	75-125	85-115	70-130	20
Chromium	Water	CLP	6010 / 200.7	95-113	85-119	85-115	70-130	20
Cobalt	Water	CLP	6010 / 200.7	94-113	84-119	85-115	70-130	20
Copper	Water	CLP	6010 / 200.7	93-112	83-115	85-115	70-130	20
Iron	Water	CLP	6010 / 200.7	94-113	58-142	85-115	70-130	20
Lead	Water	CLP	6010 / 200.7	94-114	81-122	85-115	70-130	20
Lithium	Water	CLP	6010 / 200.7	85-115	70-130	85-115	70-130	20
Magnesium	Water	CLP	6010 / 200.7	91-112	75-125	85-115	70-130	20
Manganese	Water	CLP	6010 / 200.7	95-112	82-122	85-115	70-130	20
Molybdenum	Water	CLP	6010 / 200.7	93-114	78-126	85-115	70-130	20
Nickel	Water	CLP	6010 / 200.7	93-115	82-122	85-115	70-130	20
Phosphorus	Water	CLP	6010 / 200.7	85-115	70-130	85-115	70-130	20
Potassium	Water	CLP	6010 / 200.7	89-117	75-125	85-115	70-130	20
Selenium	Water	CLP	6010 / 200.7	92-112	79-120	85-115	70-130	20
Silicon	Water	CLP	6010 / 200.7	85-115	70-130	85-115	70-130	20
Silver	Water	CLP	6010 / 200.7	94-111	80-119	85-115	70-130	20
Sodium	Water	CLP	6010 / 200.7	92-116	75-125	85-115	70-130	20
Strontium	Water	CLP	6010 / 200.7	85-115	70-130	85-115	70-130	20
Thallium	Water	CLP	6010 / 200.7	88-120	62-128	85-115	70-130	20
Tin	Water	CLP	6010 / 200.7	80-120	75-125	85-115	70-130	20
Titanium	Water	CLP	6010 / 200.7	85-115	70-130	85-115	70-130	20
Vanadium	Water	CLP	6010 / 200.7	94-110	87-116	85-115	70-130	20
Zinc	Water	CLP	6010 / 200.7	94-111	83-117	85-115	70-130	20

Where recovery limits are not calculated due to insufficient data points, the required method 200.7 limits are used.

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TABLE 2 continued

QC Limits

Element	Matrix	Digestion	Method	LCS Limit	MS Limit	RPD Limit
Aluminum	Water	3010A	6010	93-111	83-119	20
Antimony	Water	3010A	6010	95-111	82-119	20
Arsenic	Water	3010A	6010	94-113	84-116	20
Barium	Water	3010A	6010	93-114	76-127	20
Beryllium	Water	3010A	6010	93-112	82-116	20
Boron	Water	3010A	6010	89-112	76-132	20
Cadmium	Water	3010A	6010	92-116	71-145	20
Calcium	Water	3010A	6010	94-111	75-125	20
Chromium	Water	3010A	6010	95-113	85-119	20
Cobalt	Water	3010A	6010	94-113	84-119	20
Copper	Water	3010A	6010	93-112	83-115	20
Iron	Water	3010A	6010	94-113	58-142	20
Lead	Water	3010A	6010	94-114	81-122	20
Lithium	Water	3010A	6010	85-115	70-130	20
Magnesium	Water	3010A	6010	91-112	75-125	20
Manganese	Water	3010A	6010	95-112	82-122	20
Molybdenum	Water	3010A	6010	93-114	78-126	20
Nickel	Water	3010A	6010	93-115	82-122	20
Phosphorus	Water	3010A	6010	85-115	70-130	20
Potassium	Water	3010A	6010	89-117	75-125	20
Selenium	Water	3010A	6010	92-112	79-120	20
Silicon	Water	3010A	6010	85-115	70-130	20
Silver	Water	3010A	6010	94-111	80-119	20
Sodium	Water	3010A	6010	92-116	75-125	20
Strontium	Water	3010A	6010	85-115	70-130	20
Thallium	Water	3010A	6010	88-120	62-128	20
Tin	Water	3010A	6010	80-120	75-125	20
Titanium	Water	3010A	6010	85-115	70-130	20
Vanadium	Water	3010A	6010	94-110	87-116	20
Zinc	Water	3010A	6010	94-111	83-117	20

Where recovery limits are not calculated due to insufficient data points, the required method 200.7 limits are used.

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TABLE 2 continued

QC Limits

Element	Matrix	Digestion	Method	6010		200.7		RPD
				LCS Limit	MS Limit	LCS Limit	MS Limit	Limit
Aluminum	Soil	3050B	6010 / 200.7	61-152	75-125	61-152	70-130	30
Antimony	Soil	3050B	6010 / 200.7	36-147	10-128	36-147	70-130	30
Arsenic	Soil	3050B	6010 / 200.7	81-123	49-139	81-123	70-130	30
Barium	Soil	3050B	6010 / 200.7	77-139	79-125	77-139	70-130	30
Beryllium	Soil	3050B	6010 / 200.7	88-124	82-116	88-124	70-130	30
Boron	Soil	3050B	6010 / 200.7	37-174	33-164	37-174	70-130	30
Cadmium	Soil	3050B	6010 / 200.7	92-125	58-144	92-125	70-130	30
Calcium	Soil	3050B	6010 / 200.7	80-131	75-125	80-131	70-130	30
Chromium	Soil	3050B	6010 / 200.7	93-123	22-184	93-123	70-130	30
Cobalt	Soil	3050B	6010 / 200.7	78-134	84-114	78-134	70-130	30
Copper	Soil	3050B	6010 / 200.7	85-118	51-147	85-118	70-130	30
Iron	Soil	3050B	6010 / 200.7	64-154	75-125	64-154	70-130	30
Lead	Soil	3050B	6010 / 200.7	82-131	49-148	82-131	70-130	30
Magnesium	Soil	3050B	6010 / 200.7	83-130	75-125	83-130	70-130	30
Manganese	Soil	3050B	6010 / 200.7	83-130	25-178	83-130	70-130	30
Molybdenum	Soil	3050B	6010 / 200.7	84-134	75-116	84-134	70-130	30
Nickel	Soil	3050B	6010 / 200.7	92-123	74-126	92-123	70-130	30
Phosphorus	Soil	3050B	6010 / 200.7	85-115*	75-125	85-115*	70-130	30
Potassium	Soil	3050B	6010 / 200.7	80-131	75-125	80-131	70-130	30
Selenium	Soil	3050B	6010 / 200.7	68-166	68-127	68-166	70-130	30
Silver	Soil	3050B	6010 / 200.7	90-118	44-138	90-118	70-130	30
Sodium	Soil	3050B	6010 / 200.7	87-121	75-125	87-121	70-130	30
Strontium	Soil	3050B	6010 / 200.7	80-120	75-125	80-120	70-130	30
Thallium	Soil	3050B	6010 / 200.7	27-171	25-153	27-171	70-130	30
Tin	Soil	3050B	6010 / 200.7	80-120	75-125	80-120	70-130	30
Titanium	Soil	3050B	6010 / 200.7	40-160	75-125	40-160	70-130	30
Vanadium	Soil	3050B	6010 / 200.7	88-121	77-122	88-121	70-130	30
Zinc	Soil	3050B	6010 / 200.7	88-126	32-168	88-126	70-130	30

* Blank Spike limit. No value available for D045540.
LCS Material: ERA PrioityPollutant/CLP Soils Lot D045540

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TABLE 2 continued

QC Limits

Element	Matrix	Digestion	Method	LCS Limit [2]	MS Limit	RPD Limit [2]
Aluminum	Tissue	PSEP	6010	85-115	70-130	30
Antimony	Tissue	PSEP	6010	85-115	70-130	30
Barium	Tissue	PSEP	6010	85-115	70-130	30
Beryllium	Tissue	PSEP	6010	85-115	70-130	30
Boron	Tissue	PSEP	6010	85-115	70-130	30
Cadmium	Tissue	PSEP	6010	85-115	70-130	30
Calcium	Tissue	PSEP	6010	85-115	70-130	30
Chromium	Tissue	PSEP	6010	85-115	70-130	30
Cobalt	Tissue	PSEP	6010	85-115	70-130	30
Copper	Tissue	PSEP	6010	85-115	70-130	30
Iron	Tissue	PSEP	6010	85-115	70-130	30
Lead	Tissue	PSEP	6010	85-115	70-130	30
Magnesium	Tissue	PSEP	6010	85-115	70-130	30
Manganese	Tissue	PSEP	6010	85-115	70-130	30
Molybdenum	Tissue	PSEP	6010	85-115	70-130	30
Nickel	Tissue	PSEP	6010	85-115	70-130	30
Phosphorus	Tissue	PSEP	6010	85-115	70-130	30
Potassium	Tissue	PSEP	6010	85-115	70-130	30
Sodium	Tissue	PSEP	6010	85-115	70-130	30
Strontium	Tissue	PSEP	6010	85-115	70-130	30
Tin	Tissue	PSEP	6010	85-115	70-130	30
Vanadium	Tissue	PSEP	6010	85-115	70-130	30
Zinc	Tissue	PSEP	6010	85-115	70-130	30

[1] Dry Weight Basis
[2] List limits are for "Blank Spike" analysis. SRM samples have separate limits.

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TABLE 3
Standard 2 for TJA-61 ICP-OES

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Antimony	Elemental Stock	1000	5	500	10
Barium	Elemental Stock	1000	0.5	500	1
Boron	Elemental Stock	1000	5	500	10
Cadmium	Elemental Stock	1000	0.5	500	1
Chromium	Elemental Stock	1000	5	500	10
Lithium	Elemental Stock	1000	2.5	500	5
Nickel	Elemental Stock	1000	5	500	10
Phosphorus	Elemental Stock	10000	2.5	500	50
Potassium	Elemental Stock	10000	2.5	500	50
Silicon	Elemental Stock	10000	0.5	500	10
Sodium	Elemental Stock	10000	2.5	500	50
Strontium	Elemental Stock	1000	0.5	500	1
Thallium	Elemental Stock	1000	5	500	10
Tin	Elemental Stock	1000	5	500	10
Hydrochloric Acid	-	-	25	500	5%
Nitric Acid	-	-	5	500	1%

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Standard 3 for TJA-61 ICP-OES

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	Elemental Stock	10000	0.5	500	10
Arsenic	Elemental Stock	1000	2	500	4
Beryllium	Elemental Stock	1000	0.5	500	1
Calcium	Elemental Stock	10000	0.5	500	10
Cobalt	Elemental Stock	1000	0.5	500	1
Copper	Elemental Stock	1000	0.5	500	1
Iron	Elemental Stock	10000	0.5	500	10
Lead	Elemental Stock	1000	5	500	10
Magnesium	Elemental Stock	10000	0.5	500	10
Manganese	Elemental Stock	1000	0.5	500	1
Molybdenum	Elemental Stock	1000	0.5	500	1
Selenium	Elemental Stock	1000	2	500	4
Titanium	Elemental Stock	1000	0.5	500	1
Vanadium	Elemental Stock	1000	0.5	500	1
Zinc	Elemental Stock	1000	2.5	500	5
Hydrochloric Acid	-	-	25	500	5%
Nitric Acid	-	-	5	500	1%

TABLE 4
Standard A for IRIS ICP-OES

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Antimony	(1)	100	10	1000	1.0
Antimony	Elemental Stock	1000	4	1000	4.0
Arsenic	(1)	100	10	1000	1.0
Arsenic	Elemental Stock	1000	4	1000	4.0
Boron	(1)	100	10	1000	1.0
Cadmium	(1)	100	10	1000	1.0
Calcium	(1)	100	10	1000	1.0
Calcium	Elemental Stock	10000	0.4	1000	4.0
Chromium	(1)	100	10	1000	1.0
Cobalt	(1)	100	10	1000	1.0
Copper	(1)	100	10	1000	1.0
Iron	(1)	100	10	1000	1.0
Lead	(1)	100	10	1000	1.0
Lead	Elemental Stock	1000	4	1000	4.0
Magnesium	(1)	100	10	1000	1.0
Magnesium	Elemental Stock	1000	3	1000	3.0
Manganese	(1)	100	10	1000	1.0
Manganese	Elemental Stock	1000	1	1000	1.0
Molybdenum	(1)	100	10	1000	1.0
Molybdenum	Elemental Stock	1000	1	1000	1.0
Nickel	(1)	100	10	1000	1.0
Selenium	(1)	100	10	1000	1.0
Selenium	Elemental Stock	1000	4	1000	4.0
Silver	(1)	100	10	1000	1.0
Tin	Elemental Stock	1000	5	1000	5.0
Thallium	(1)	100	10	1000	1.0
Thallium	Elemental Stock	1000	9	1000	9.0
Titanium	(1)	100	10	1000	1.0
Vanadium	(1)	100	10	1000	1.0
Zinc	(1)	100	10	1000	1.0
Hydrochloric Acid	-	-	50	1000	5%
Nitric Acid	-	-	10	1000	1%

(1) Mixed Standard, QCS-26

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TABLE 4 continued
Standard B for IRIS ICP-OES

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	Elemental Stock	10000	1	1000	10
Barium	Elemental Stock	10000	0.5	1000	5.0
Beryllium	Elemental Stock	1000	0.1	1000	0.1
Calcium	Elemental Stock	10000	5	1000	50
Iron	Elemental Stock	10000	5	1000	50
Lithium	Elemental Stock	1000	1	1000	1.0
Magnesium	Elemental Stock	10000	5	1000	50
Manganese	Elemental Stock	1000	20	1000	20
Phosphorus	Elemental Stock	10000	2	1000	20
Potassium	Elemental Stock	10000	2	1000	20
Silicon	Elemental Stock	10000	0.5	1000	5
Sodium	Elemental Stock	10000	2	1000	20
Strontium	Elemental Stock	1000	1	1000	1.0
HCl	-	-	50	1000	5%
HNO3	-	-	10	1000	1%

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TABLE 5
Standard A for ICAP 6500 ICP-OES

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Antimony	(1)	100	5	1000	0.5
Beryllium	(1)	100	5	1000	0.5
Boron	(1)	100	5	1000	0.5
Cadmium	(1)	100	5	1000	0.5
Calcium	(1)	100	5	1000	0.5
Chromium	(1)	100	5	1000	0.5
Cobalt	(1)	100	5	1000	0.5
Copper	(1)	100	5	1000	0.5
Iron	(1)	100	5	1000	0.5
Lead	(1)	100	5	1000	0.5
Magnesium	(1)	100	5	1000	0.5
Manganese	(1)	100	5	1000	0.5
Molybdenum	(1)	100	5	1000	0.5
Nickel	(1)	100	5	1000	0.5
Selenium	(1)	100	5	1000	0.5
Silver	(1)	100	5	1000	0.5
Tin	Elemental Stock	1000	0.5	1000	0.5
Thallium	(1)	100	5	1000	0.5
Titanium	(1)	100	5	1000	0.5
Vanadium	(1)	100	5	1000	0.5
Zinc	(1)	100	5	1000	0.5
Hydrochloric Acid	-	-	50	1000	5%
Nitric Acid	-	-	10	1000	1%

(1) Mixed Standard, QCS-26

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TABLE 5 continued
Standard B for ICAP 6500 ICP-OES

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	Elemental Stock	10000	2	1000	20
Arsenic	Elemental Stock	1000	2	1000	2
Barium	Elemental Stock	10000	2	1000	20
Calcium	Elemental Stock	10000	2	1000	20
Iron	Elemental Stock	10000	2	1000	20
Lithium	Elemental Stock	1000	2	1000	2
Magnesium	Elemental Stock	10000	2	1000	20
Phosphorus	Elemental Stock	10000	2	1000	20
Potassium	Elemental Stock	10000	2	1000	20
Silicon	Elemental Stock	10000	2	1000	20
Sodium	Elemental Stock	10000	2	1000	20
Strontium	Elemental Stock	1000	2	1000	2
HCl	-	-	50	1000	5%
HNO3	-	-	10	1000	1%

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TABLE 6
ICP ICV Standards

ICV1 Solution

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	QCP-CICV-1	1000	2.5	500	5.0
Antimony	QCP-CICV-2	500	2.5	500	2.5
Arsenic	QCP-CICV-3	500	2.5	500	2.5
Barium	QCP-CICV-1	1000	2.5	500	5.0
Beryllium	QCP-CICV-1	25	2.5	500	0.125
Cadmium	QCP-CICV-3	250	2.5	500	1.25
Calcium	QCP-CICV-1	2500	2.5	500	12.5
Chromium	QCP-CICV-1	100	2.5	500	0.5
Cobalt	QCP-CICV-1	250	2.5	500	1.25
Copper	QCP-CICV-1	125	2.5	500	0.625
Iron	QCP-CICV-1	500	2.5	500	2.5
Lead	QCP-CICV-3	500	2.5	500	2.5
Magnesium	QCP-CICV-1	2500	2.5	500	12.5
Manganese	QCP-CICV-1	250	2.5	500	1.25
Molybdenum	Elemental Stock	1000	1.0	500	2.0
Nickel	QCP-CICV-1	250	2.5	500	1.25
Potassium	QCP-CICV-1	2500	2.5	500	12.5
Selenium	QCP-CICV-3	500	2.5	500	2.5
Silver	QCP-CICV-1	125	2.5	500	0.625
Sodium	QCP-CICV-1	2500	2.5	500	12.5
Thallium	QCP-CICV-3	500	2.5	500	2.5
Titanium	Elemental Stock	1000	1.0	500	2.0
Vanadium	QCP-CICV-1	250	2.5	500	1.25
Zinc	QCP-CICV-1	250	2.5	500	1.25
Hydrochloric Acid	-	-	25	500	5%
Nitric Acid	-	-	5	500	1%

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TABLE 6 continued

ICVB Solution

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	Elemental Stock	1000	0.5	500	1
Boron	Elemental Stock	1000	2.5	500	5
Calcium	Elemental Stock	1000	2.5	500	5
Iron	Elemental Stock	1000	12.5	500	25
Lithium	Elemental Stock	1000	2.5	500	5
Magnesium	Elemental Stock	1000	2.5	500	5
Manganese	Elemental Stock	1000	6.25	500	12.5
Phosphorus	Elemental Stock	1000	2.5	500	5
Silicon	Elemental Stock	1000	2.5	500	5
Strontium	Elemental Stock	1000	1	500	2
Tin	Elemental Stock	1000	2.5	500	5
Hydrochloric Acid	-		25	500	5%
Nitric Acid	-		5	500	1%

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TABLE 7
ICP Interference Check Solutions

ICSA Solution

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	CLPP-ICS-A	5000	50	500	500
Calcium	CLPP-ICS-A	5000	50	500	500
Iron	CLPP-ICS-A	2000	50	500	200
Magnesium	CLPP-ICS-A	5000	50	500	500
Hydrochloric Acid	-	-	25	500	5%
Nitric Acid	-	-	5	500	1%

ICSAB Solution

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	CLPP-ICS-A	5000	50	500	500
Antimony	Elemental Stock	1000	0.5	500	1
Barium	CLPP-ICS-B	50	5	500	0.5
Beryllium	CLPP-ICS-B	50	5	500	0.5
Cadmium	CLPP-ICS-B	100	5	500	1
Calcium	CLPP-ICS-A	5000	50	500	500
Chromium	CLPP-ICS-B	50	5	500	0.5
Cobalt	CLPP-ICS-B	50	5	500	0.5
Copper	CLPP-ICS-B	50	5	500	0.5
Iron	CLPP-ICS-A	2000	50	500	200
Lead	CLPP-ICS-B	100	5	500	1
Magnesium	CLPP-ICS-A	5000	50	500	500
Manganese	CLPP-ICS-B	50	5	500	0.5
Nickel	CLPP-ICS-B	100	5	500	1
Silver	CLPP-ICS-B	100	5	500	1
Vanadium	CLPP-ICS-B	50	5	500	0.5
Zinc	CLPP-ICS-B	100	5	500	1
HCl	-	-	25	500	0.05
HNO3	-	-	5	500	0.01

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TABLE 8
TJA61 Analytical Wavelengths

Analyte	Wavelength	
Aluminum	308.2	
Antimony	206.8	
Arsenic	193.6	
Barium	493.4	
Beryllium	313.0	
Boron	249.6	
Cadmium	228.8	
Calcium	317.9	
Chromium	267.7	
Cobalt	228.6	
Copper	324.7	
Iron	259.9	
Lead	220.3	
Lithium	670.7	
Magnesium	279.5	
Magnesium	383.2	High Line
Manganese	257.6	
Molybdenum	202.0	
Nickel	231.6	
Phosphorus	214.9	
Potassium	766.5	
Selenium	196.0	
Silicon	288.1	
Silver	328.0	
Sodium	588.9	
Strontium	421.5	
Thallium	190.8	
Tin	189.9	
Titanium	334.9	
Vanadium	292.4	
Zinc	213.8	

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TABLE 9
IRIS Analytical Wavelengths

Analyte	Wavelength	
Aluminum	237.3	
Antimony	206.8	
Arsenic	189.0	
Barium	233.5	
Beryllium	313.0	
Boron	249.7	
Cadmium	226.5	
Calcium	317.9	
Calcium	211.2	High Line
Chromium	267.7	
Cobalt	228.6	
Copper	324.7	
Iron	259.9	
Iron	271.4	High Line
Lead	220.3	
Lithium	670.7	
Magnesium	279.5	
Magnesium	202.5	High Line
Manganese	257.6	
Manganese	293.9	High Line
Molybdenum	202.0	
Nickel	231.6	
Phosphorus	214.9	
Potassium	766.4	
Selenium	196.0	
Silicon	251.6	
Silver	328.0	
Sodium	589.5	
Strontium	407.7	
Thallium	190.8	
Tin	189.9	
Titanium	323.4	
Vanadium	310.2	
Zinc	206.2	

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TABLE 10
ICAP 6500 Analytical Wavelengths

Analyte	Wavelength	
Aluminum	167.0	Low Line
Aluminum	394.4	
Antimony	206.8	
Antimony	217.5	Alternate
Arsenic	189.0	
Barium	455.4	
Beryllium	234.8	
Boron	249.6	
Cadmium	226.5	
Calcium	315.8	
Calcium	393.3	Low Line
Chromium	267.7	
Cobalt	230.7	
Copper	327.3	
Iron	259.9	
Lead	220.3	
Lithium	670.7	
Magnesium	279.0	High Line
Magnesium	279.5	Low Line
Magnesium	285.2	
Manganese	257.6	
Manganese	260.5	High Line
Molybdenum	202.0	
Nickel	221.6	
Phosphorus	214.9	
Potassium	766.4	
Selenium	196.0	
Silicon	251.6	
Silver	328.0	
Sodium	588.9	Alternate
Sodium	589.5	
Strontium	407.7	
Thallium	190.8	
Tin	189.9	
Titanium	336.1	
Vanadium	292.4	
Zinc	206.2	

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STANDARD OPERATING PROCEDURE

For

GEL PERMEATION CHROMATOGRAPHY

SOP No.: SOC-3640A

Revision: 6
January 14, 2009

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Approved by: _____ Supervisor — 1/15/09 Date

_____ QA Manager — 1/15/09 Date

_____ Laboratory Manager — 1/15/09 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

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Annual review of this SOP has been performed and the SOP still reflects current practice.
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Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

GEL PERMEATION CHROMATOGRAPHY

1. SCOPE AND APPLICATION

This procedure describes the Gel Permeation Chromatography (GPC) cleanup of semivolatile organics sample extracts by EPA Method 3640A. The procedure is most often applied to soil, sediment, and tissue matrices, but may be used on any matrix which may require cleanup to remove interfering co-extracted material.

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2. METHOD SUMMARY

2.1. Gel Permeation Chromatography is the separation of compounds by size exclusion. This is accomplished by using organic solvents and hydrophobic gels in the separation of synthetic macromolecules. The packing gel is porous and made of Bio-Beads S-X3 (spherical porous styrene divinyl benzene copolymer cross-linked).

2.2. GPC is routinely done for all soil/sediment, and tissue samples. This procedure is effective in removing a broad range of interferences that could hinder the identification of target analytes.

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2.3. This type of cleanup is effective for removing high boiling compounds that condense in the injection port area or in front of the column of a GC.

3. DEFINITIONS

3.1. ABC system - The GPC configuration which uses the ABC GPC unit, detector, integrator, and related components as described in the Apparatus section.

3.2. J2 AccuPrep MPS system - The GPC configuration which uses the J2 Accuprep system and related components as described in the Apparatus section.

4. INTERFERENCES

4.1. Steps should be taken to minimize the introduction of interferences from reagents, glassware, and sample handling hardware. Refer to the determinative procedure for a description of potential interferences.

4.2. The level of extracted material detected may vary considerably for field samples. The analyst should be aware of the possibility of contamination of the system from a high concentration sample. If contamination is suspected, a blank should be analyzed to establish that the system is clean.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. This method uses Dichlororomethane (DCM), a known human carcinogen. Viton brand gloves should be used while rinsing, pouring or transferring the solvent.

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6. SAMPLE PRESERVATION, STORAGE, AND HOLDING TIME

Samples must be extracted within the holding time specified for the analytical method being used. Sample extract cleanup should be performed in time to allow the extract holding time to be met after cleanup.

7. APPARATUS AND MATERIALS

7.1. ABC System:

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7.1.1. Chromatographic Column - 700mm x 25mm i.d. ABC S-X3 glass column. The flow is upward.

7.1.2. Bio-Beads (S-X3), 200-400 mesh, 70g - Beads are porous styrene divinyl benzene copolymer. J2 Scientific Cat # C0070G or equivalent.

7.1.3. ABC GPC system with UV detector and integrator.

7.1.3.1. Ultraviolet Detector - fixed wavelength of 254 nm with a semi-prep flow-through cell, ABC UVD-1.

7.1.3.2. Integrator or strip-chart recorder, ABC SCR-1 or equivalent.

7.1.4. External DCM solvent reservoir.

7.1.5. External waste collection reservoir.

7.2. J2 AccuPrep MPS System:

7.2.1. Chromatographic Column - 700mm x 25mm i.d. ABC S-X3 glass column. The flow is upward.

7.2.2. Bio-Beads (S-X3), 200-400 mesh, 70g - Beads are porous styrene divinyl benzene copolymer. J2 Scientific Cat # C0070G or equivalent.

7.2.3. J2 Scientific DT0001 Fixed Wavelength Detector.

7.2.4. Computer installed with JT Scientific AccuPrep software.

7.2.5. AutoPrep sample load tray.

7.2.6. AutoPrep sample collection tray.

7.2.7. External DCM solvent reservoir.

7.2.8. External solvent waste reservoir.

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8. STANDARDS, REAGENTS, AND CONSUMABLES

8.1. GPC calibration solution (Restek Cat # 32032 or equivalent) must contain the following analytes:

Compound	Conc. (mg/ml)
Corn oil	250
Bis(2-ethylhexyl)phthalate	10.0
Methoxychlor	2.0
Perylene	0.2
Sulfur (optional)	0.8

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8.2. Any dilution of the GPC standard made prior to calibration runs must be made in DCM and stored in an amber glass bottle with a Teflon-lined screw-cap at 4°C protected from light. Replace any calibration standard dilution after 6 months.

8.3. Dichloromethane (DCM). Pesticide quality or equivalent. A high res/GC^2 grade of dichloromethane is required for GCMS applications.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument. Documentation that demonstrates a return to control must be recorded in the maintenance logbook.

9.2. Each GPC must be calibrated at a minimum rate of once every seven days to ensure that the proper collection windows are being utilized and the calibration peak resolutions are acceptable.

9.3. Weekly LCS checks are required for 8270 and 8270-LL GPC runs to make sure the column remains in proper condition for optimal analyte recoveries. An LCS check is also required after clean-up of highly reactive samples (this currently includes all Philip Services 8270-LL waters). The LCS recoveries should exceed 70%, however some very reactive analytes (ie. 2,4-Dimethylphenol, Hexachlorocyclopentadiene, 3,3'-Dichlorobenzidine) may show lower recoveries.

9.4. Repacking of the GPC column is necessary when the column fails to produce proper resolution, becomes contaminated, or colored, or forms channels. The GPC column should be repacked every 6 months or sooner if results of the weekly LCS show poor recoveries. The following procedures describe repacking procedure for either 70 grams of Envirobeads S-X3 in 100% dichloromethane or 60 grams in 1:1 dichloromethane:cyclohexane.

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9.4.1. Weigh out 60 grams (for dichloromethane/cyclohexane column) or 70 grams (for 100% Dichloromethane column) of Envirobeads S-X3 into a 400 ml beaker or other suitable container.

9.4.2. Add about 300 ml of desired solvent and gently stir.

9.4.3. Cover with clean aluminum foil and allow the beads to swell for minimum of two hours. Maintain enough solvent to sufficiently cover the beads at all times.

9.4.4. Disassemble and clean the column. Remove o-ring and column mesh from the assembly, leaving the Teflon screen in place. Inspect the Teflon screen and replace if it is severely clogged or damaged. Discard old mesh and replace with new mesh.

9.4.5. Position and tighten the top plunger assembly in the tube by inserting the plunger and turning it clockwise until snug. Install the plunger so that the bed support holder is just past the logo on the column barrel.

9.4.6. Clamp the column upside-down on a ring stand with the open end up. Place the tubing from the top plunger assembly into a waste beaker below the column.

9.4.7. Slurry the bead/solvent mixture and pour the mixture into the open end of the column. Transfer as much as possible in one continuous pour. Then wait for excess solvent to drain out before pouring in the rest. Use additional solvent to transfer remaining beads and to rinse the beaker and sides of the column. Use only Teflon® wash bottles or glass syringes for rinsing.

9.4.8. For the dichloromethane/cyclohexane column, watches the layer of solvent above the gel go down as the excess solvent drains. When this layer meets the gel layer, rinse the beads from the inner walls of the column and wait for the additional solvent to drain again (to the level of the gel layer). For the dichloromethane column, the layers are not

always clearly distinguishable, since the gel is lighter than the solvent. Wait until the gel settles and all excess solvent is drained. The top of the gel will begin to look dry. Raise the waste container and line from the plunger to bench level to prevent further draining of the solvent. Do not allow the beads to be drained dry. If this should happen, rewet the beads by adding more solvent.

9.4.9. Wipe any remaining beads and solvent from the inner walls of the column at the top with a laboratory tissue. Loosen the seal slightly on the other plunger assembly and insert it into the column. Make the seal just tight enough so that any beads on the glass surface will be pushed forward, but loose enough so that the plunger can be pushed forward.

9.4.10. Push the plunger until it meets the gel, then compress the column bed (approximately 4 centimeters).

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9.4.11. When the plunger is properly inserted, tighten the plunger by turning it clockwise until snug.

9.4.12. Connect the column input and output lines together to prevent drying while being stored.

9.4.13. To prepare for conditioning, install the column on the column support arms. Connect column input line (long plunger) to the GPC's output line.

9.4.14. Place a column output line in a waste container.

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9.4.15. Turn on the solvent pump and allow it to pump for at least 30 minutes into the glass beaker, using the same solvent in which the column was packed. The pump flow rate should be set at 5.0 ml per minute.

9.4.16. Collect column effluent in a glass beaker for a few minutes and inspect the beaker to insure that gel beads are not escaping from the column. If beads are present, pump for several minutes more and then check again for leakage of beads. If gel beads are still present in the column effluent after 30 minutes, the column must be disassembled, cleaned, inspected, and repacked.

9.4.17. Closely monitor the column pressure. High pressures are occasionally encountered while the gel is being rewetted, especially for the 100% dichloromethane column. If the pressure continues to rise above 15 psi, reverse direction of flow through the column by exchanging the connections to the top and bottom of the column until pressure begins to fall, then replace the connections in their original configuration. If the system pressure exceeds 20 psi, an overpressure switch will turn off the pump. The pressure should begin to decline to between 7 and 10 psi as the gel is uniformly wetted and swollen. If this is not the case, check for any restrictions or blockages.

9.4.18. After 30 minutes, turn the pump off. If no Bio-Beads are present in the column effluent, connect the column output line to the GPC module (or the bypass valve), then restart the pump.

9.4.19. After an hour or two the column pressure should stabilize between 7 and 10 psi. For either type of column, it may be necessary to adjust the column bed length to achieve a column pressure of 7-10 psi. Loosen the inlet plunger enough to allow some movement, but not enough to allow solvent or beads to escape around the seal. Push plunger in to increase pressure or slowly pull outward to reduce pressure.

9.4.20. The UV detector should be cleaned as needed.

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10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. ABC System

11.1.1. Cleaning GPC ports between samples

11.1.1.1. After each run, each port must be rinsed prior to loading the next run of samples.

11.1.1.2. Rinse the sample tube 3 times with DCM. Add 10mL of DCM to each sample tube.

11.1.1.3. Load the appropriate clean cycle method and start the program. Collect and dispose of the DCM.

11.1.2. Calibration of the ABC GPC column

11.1.2.1. Calibrate the GPC once every seven days at a minimum. Retention times must be within 5% of the previous calibration retention times. A copy of the

UV trace should be kept in a logbook and may be submitted with the data for the associated samples. If retention times are not correct and resolution between samples does not meet requirements, maintenance may be necessary

11.1.2.2. Route the column exit flow through the UV detector. Turn detector on at least 30 minutes prior to calibration run in order to warm up the detector. Start purging the system for at least one half hour by turning on the pumps at a flow of 5.0 ± 0.5 mL/min. This will force DCM to flow through the system. Open the nitrogen tank valve. Set the regulator at 30 psi. Set rinse pressure on the GPC at 12-15 psi.

11.1.2.3. If the GPC is not equipped with a liquid sensor, the sample load time must be measured. This is done by placing 10 mL of DCM in a tube at position #1 and timing how long it takes to pull up all of the solvent. The amount of time it would take to pull up the smallest quantity of sample to be run (at least 8 mL) is then saved to the GPC program. Even if the GPC is equipped with a liquid sensor, the sample time of the weekly calibration run and that of at least one sample per cleanup run should be measured. The time required to load 10 mL of DCM should be less than 12 seconds. In addition, no rinse should accumulate in the sample tube (it should be pulled up as soon as it reaches the bottom of the tube). If these conditions are not met, maintenance is necessary.

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11.1.2.4. Load 10mL of the standard into the system. A 5 ml loop requires a minimum of 8 mL of the calibration standard. Put a 10 ml aliquot of an appropriate dilution of the standard into a sample pickup tube and place it in a sample container support arm.

11.1.2.5. Using the integrator, begin plotting UV traces adjusting as needed the UV and/or the integrator itself to obtain discrete peaks for each component. For the ABC system, the control box should have the chart speed set at 20 cm/hr and the input at 2 mV.

11.1.2.6. From the UV trace, establish collect and dump times. Use a wash time of 10 minutes after sulfur has eluted. The organochlorine pesticide/PCB collection window starts at the valley between the phthalate and methoxychlor peaks. The 8270/OP collection window starts at the valley between the large corn oil and phthalate peaks. Both collection windows end at the valley between the perylene and sulfur peaks. All the information should be documented on the 'GPC Calibration Record' form (See Attachments).

11.1.2.7. Verify the flow rate by collecting eluting solvent for 1 minute in a graduated cylinder. This should be between 4.5 - 5.5 mL. If flow rate is not within range, corrective action must be taken.

11.1.2.8. Record the room temperature on the calibration record, as this may cause a shift in the retention times.

11.1.2.9. CLP work requires peak resolution > 85% with Perylene and sulfur peaks at a >90% resolution. (Refer to the specific CLP SOP and Attachments CLP Forms for further details).

11.1.2.10. The resolution is calculated by measuring the height of the shortest peak of a pair, minus the valley between them, divided by the height of the shortest peak. The result is then multiplied by 100.

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11.1.3. Sample Extract Cleanup with the ABC GPC

11.1.3.1. Refer to Table 1 for test-specific intermediate volumes and final volumes.

11.1.3.2. Run the clean cycle program on all positions used before starting the next run.

11.1.3.3. Dilute sample extracts as needed in DCM by taking to the method's desired intermediate volume. Record any dilutions made.

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11.1.3.4. Filter 8-10 mL of the sample extract through a 0.45 filter on a 10 mL syringe and collect into its pre-labeled GPC test tube in numerical order. Screw the test tube into place on the GPC. A minimum of 8 mL of extract can be loaded on the GPC. If below after filtering, a dilution will need to be made and a new aliquot loaded.

11.1.3.5. An Erlenmeyer flask or collection bottle acts as the extract collector and is labeled the same as the test tube. 8270 extracts should be collected in amber flasks or bottles to avoid light degradation of some analytes.

11.1.3.6. Scroll through the control box panel by pressing *index* and load the appropriate program for the analysis. Check to make sure the correct dump and collection times are entered into the program being used. Load the beginning and ending sample positions, and then press the run button. The pump should be turned on for a minimum 10 minutes to rehydrate the column and allow for the GPC to build up to normal running pressure.

11.1.3.7. Monitor sample volumes collected. If changes occur, check the flow rate, column pressure, or look for leaks in the system.

11.1.3.8. Perform final concentration, transfer, and labeling of extracts as described in the extraction SOP.

11.2. J2 Scientific AccuPrep MPS System

11.2.1. Calibration of the AccuPrep GPC column

11.2.1.1. Calibrate the GPC once every seven days at a minimum. Retention times must be within 5% of the previous calibration retention times. A copy of the UV graph should be kept in a logbook and may be submitted with the data for the associated samples. If retention times are not correct and resolution between samples does not meet requirements, rinse the column using butyl chloride.

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11.2.1.2. Turn the detector on and let warm up for a minimum 1 hour.

11.2.1.3. Load 10 mL of the calibration standard into a test tube in position 1.

11.2.1.4. Click on the AccuPrep Icon to open the AccuPrep GPC software.

11.2.1.5. Click on the pump flow icon in the tool bar, labeled "Change pump flow or calibration". Click on the "Enable" icon to start the DCM pump.

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11.2.1.6. Click the "Zero Det 1" icon to zero out the detector. Once detector keeps steady at "0000", it is ready for calibration.

11.2.1.7. Click on the "Sequence" icon in the tool bar. Click on the "Create new sequence icon" to bring up blank sequence page.

11.2.1.8. Enter current date into position 1.

11.2.1.9. Change the method column for position 1 to "Calibration".

11.2.1.10. Save the sequence to the current date (mmddyy cal 5).

11.2.1.11. Close the sequence window and click the "Execute" icon to start the run.

11.2.1.12. Once the sequence has finished. Exit the AccuPrep program and open the "AccuChrome" program on the desktop.

11.2.1.13. Click on "File" and import "V1.0 Data". Open the file created for the current calibration date. It may take a few minutes to open.

11.2.1.14. Click on the first square icon in the tool bar. Click on "Sequence", "Sample", "Method", and then "Data Plot".

11.2.1.15. Once the graph page opens, click on the "Find Peaks" icon. Line up the calibration lines with the corresponding peaks and adjust until the highest percentage is achieved at the top of the graph for each peak.

11.2.1.16. Print the graph. From the graph, establish collect and dump times. Use a wash time of 10 minutes after sulfur has eluted. Calculate the collection windows with the computer generated values. The organochlorine pesticide/PCB collection window starts at the valley between the phthalate and methoxychlor peaks. The 8270/OP collection window starts at the valley between the large corn oil and phthalate peaks. Both collection windows end at the valley between the perylene and sulfur peaks. Close graph window. Exit the AccuChrome program.

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11.2.1.17. Reopen the AccuPrep program. To make changes to the collection window, click on the "Method Time 1,2,3" icon in the tool bar. Enter the new calculated "dump" and "collection" times for each collection method in the fraction column. Save and click on the "Column Description: DCM" box.

11.2.1.18. Log the current room temperature and flow rate. Verify the flow rate by collecting eluting solvent for 1 minute in a graduated cylinder. This should be between 4.5 - 5.5 ml. If flow rate is not within range, corrective action must be taken.

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11.2.2. Sample Extract Cleanup with the AccuPrep GPC

11.2.2.1. Refer to Table 1 for test-specific intermediate volumes and final volumes.

11.2.2.2. Dilute sample extracts as needed in DCM by taking to the method's desired intermediate volume. Record any dilutions made. The J2 Scientific AccuPrep GPC loads exactly 5 mL of sample extract, so intermediate volumes can range from 5-10 mL loaded depending on the method desired.

11.2.2.3. Filter 5-10 mL of the sample extract through a 0.45 filter and collect into its pre-labeled 10 mL screw-top test tube in numerical order. Screw an open-top test tube cap with a Teflon liner onto the test tube.

11.2.2.4. Place the filtered sample extracts into the AccuPrep sample tray in numerical order.

11.2.2.5. Load the extract collection tray with the appropriate number of pre-rinsed collection tubes. Label the collection tubes to correspond with the sample

extracts in the sample tray. Cover the collection tubes with a layer of aluminum foil to diminish solvent evaporation of the sample extracts.

11.2.2.6. Click on the AccuPrep Icon to open the AccuPrep GPC software.

11.2.2.7. Click on the pump flow icon in the tool bar, labeled "Change pump flow or calibration". Click on the "Enable" icon to start the DCM pump.

11.2.2.8. Click on the "Sequence" icon in the tool bar. Click on the "Create new sequence icon" to bring up blank sequence page.

11.2.2.9. Build the desired sequence by typing in samples corresponding with sample positions in sample test tube tray. Press enter after each sample entry to advance to the next sample position. Enter correct collection window.

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11.2.2.10. Save the sequence by clicking on the "Save" icon. Label the sequence as the current run date (mmddyy).

11.2.2.11. Print sequence by clicking on the "Print" icon. Attach one printed sequence to the sample benchsheet. Place another printed sequence into the GPC runlog notebook.

11.2.2.12. Close the sequence window and click the "Execute" icon to start the run.

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11.2.2.13. Monitor sample volumes collected. If changes occur, check the flow rate, column pressure, or look for leaks in the system.

11.2.2.14. Perform final concentration, transfer, and labeling of extracts as described in the extraction SOP.

12. CORRECTIVE ACTION

12.1. Refer to the SOP for *Nonconformity and Corrective Action* for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

12.2. Handling out-of-control or unacceptable data

12.2.1. On-the-spot corrective actions that are routinely made by analysts should be documented in the GPC maintenance logbook, or the GPC runlogs.

12.2.2. Documentation of a nonconformity must be done using a Nonconformity and Corrective Action Report (NCAR) when: a) corrective action is not taken or not possible b) corrective action fails to correct an out-of-control problem on a laboratory QC or calibration analysis c) reanalysis corrects the nonconformity but is not a procedurally compliant analysis.

13. METHOD PERFORMANCE

Available method performance data is given in the reference method. In addition, this procedure was validated through single laboratory studies of accuracy and precision as specified in the determinative procedures.

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14. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

15. WASTE MANAGEMENT

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15.1. The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

15.2. This method uses Dichloromethane and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and recycled off site.

16. TRAINING

16.1. Training outline

16.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

16.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

16.1.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

16.2. Training is documented following the SOP *for Documentation of Technical Personnel Training*.

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

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17. REFERENCES

17.1. EPA Method 3640A, "Gel Permeation Cleanup", SW-846 Update II, USEPA, September, 1994.

17.2. ABC/GPC Operation Manual, Revision 1, 1994.

17.3. J2 Scientific AccuPrep MPS for GPC Cleanup-Direct Infect Configuration Hardware Installation Guide, Version 1.0, 2005.

17.4. J2 Scientific AccuPrep MPS Software Users Guide, 2001.

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Table 1

Test-specific Volumes
(Nominal values)

Analysis	Intermediate Volume (mL)	Final Volume (mL)	Dilution	True Final Volume (mL)
8081/8082-AR Soils	10	2	4 mL to 10 mL	10
8081/8082-AR LL Soils	10	2	-	4
PCB Congener Soils	10	2	-	4
Pest/PCB Tissues	10	2	-	4
PCB Congener Tissues	10	2	-	4
8270 Soils	10	0.5	-	1
8270-LL Soils	10	1	-	2
SIM-PAH-PCP Soils, SIM Tissues	10	5	-	10
Note: Intermediate and final volumes are dependent upon the required detection limits. Therefore, they may vary depending on the client or project. It is the true final volume that is critical in meeting these requirements.				

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Attachments

GPC Forms

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Table 1

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PCB Congener Tissues	10	2	-	4
8270 Soils	10	0.5	-	1
8270-LL Soils	10	1	-	2
SIM-PAH-PCP Soils, SIM Tissues	10	5	-	10
Note: Intermediate and final volumes are dependent upon the required detection limits. Therefore, they may vary depending on the client or project. It is the true final volume that is critical in meeting these requirements.				

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GPC RUNLOG _____

RUN DATE:_____ STANDARD DATE:_____

OPERATOR:_____ PROGRAM*:_____

LAB I.D.	POS.	TEST	DIL.	COMMENTS
	1			
	2			
	3			
	4			
	5			
	6			
	7			
	8			
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*When multiple programs are used, note change in comment section and program settings below.

Program#_____ Dump_____ Collect_____

Program#_____ Dump_____ Collect_____

ABC GPC BENCHSHEET

Analyst: _____ Matrix: Tissue / Reg. Soil / Low Level Soil / Water / Oil

Date: _____ Work Order: _____

A. GPC Calibrated on this date: _____

B. (1) Intermediate Volume before GPC: _____(ml)

(2) Aliquot taken from intermediate volume: _____(ml)

(3) Aliquot diluted up to..... _____(ml)

(4) Volume injected onto column: _____(ml)

(5) GPC'd Extract brought to the Final Volume of _____(ml)

(6) Calculate the True Final Volume _____(ml) $\frac{(1) \times (3)}{(2) \times (4)} \times (5) = (6)$

C. Does a split for PCB's or PAH's need to be done? YES NO

Checklist for the GPC Run:

_____ 1. Is the column reservoir full?

_____ 2. Is the rinse reservoir full?

_____ 3. Is the waste bottle empty?

_____ 4. Did you check the flow rate?

_____ 5. Is there a collection vessel for each extract tube?

_____ 6. Do the extract tube labels correspond to the collection vessel labels?

_____ 7. Are the extracts diluted properly, if needed?

_____ 8. Are all extracts filtered? (They MUST be)

_____ 9. Did you record where you put the remaining extract?

_____ 10. Is the correct method/collection window programmed?

_____ 11. Is the By-pass valve set to **In-line**?

_____ 12. Is the run log filled out?

_____ 13. Is sample load time set correctly for extract with lowest volume? (VERY IMPORTANT)

_____ 14. Is the nitrogen tank valve open?

_____ 15. Is there enough nitrogen (at least 500lb/23 extracts)?

_____ 16. Did you double check your GPC setup?

Operator date and initial _____

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GPC Calibration Record Instrument:_____ Column:_____

Date: _____ Analyst:_____ Reviewed:_____

GPC Standard ID:_____ Standard Dilution:_____ Room Temp:_____ °C

System Pressure:_____ psi

Flow Rate:_____ mL/min

RETENTION TIMES (minutes)

Corn Oil: _____

BEHP: _____

Methoxychlor: _____

Perylene: _____

Sulfur: _____

PROGRAM TIMES (minutes)

	OC-Pest/PCBs	BNA/**OP**-Pest
Program Number	2	1
Dump		
Collect		
Wash		

Notes:

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STANDARD OPERATING PROCEDURE

ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY

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SOC-8081
Revision 14
June 25, 2009

Approved By: _____ Supervisor — 6/25/09 Date

_____ QA Manager — 6/29/09 Date

_____ Laboratory Manager — 6/29/09 Date

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COLUMBIA ANALYTICAL SERVICES, INC.
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Kelso, Washington 98626

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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY

1. SCOPE AND APPLICATION

This procedure is used to determine the concentrations of Organochlorine Pesticides in water and soil/sediment using EPA Method 8081A. Table 1 indicates compounds that are routinely determined by this procedure and lists the method reporting limits (MRLs) in water and soil/sediment. Additional analytes may also be determined as required by specific projects. The reported MRL may be adjusted if required for specific project requirements; however, the capability of achieving other reported MRLs must be demonstrated. Method Detection Limits that have been achieved are given in Attachment A. MDLs may change as repeat studies are conducted. A low-level sediment option may be used, with MRLs given in Table 2.

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2. SUMMARY OF METHOD

2.1. This procedure provides gas chromatographic conditions for the detection of low concentration (typically parts-per-billion level organochlorine pesticides) pesticides. Target analytes are extracted from the sample and isolated via extract cleanup if needed. Liquid samples are extracted using continuous liquid-liquid extraction (Method 3520, CAS SOP EXT-3520), or solid phase extraction (Method 3535, CAS SOP EXT-3535). TCLP leachates are extracted using separatory funnel (Method 3510, CAS SOP EXT-3510). In general, soil/sediment samples are extracted using Soxhlet (Method 3540, CAS SOP EXT-3540) or automated Soxhlet extraction (Method 3541, CAS SOP EXT-3541).

2.2. A portion of the extract is analyzed using a gas chromatograph (GC) equipped with dual column fused silica capillary columns and dual electron capture detectors (ECD). Identification is based on comparison of sample retention times to the retention times of known target compounds. Quantitative analysis is performed by using certified standards to produce a calibration curve response factor. Analyte concentration can then be calculated using the curves response factor. Sample concentration is calculated using the extract concentration and the extracted sample weights, volumes and dilution factors.

2.3. The sensitivity of the procedure depends on the level of interferences rather than instrument limitations. If interferences prevent the detection of analytes, GPC cleanup, Florisil cleanup, sulfur cleanup, carbon cleanup, and other appropriate techniques may be used to reduce interferences.

3. DEFINITIONS

3.1. Analysis Sequence - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample extracts interspersed with calibration standards. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded. See the SOP *ADM-BATCH*.

3.2. Independent Calibration Verification (ICV) - Verification of the ratio of instrument response to analyte amount, a calibration check, is done by analyzing for analyte standards in an appropriate solvent. ICV solutions are made from a stock solution which is different from the stock used to prepare calibration standards.

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3.3. Internal Standards - Internal standards are organic compounds which are similar to the analytes of interest but which are not found in the samples. The chosen internal standards are used to help calibrate the instrument's response and to compensate for slight instrument variations from injection to injection.

3.4. Matrix Spike/Duplicate Matrix Spike Analysis - In the matrix spike analysis, predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, spiked, and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

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3.5. Standard Curve - A standard curve is a curve which plots concentrations of a known analyte standard versus the instrument response to the analyte.

3.6. Surrogate - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples, and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

3.7. Method Blank - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.8. Continuing Calibration Verification Standard (CCV) - A standard injected into the instrument at specified intervals and is used to verify the initial calibration.

3.9. Instrument Blank (CCB) - The instrument blank (also called continuing calibration blank) is a volume of clean solvent analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples.

4. INTERFERENCES

4.1. Solvents, reagents, glassware, gases, and sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by running method blanks.

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4.2. Interferences from phthalate esters introduced during sample handling can pose a problem with pesticide determinations. Analysts should take precautions not to introduce phthalates during the analysis and sample preparation process. Many interferences can be removed using GPC (SOP *SOC-3640A*) and/or Florisil cleanup (SOP *EXT-FLOR*). Florisil cleanup is typically used to reduce matrix interferences caused by polar compounds. The presence of elemental sulfur will result in peaks interfering with early eluting pesticides. Cleanup via method 3660 (SOP *SOC-3660*) may be used for the removal of sulfur if GPC cleanup is inadequate. Other co-extractables such as lipids, waxes, etc., are removed via GPC cleanup.

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5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS *Environmental, Health and Safety Manual* and the appropriate MSDS prior to beginning this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Containers used to collect samples should be purchased pre-cleaned containers. Alternatively, containers used to collect samples for the determination of semivolatile organic compounds may be soap and water washed followed by methanol (or isopropanol) rinsing. The sample containers should be of glass or Teflon and have screw-top covers with Teflon liners. In situations where Teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may <u>not</u> be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons

within the plastic. Sampling should be performed according to SW-846 guidelines or project-specific procedures.

6.2. Water and soil samples must be iced or refrigerated at 4 ± 2°C from time of collection until extraction. Water samples must be extracted within 7 days of collection and soil/sediment samples must be extracted within 14 days of collection.

6.3. Sample extracts are stored at 4° in the dark and must be analyzed within 40 days.

7. STANDARDS, REAGENTS AND EQUIPMENT

7.1. Standards

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7.1.1. The following commercially prepared stock standards and ICV stock standards are purchased from various vendors and must be certified by the manufacturer. The ICV stock standards are obtained from a different source from the initial calibration. The expiration date for unopened ampules is the manufacturer's assigned expiration date. If the manufacturer does not assign a date, use an expiration date of 1 year from receipt. Stock standards are stored at -10°C.

Standard	Concentration	Use
8081	1000 ug/mL	ICAL
8081 Surrogate[a]	200 ug/mL	ICAL
Isodrin	100 ug/mL	ICAL
Hexachlorobenzene	100 ug/mL	ICAL
8081 Misc. compounds*	250 ug/mL	ICAL
Chlordane	5000 ug/mL	ICAL
Toxaphene	1000 ug/mL	ICAL
8081	1000 ug/mL	ICV
Isodrin	100 ug/mL	ICV
Hexachlorobenzene	100 ug/mL	ICV
8081 Misc. compounds	100 ug/mL	ICV
Chlordane	1000 ug/mL	ICV
Toxaphene	1000 ug/mL	ICV
8081 Internal Standard[b]	5000ug/mL	Internal standard

[a] Surrogates are decachlorobiphenyl (DCB) and 2,4,5,6-Tetrachlorometaxylene (TCMX).
[b] Internal standard is 1-Bromo-2-nitrobenzene.

7.1.2. Intermediate standard solutions and ICV solutions are made in hexane by diluting stock standards to intermediate concentrations listed below. The ICV intermediates are prepared from the ICV stock standards obtained from a different source from the initial calibration stocks.

ICAL Intermediate	Concentration	Preparation
Combined 8081, isodrin, hexachlorobenzene, and surrogate.	10 ug/mL	1:100 dilution of 8081 stock, 1:10 dilution of isodrin & hexachlorobenzene stocks, 1:20 dilution of surr. stock.
8081 Misc. compounds*	10 ug/mL	1:25 dilution of stock
Chlordane	250 ug/mL	1:20 dilution of stock
Toxaphene	100 ug/mL	1:10 dilution of stock

Intermediate ICV Standard	Concentration	Preparation
Combined 8081, isodrin, and hexachlorobenzene	10 ug/mL	1:100 dilution of 8081 stock, 1:10 dilution of isodrin & hexachlorobenzene stocks.
8081 Misc. compounds*	10 ug/mL	1:10 dilution of stock
8081 Internal Stanard	5ug/mL	1:1000 dilution of stock

* Prepare only as needed for projects requiring non-routine additional compounds.

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7.1.3. Working Standard Solutions

7.1.3.1. Calibration standards are prepared containing surrogates and analytes in hexane. Calibration standards are stored at 4° for up to six months. A series of standards are prepared from a common intermediate representing the MRL (or lower) to a value near the high end of the linear range. Calibration standards are prepared in hexane. See Table 4 for preparation and concentrations, including standards designated as CCVs.

7.1.3.2. The independent calibration verification (ICV) standards are prepared from stock solutions from a different source from the initial calibration as listed below. Expiration periods are the same as for equivalent stock and calibration standards.

ICV Standard	Concentration	Preparation
Combined 8081, isodrin, and hexachlorobenzene	40 ug/L	1:250 dilution of combined 8081 ICV intermediate.
8081 Misc. compounds	40 ug/L	1:250 dilution of 8081 misc. compounds intermediate.
Chlordane	800 ug/L	1:1250 dilution of Chlordane ICV stock.
Toxaphene	1000 ug/L	1:1000 dilution of Toxaphene ICV stock.

7.1.3.3. A surrogate spiking solution is prepared at 2 ug/mL by making a 1:100 dilution of the surrogate stock standard in acetone. The surrogate solution is stored at 4° or up to six months.

7.1.3.4. An internal standard solution is prepared at 5.0ug/ml by making a 1:1000 dilution of the surrogate stock standard in hexane. 10uL of internal standard is added to each standard, blank and sample prior to analysis for a final concentration of 50ng/mL.

7.1.3.5. A matrix spike solution at 4 ug/mL is prepared by diluting the stock solution in methanol. This solution is stored at 4° for 2 weeks. The matrix spike solution is added to all matrix spikes and lab control samples as outlined in section 12.

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Spiking Solution	Concentration	Preparation
8081, isodrin, and hexachlorobenzene.	4 ug/mL	1:250 dilution of 8081 stock, 1:25 dilution of isodrin & hexachlorobenzene stocks.
8081 Misc. compounds*	4 ug/mL	1:25 dilution of stock
Chlordane**	10 ug/mL	1:500 dilution of stock
Toxaphene**	10 ug/mL	1:100 dilution of stock

* Prepare only as needed for projects requiring non-routine additional compounds.
** Prepare only as needed for project requirements.

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7.1.4. Solvents: Hexane, acetone, methylene chloride, isooctane, and methanol. Pesticide grade or equivalent

8. APPARATUS AND EQUIPMENT

8.1. GC Instrumentation - Dual Column

8.1.1. The dual column approach involves a single injection split between two columns mounted in a single gas chromatograph (Hewlett Packard 5890, 6890, 7890 or equivalent) equipped with cool-on-column, split/splitless, or temperature programmable injection system; with dual ECDs. See Table 3 for typical chromatographic conditions. Helium is used as the carrier gas. Argon/methane mixture is used as the detector make-up gas. Current instrumental systems are identified as follows:

Instrument I.D.	Analytical System	Routine Matrix
GC23	Agilent 6890	Water/Soil/Tissue
GC28	Agilent 6890	Water/Soil/Tissue

8.1.2. Columns, J&W columns typically are used;

Column 1 : DB-XLB 30m x 0.32mm ID, 0.50 μm df or equivalent*
Column 2 : DB-35MS 30m x 0.32mm ID, 0.25 μm df or equivalent*
Guard Column: Varian CP-Sil8 5m x 0.53mm ID, 0.50 μm df, or equivalent

Note: Column diameter and film thickness may vary depending on instrument. Refer to the instrument maintenance logbook for the columns used for a specific instrument configuration.

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8.1.3. Autosampler, capable of reproducible injections, Hewlett Packard/Agilent 7673 or equivalent.

8.1.4. Data System - A computer data system must be interfaced to the GC/ECD. The system must allow the continuous acquisition and storage on machine-readable media of all chromatographic data obtained throughout the duration of the chromatographic program. The computer must have software that includes automated calibration, identification, and quantitation routines. The software must also be capable of integrating the chromatographic peaks abundances. The most recent version of the manufacturer's software is preferred. HP Enviroquant.

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9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument.

9.2. Carrier gas - Inline purifiers or scrubbers should be in place for all sources of carrier gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3. Gas Chromatograph

9.3.1. Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance includes changing the injection port liner, seal, washer, o-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chromatographic performance. In some cases liners and seals may be cleaned and re-used.

9.3.2. Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column cutting tool. Clipping the column should be done from the guard

column as the first option. If needed, replace the guard column. Only clip from the analytical column as a last option.

9.3.3. Over time, the column will exhibit poorer overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

10. RESPONSIBILITIES

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10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in 8081A, is also the responsibility of the department supervisor/manager.

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11. PROCEDURE

11.1. Sample Preparation

11.1.1. Water samples (1L) are extracted at a pH of 5-9 with methylene chloride, using Method 3520 (SOP *EXT-3520)*, or solid phase extraction (SOP *EXT-3535*). TCLP leachates are extracted using separatory funnel (SOP *EXT-3510*). Refer to the applicable extraction SOP. For extraction by 3535, acidification of the sample prior to extraction may be allowable if project objectives and performance requirements of methods 3535 and 8081 are met.

Note: For projects originating from South Carolina and under the SC DHEC lab certification, use the 3535 extraction method only.

11.1.2. Soil/sediment samples are extracted using either EPA Method 3540 (SOP *EXT-3540*) or method 3541 (SOP *EXT-3541*). A low-level sediment option may be used where the sample weight or volume is adjusted to lower the level of detection in the sample. Refer to the applicable extraction SOP.

Note: For projects originating from South Carolina and under the SC DHEC lab certification, use the 3541 extraction method only.

11.1.3. Additional sample cleanup procedures may be used as appropriate for the samples. See Section 4.2 and refer to the applicable cleanup SOP.

11.2. Calibration

11.2.1. Refer to the SOP *Sample Batches* (*ADM-BATCH*) for guidance on analytical calibration and sample batches.

Note: The default calibration procedures are given in the following sections and, at the time of this revision, follow Method 8000B. Refer to the *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOC-CAL)*, where calibration procedures and policies are described. The calibration procedure(s) and options chosen must follow the SOP *SOC-CAL*.

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Note: Certain state, projects or programs require using the most recent versions of SW-846. This may include all or part of Method 8000C. Refer to the Columbia Analytical Services, Inc. SOP *Calibration of Instruments for Organics Chromatographic Analysis using EPA 8000C (SOC-CAL8000C)*. The analyst must ensure that the correct procedures are used. Known exceptions to 8000B are as follows:

- Arizona DHS requires the use of Method 8000C calibration procedures for projects originating in Arizona and under the AZ DHS lab licensure. Refer to the AZDHS calibration guidance document at the following online location: http://www.azdhs.gov/lab/license/tech/8000cmethod.pdf.
- The use of 'averaging' of %RSDs or CCV %D (a.k.a grand mean) is not allowed for projects which use DoD QSM protocols.

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11.2.2. Check for degradation of 4,4'-DDT and Endrin by injecting a standard containing only 4,4'-DDT at 100ppb and Endrin at 50 ppb.

$$\%\,Breakdown = \frac{Total\ DDT\ degratation\ peak\ area\ (DDE + DDD)}{Total\ DDT\ peak\ area\ (DDT + DDE + DDD)} \times 100$$

$$\%\,Breakdown = \frac{\begin{matrix} Total\ endrin\ degratation\ peak\ area \\ endrin\ aldehyde + endrin\ Ketone \end{matrix}}{\begin{matrix} Total\ endrin\ peak\ area \\ endrin + endrin\ aldehyde + endrin\ Ketone \end{matrix}} \times 100$$

If degradation of either DDT or endrin exceeds 15%, perform any necessary maintenance before proceeding with calibration. The breakdown of DDT and Endrin must be measured before samples are analyzed and at the beginning of each analytical sequence.

11.2.3. After determining that degradation is within acceptance, calibrate the system immediately prior to conducting any analyses. Analyze 1.0 µL of each calibration standard (containing internal standards) and tabulate the area against concentration for each compound. For multi-component analytes, only those specified in the project plan or work specification are used for calibration. Calculate response factors (RFs) for each compound relative to one of the internal standards as follows:

$$RF = (A_x C_{is})/(A_{is} C_x)$$

where:
A_x = Area of the compound being measured.
A_{is} = Area of the specific internal standard.
C_{is} = Concentration of the specific internal standard (ng/µL).
C_x = Concentration of the compound being measured (ng/µL).

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Note: For Chlordane, a minimum of 3 peaks must be chosen and for Toxaphene a minimum of 4 peaks must be chosen. The peaks must be characteristic of the compound of interest.

11.2.2.2 Calculate the mean response factor $(\overline{RF_x})$ for each analyte and surrogate from the calibration levels. Calculate standard deviation (SD) and the percent relative standard deviations (%RSD) for each analyte from the mean with:

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$$\%RSD = \frac{(SD)}{(\overline{RF_x})} 100.$$

11.2.2.3 The % RSD should be less than 20% for each compound.

11.2.2.4 If the % RSD for any compound is 20% or less, linearity can be assumed over the calibration range, and the relative response factor for each analyte and surrogate is used to quantitate sample analytes.

11.2.2.5 If the %RSD exceeds 20%, then a linear curve or a quadratic calibration with a correlation coefficient of 0.990 or greater may be used.

11.2.4. Following initial calibration, analyze an ICV standard. The ICV solution must contain all analytes in the calibration standards. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. Evaluate the ICV as described in the SOC-CAL SOP.

Note: DoD acceptance criteria is ±20% from the analytes assigned value.

11.2.5. Continuing Calibration Verification

11.2.5.1. The working calibration curve or calibration response factors must be verified on each analytical sequence by the analysis of one or more mid-range calibration standards (CCV). A CCV must be injected at the start of each 12 hour shift or every 20 samples, whichever is first. The 12 hour window starts with the injection time of the first CCV. The use of internal standard calibration technique does not require that all sample results be bracketed with calibration verification standards.

Note: DoD projects require a CCV analysis every 10 field samples.

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11.2.5.2. The acceptance criteria for all analytes in the CCV analysis are a response (RF or concentration) within ±15% D of the expected value, as compared to the initial calibration. Refer to the SOP *SOC-CAL* .

Note: DoD acceptance criteria is ±20% of the expected value.

11.2.5.3. The measured area of the internal standard must be no more than -50% to + 100% difference from the average area calculated during initial calibration.

11.2.5.4. The retention time of the internal standard must also be evaluated. A retention time shift of >30 sec necessitates system maintenance and reanalysis of CCV.

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11.2.6. Retention Time Windows

11.2.6.1. Establish retention time windows with the GC system in acceptable operating conditions. Make three injections of all analytes throughout the course of a 72-hour period. Serial injections over less than a 72-hour period may result in retention time windows that are too tight. Using retention times from these analyses, calculate retention time windows. Refer to EPA Method 8000B for detailed instructions.

11.2.6.2. Plus or minus three times the standard deviation of the absolute retention times for each standard will be used to define the retention time window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms. In those cases where the standard deviation for a particular standard is zero, the laboratory may use a default window of ± 0.03 minutes. If the peak width is > 0.06 minutes, use a default window of 0.1 minutes.

11.2.6.3. Calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. Retain this data in the method file.

11.3. Sample Analysis

11.3.1. Table 3 indicates the typical operating conditions for the GC. Setup the analysis sequence of sample and QC samples. Also, refer to the SOP for *Sample Batches* for guidance.

11.3.2. Calibrate the system as described in Section 11.2. Evaluate the CCVs as discussed in Section 11.2.4. If any standard falls outside of their daily retention time window, evaluate the chromatogram for possible causes such as carryover from a highly contaminated sample. If a problem related to GC system has been determined to be the cause of retention time shift, perform whatever maintenance is necessary before re-injecting a CCV or recalibrating and proceeding with sample analysis.

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11.3.3. Spike 10 ul of the internal standard 1-Bromo-2-nitrobenzene at 5000 ng/ml into each 1 ml of sample extract to give a final concentration of 50ng/ml. The measured area of the internal standard must be -50 to +100% as measured from the average of the most recent calibration. Any samples falling outside of this criterion require reanalysis.

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11.3.4. The retention time of the internal standard must also be evaluated. A retention time shift of >30 sec requires reanalysis of all affected samples.

11.4. Identification of Analytes

11.4.1. Tentative identification begins with a response that is >3 times the baseline noise level(s/n). The retention time of the peak is compared against the retention time of a known compound in the daily standard chromatogram.

11.4.2. Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window and the s/n ratio of the peak is >3. A tentatively identified compound is confirmed when the retention time for the compound on the confirmatory detector is within the retention time window on that system.

11.4.3. Confirmation of all tentative hits should be made. Confirmation is made by injecting the sample extract on two columns with dissimilar phases simultaneously. If the retention time matches on both columns, then the hit for the analyte is considered a confirmed hit. Refer to the SOP for *Confirmation of Organic Analytes*.

11.4.4. For Chlordane a minimum of 3 peaks must be chosen, and for Toxaphene a minimum of 4 peaks must be chosen for identification purposes. Refer to Section 13 for quantitation procedures for multi-response analytes.

11.5. Perform all necessary calculations as described in Sections 12 and 13.

12. QUALITY CONTROL

12.1. Initial Precision and Recovery Validation

The precision of the extraction procedure and the GC procedure must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made. To do this, four reagent water samples are spiked at a level near t he midpoint of the calibration range (typically the LCS level), then extracting and proceeding with Section 11. The (spiking solution may be prepared from pure standard materials, or purchased as certified solutions. If prepared by the laboratory, stock standards prepared independently from those used for calibration should be used. The concentration of the analytes to be spiked is 20x the MRL.

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12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. Refer to the *SOP for The Determination of Method Detection Limits and Limits of Detection*.

12.2.2. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates (i.e. 1L of reagent water for water MDLs, 10 or 20g sand for soil MDLs) with MDL spiking solution at a level below the MRL. Follow the procedures starting in Section 11 to analyze the samples.

12.3. Ongoing QC Samples required are described in the CAS-Kelso *Quality Assurance Manual* and in the *SOP for Sample Batches*. In general, these include:

12.3.1. Method blank - A method blank is extracted and analyzed with every batch of 20 or fewer samples to demonstrate that there are no method interferences. The method blank must demonstrate that interferences from the analytical and preparation steps are minimized. No target analytes should be detected above the MRL in the method blank.

Note: DoD projects require that no analyte be detected > ½ the RL or 1/10 the regulatory limit, whichever is greater.

12.3.2. A lab control sample (LCS) must be extracted and analyzed with every batch of 20 samples. The water LCS is prepared by adding 50 μL of the matrix spike solution (250uL for Toxaphene/Chlordane) to 1L of reagent water, resulting in concentrations of 0.2 ug/L for single-response analytes (2.5 ug/L for Toxaphene or Chlordane). The soil LCS is prepared by adding 100 μL of spike solution (400uL for Toxaphene/Chlordane) to 20g of sand, resulting in concentrations of 20 ug/kg for single-response analytes (200 ug/kg for Toxaphene or Chlordane). For project-specific low-level extractions, spiking amounts can be adjusted accordingly.

$$\%R = X/TV \times 100$$

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Where X = Concentration of the analyte recovered
TV = True value of amount spiked

Current CAS QC acceptance criteria for lab control samples are listed in Attachment A. Project-specific or program-specific acceptance criteria may supersede CAS criteria. For example, for samples requiring South Carolina DHEC certification the acceptance criteria are 70-130 % recovery. If the lab control sample (LCS) fails acceptance limits for any of the compounds, corrective action must be taken. Corrective action includes recalculation, reanalysis, or re-extraction and reanalysis. Refer to the *Quality Assurance Manual (QAM)* for guidance in evaluating recoveries that exceed LCS limits.

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12.3.3. A matrix spike (MS) and duplicate matrix spike (DMS) must be extracted and analyzed with every batch of 20 samples. The MS/DMS is prepared by adding the same volume of the matrix spike solution to the sample as listed for the LCS, then proceeding with Section 11. Calculate percent recovery (%R) as:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

Calculate Relative Percent Difference (RPD) as:

$$RPD = \frac{R1 - R2}{(R1 + R2)/2} \times 100$$

Where R1 = %recovery of the MS
R2 = %recovery of the DMS

Current CAS QC acceptance criteria for MS/DMS are listed in Attachment A. Project-specific acceptance criteria may supersede CAS criteria. If the MS/DMS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken. Corrective action includes recalculation, reanalysis, or re-extraction and reanalysis.

12.3.4. Surrogate spike is added to every sample, blank and spike prior to extraction. Two surrogate standards (tetrachloro-m-xylene and decachlorobiphenyl) are added to each sample. For water, 100μL of the surrogate spike is added to 1L, resulting in 0.2ug/L. For soil, 200μL of the surrogate spike is added to 20g, resulting in 20ug/kg. Calculate surrogate percent recovery (%R) as:

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$$\%R = S/V \times 100$$

Where S = The amount of surrogate recovered
V = The amount spiked/final volume

12.4. Current CAS QC acceptance criteria for surrogates are listed in Attachment A. Project-specific acceptance criteria may supersede CAS criteria. Both surrogate recoveries must be within the acceptance limits. If either (or both) surrogate is outside of acceptance limits for reasons other than matrix interferences, corrective action must be taken. Corrective actions include recalculation, reanalysis, or re-extraction and reanalysis. The acceptance criteria listed are current criteria, but are subject to change as control limits are updated.

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12.5. Additional QA/QC measures include control charting of QC sample results and periodic analysis of certified reference materials.

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. Both detectors are used as primary and/or confirmatory systems when not interfered with by the sample matrix.

13.2. Calculations

13.2.1. Quantitation of analytes in sample extracts is performed by comparing total area of residue peaks to total area or peaks from the appropriate reference materials.

13.2.2. Quantitation of multi-response analytes:

13.2.2.1. The quantitation of Chlordane, Toxaphene, and other multi-response analytes is accomplished by comparison of the sample chromatogram to that of the authentic standard. All calibration acceptance criteria as described in section 11 must be met before reporting any results. Sample results should then be reported according to the organics confirmation SOP (*SOC-CONF*). Results may be reported from either of the columns as long as all calibration acceptance criteria are met.

13.2.2.2. Once the analyte pattern has been identified, compare the responses of the major peaks in the calibration standard with the peaks observed in the sample extract. The amount of analyte is calculated using the individual calibration factor for each peak and the calibration model selected in section 11. The concentration is determined using the characteristic peaks and then the concentrations are averaged to determine the concentration. If there are interfering peaks that cause the average to be falsely overstated, then that interference peak is Q-deleted using the data system, provided 3 peaks remain for Chlordane and 4 peaks for Toxaphene. The average is then recalculated so that the average more truly represents the concentration in the sample.

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The concentration of each analyte in the sample extract (Cex) is computed.. When $\overline{RF_x}$ is used, calculate the extract concentration as follows:

$$C_{ex} = \frac{(Resp_x)(Amt_{ISTD})}{(Resp_{ISTD})(\overline{RF_x})}$$

Where: C_{ex} = the concentration in the sample extract (ppm);
$Resp_x$ = the peak area of the analytes of interest;
$Resp_{ISTD}$ = the peak area of the associated internal standard;
Amt_{ISTD} = the amount, in ppm, of internal standard added
$\overline{RF_x}$ = the average response from the initial calibration.

13.2.3. The concentration of analytes in the original sample is computed using the following equations:

Aqueous Samples: $$Concentration\ (\mu g / L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$$

Where	Cex	=	Concentration in extract in μg/mL
	Vf	=	Final volume of extract in mL
	D	=	Dilution factor
	Vs	=	Volume of sample extracted, liters

Nonaqueous Samples: $$Concentration\ (mg / Kg) = \frac{(Cex)\ (Vf)\ (D)}{(W)}$$

Where	Cex	=	Concentration in extract in μg/mL
	Vf	=	Final volume of extract in mL
	D	=	Dilution factor
	W	=	Weight of sample extracted in grams.

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13.3. Data Review

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP for *Laboratory Data Review Process* for details.

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13.4. Reporting

13.4.1. Reports are generated using the STEALTH Data Reporting System which compiles the SMO login information. This compilation is then transferred to a file, which STEALTH uses to generate a report. The forms generated may be CAS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.4.2. As an alternative, reports are generated using Excel© templates located in R:\SVG\forms. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The detected analytes, surrogate and matrix spikes are then transferred, by hand or electronically, to the templates.

13.4.3. Sample concentrations are reported when all QC criteria for the analysis has been met or the results are qualified with an appropriate footnote. For Arizona projects the appropriate Arizona qualifier must be used.

14. METHOD PERFORMANCE

14.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

14.2. The method detection limit (MDL) is established using the procedure described in the SOP for *The Determination of Method Detection Limits and Limits of Detection* (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS *Quality Assurance Manual (QAM)*.

15. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

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16. WASTE MANAGEMENT

The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

This method uses non-halogenated solvents and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and disposed of in accordance with Federal and State regulations.

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18. TRAINING OUTLINE

18.1. The following items provide guidelines for training analysts. Refer to the SOP *Documentation of Training (ADM-TRNDOC)* for further requirements.

18.1.1. Review applicable references listed in section 19 and this SOP. Review the MSDS for all chemicals used in the analysis.

18.1.2. Observe the procedure as performed by an experienced analyst at least three times.

18.1.3. Assist in the procedure under the guidance of an experienced analyst for at least one month, preferably three months. During this training period, the analyst is expected to progress from a role of assisting to a role of performing the procedure with minimal oversight.

18.1.4. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests,

IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

18.2. Training is documented following the SOP for Documentation of Technical Personnel Training.

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

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19. REFERENCES

19.1. EPASW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Method 8081A, Revision 1.

19.2. EPASW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Method 8000B, Revision 2.

19.3. EPASW846, Test Methods for Evaluating Solid Waste, Third Edition, Final Update IV, 8081B.

19.4. *DoD Quality systems Manual for Environmental Laboratories* Version 4.1 4/22/2009

19.5. 8000C Method criteria, Arizona DHS, 2/13/2007. Available online at http://www.azdhs.gov/lab/license/tech/8000cmethod.pdf

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20. CHANGES SINCE THE LAST REVISION

20.1. Sec 8.11 Removed AFCEE & HTRW reference.
20.2. Sec 11.2 Removed reference to SC and 8000C calibration procedure.
20.3. Sec 11.1.1 Removed HTRW extraction requirement by 3535.
20.4. Sec 11.2.5 Added DoD acceptance criteria.
20.5. Sec 12.3 Added DoD MB criteria.

TABLE 1

Organochlorine Pesticides
Target Analytes and Method Reporting Limits

Analyte	Water MRL (ug/L)	Soil MRL (ug/kg)
Alpha-BHC	0.01	5.0
Beta-BHC	0.01	5.0
Gamma-BHC (Lindane)	0.01	5.0
Delta-BHC	0.01	5.0
Heptachlor	0.01	5.0
Aldrin	0.01	5.0
Heptachlor Epoxide	0.01	5.0
gamma-Chlordane	0.01	5.0
Endosulfan I	0.01	5.0
alpha-Chlordane	0.01	5.0
Dieldrin	0.01	5.0
4,4'-DDE	0.01	5.0
Endrin	0.01	5.0
Endosulfan II	0.01	5.0
4,4'-DDD	0.01	5.0
Endrin Aldehyde	0.01	5.0
Endosulfan Sulfate	0.01	5.0
4,4'-DDT	0.01	5.0
Endrin Ketone	0.01	5.0
Methoxychlor	0.01	5.0
Chlordane, technical	0.2	100
Toxaphene	0.5	250

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TABLE 2

Low Level Option for Organochlorine Pesticides

Analyte	Soil/ Sediment MRL (ug/kg)	Tissue MRL (ug/kg)	Water MRL (ng/L)
Alpha-BHC	1.0	1.0	0.5
Beta-BHC	1.0	1.0	0.5
Gamma-BHC (Lindane)	1.0	1.0	0.5
Delta-BHC	1.0	1.0	0.5
Heptachlor	1.0	1.0	0.5
Aldrin	1.0	1.0	0.5
Heptachlor Epoxide	1.0	1.0	0.5
gamma-Chlordane	1.0	1.0	0.5
Endosulfan I	1.0	1.0	0.5
alpha-Chlordane	1.0	1.0	0.5
Dieldrin	1.0	1.0	0.5
4,4'-DDE	1.0	1.0	0.5
Endrin	1.0	1.0	0.5
Endosulfan II	1.0	1.0	0.5
4,4'-DDD	1.0	1.0	0.5
Endrin Aldehyde	1.0	1.0	0.5
Endosulfan Sulfate	1.0	1.0	0.5
4,4'-DDT	1.0	1.0	0.5
Endrin Ketone	1.0	1.0	0.5
Methoxychlor	1.0	1.0	0.5
Chlordane, technical	10	10	10
Toxaphene	50	50	25

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TABLE 3

Gas Chromatograph Operating Conditions*

Gas Chromatograph:	Agilent 6890
Injection Port Temperature:	40°C, 40-250°C at 250°C/min., hold for 15 min., 250-325°C at 250°C/min., hold for 5 min.
Oven Temperature Program:	50°C hold for 0.5min., 50-150°C at 40°C/min.; Ramp 13°C/min. to 320°C, hold for 3.92 min.
Detector Temperature:	325°C
Injection Volume:	1 µL (5 µL for ultra low level analysis)
Column 1:	30-m, 0.32mm id, DB-XLB, 0.50µm film thickness or equivalent
Column 2:	30-m, 0.32mm id, DB-35MS, 0.25µm film thickness or equivalent.
Carrier Gas:	Helium
Auxillary Gas:	Argon/Methane
Data System:	HP Enviroquant

*The above instrument temperatures may be modified when determining additional single response or multi-response pesticides.

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TABLE 4

CALIBRATION STANDARD PREPARATION

Initial Calibration Standards*

8081 Intermediate Std.	Initial Concentration	Final Volume	Solvent	Final Concentration	
10 uL	10 ug/mL	50 mL	Hexane	2 ug/L	
25 uL		50 mL		5 ug/L	
100 uL		50 mL		20 ug/L	
250 uL		50 mL		50 ug/L	CCV Standard
500 uL		50 mL		100 ug/L	
1.0 mL		50 mL		200 ug/L	
Chlordane Intermediate Std.	**Initial Concentration**	**Final Volume**	**Solvent**	**Final Concentration**	
5 uL	250 mg/L	50 mL	Hexane	25 ug/L	
10 uL				50 ug/L	
20 uL				100 ug/L	
100 uL				500 ug/L	CCV Standard
200 uL				1000 ug/L	
400 uL				2000 ug/L	
Toxaphene Intermediate Std.	**Initial Concentration**	**Final Volume**	**Solvent**	**Final Concentration**	
50 uL	100 mg/L	50 mL	Hexane	100 ug/L	
125 uL		50 mL		250 ug/L	
250 uL		50 mL		500 ug/L	
500 uL		50 mL		1000 ug/L	CCV Standard
500 uL		25 mL		2000 ug/L	
1250 uL		25 mL		5000 ug/L	

* As needed for projects requiring non-routine additional compounds, similar dilutions are prepared to obtain calibration standards for these compounds.

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ATTACHMENT A

QC Acceptance Criteria
Method Reporting Limits
Method Detection Limits

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Method Information

Method: 8081A
Prep: EPA 3535
Matrix: WATER
Title: SPE OC Pest in Water IS

Source: SW-846
Revision:
Level: LOW

Method ID: MJ1013
Last User: AKamawal
Last Date: 05/28/2009 13:32

Optimal Prep

Sample Amount:	1,000.00 m	0.00
Final Volume:	2.00 ml	0.00

Hold Time

Analytical :	40 days from date Prep
Preparation :	7 days from date Sampled

Require Confirmation?	YES
AFCEE ?	NO

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
1-Bromo-2-nitrobenzene	INT_ST				0.5-2.0		-					577-19-5
1-Bromo-2-nitrobenzene {2}	INT_ST				0.5-2.0		-					CASID39207
1-Bromo-2-nitrobenzene {3}	INT_ST				0.5-2.0		-					CASID39208
1-Bromo-2-nitrobenzene {4}	INT_ST				0.5-2.0		-					CASID39209
1-Bromo-2-nitrobenzene {5}	INT_ST				0.5-2.0		-					CASID39210
2,4'-DDD	MS		0.00057	0.01	70-130	30	16-143	30	ug/L	100		53-19-0
2,4'-DDE	MS		0.00050	0.01	70-130	30	29-123	30	ug/L	100		3424-82-6
2,4'-DDT	MS		0.00059	0.01	70-130	30	28-137	30	ug/L	100		789-02-6
4,4'-DDD	MS		0.00150	0.01	22-141	30	38-140	30	ug/L	100		72-54-8
4,4'-DDE	MS		0.00036	0.01	21-139	30	36-137	30	ug/L	100		72-55-9
4,4'-DDT	MS		0.00058	0.01	30-143	30	45-146	30	ug/L	100		50-29-3
Alachlor	MS		0.0017	0.01	70-130	30	70-130	30	ug/L	100		15972-60-8
Aldrin	MS		0.00040	0.01	11-124	30	10-125	30	ug/L	100		309-00-2
alpha-BHC	MS		0.00033	0.01	30-126	30	43-127	30	ug/L	100		319-84-6
alpha-Chlordane	MS		0.0040	0.01	25-134	30	48-119	30	ug/L	100		5103-71-9
beta-BHC	MS		0.00083	0.01	28-121	30	41-129	30	ug/L	100		319-85-7
Chlordane	TRG		0.015	0.2	11-147	30	34-156	30	ug/L			57-74-9
Chlordane {1}	MULTI		0.015	0.2	11-147	30	34-156	30	ug/L			CASID30272
Chlordane {2}	MULTI		0.015	0.2	11-147	30	34-156	30	ug/L			CASID30273
Chlordane {3}	MULTI		0.015	0.2	11-147	30	34-156	30	ug/L			CASID30274
Chlordane {4}	MULTI		0.015	0.2	11-147	30	34-156	30	ug/L			CASID30275
Chlordane {5}	MULTI		0.015	0.2	11-147	30	34-156	30	ug/L			CASID30276
Chlordane {6}	MULTI		0.015	0.2	11-147	30	34-156	30	ug/L			CASID30277
Chlorpyrifos	MS		0.00083	0.01	70-130	30	10-124	30	ug/L	100		2921-88-2
cis-Nonachlor	MS		0.00060	0.01	70-130	30	12-161	30	ug/L	100		5103-73-1
Decachlorobiphenyl	SURR				17-150		-		PERCE	100		2051-24-3
delta-BHC	MS		0.00057	0.01	35-138	30	47-141	30	ug/L	100		319-86-8
Dieldrin	MS		0.00035	0.01	25-134	30	50-120	30	ug/L	100		60-57-1

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Method: 8081A
Prep: EPA 3535
Matrix: WATER
Title: SPE OC Pest in Water IS

Source: SW-846
Revision:
Level: LOW

Method ID: MJ1013
Last User: AKamawal
Last Date: 05/28/2009 13:32

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
Endosulfan I	MS		0.00044	0.01	23-109	30	30-115	30	ug/L	100		959-98-8
Endosulfan II	MS		0.00040	0.01	23-119	30	32-123	30	ug/L	100		33213-65-9
Endosulfan Sulfate	MS		0.00047	0.01	25-130	30	46-120	30	ug/L	100		1031-07-8
Endrin	MS		0.00068	0.01	35-137	30	53-132	30	ug/L	100		72-20-8
Endrin Aldehyde	MS		0.00046	0.01	10-126	30	30-114	30	ug/L	100		7421-93-4
Endrin Ketone	MS		0.00066	0.01	38-117	30	45-127	30	ug/L	100		53494-70-5
gamma-BHC (Lindane)	MS		0.00044	0.01	30-126	30	42-128	30	ug/L	100		58-89-9
gamma-Chlordane	MS		0.00032	0.01	35-121	30	48-119	30	ug/L	100		5103-74-2
Heptachlor	MS		0.00036	0.01	21-127	30	34-126	30	ug/L	100		76-44-8
Heptachlor Epoxide	MS		0.00032	0.01	24-132	30	45-124	30	ug/L	100		1024-57-3
Hexachlorobenzene	MS		0.00031	0.01	10-124	30	23-97	30	ug/L	100		118-74-1
Hexachlorobutadiene	MS		0.0019	0.01	70-130	30	10-109	30	ug/L	100		87-68-3
Hexachlorocyclopentadiene	MS		0.0011	0.01	70-130	30	70-130	30	ug/L	100		77-47-4
Hexachloroethane	MS		0.0012	0.01	70-130	30	11-99	30	ug/L	100		67-72-1
Isodrin	MS		0.00056	0.01	12-123	30	10-138	30	ug/L	100		465-73-6
Methoxychlor	MS		0.00093	0.01	38-134	30	48-140	30	ug/L	100		72-43-5
Mirex	MS		0.00081	0.01	70-130	30	24-147	30	ug/L	100		2385-85-5
Oxychlordane	MS		0.0010	0.01	70-130	30	22-146	30	ug/L	100		27304-13-8
Permethrin	MS		0.01	0.01	70-130	30	70-130	30	ug/L	100		52645-53-1
Tetrachloro-m-xylene	SURR				10-121		-		PERCE	100		877-09-8
Toxaphene	TRG		0.028	0.5	25-134	30	37-142	30	ug/L			8001-35-2
Toxaphene {1}	MULTI		0.028	0.5	25-134	30	37-142	30	ug/L			CASID30266
Toxaphene {2}	MULTI		0.028	0.5	25-134	30	37-142	30	ug/L			CASID30267
Toxaphene {3}	MULTI		0.028	0.5	25-134	30	37-142	30	ug/L			CASID30268
Toxaphene {4}	MULTI		0.028	0.5	25-134	30	37-142	30	ug/L			CASID30269
Toxaphene {5}	MULTI		0.028	0.5	25-134	30	37-142	30	ug/L			CASID30270
Toxaphene {6}	MULTI		0.028	0.5	25-134	30	37-142	30	ug/L			CASID30271
trans-Nonachlor	MS		0.00092	0.01	70-130	30	27-145	30	ug/L	100		39765-80-5

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Method Information

Method: 8081A
Prep: EPA 3535
Matrix: WATER
Title: OC Pest in UL-Water IS

Source: SW-846
Revision:
Level: LOW

Method ID: MJ958
Last User: AKamawal
Last Date: 06/04/2009 16:25

Optimal Prep

Sample Amount:	1,000.00 m	0.00
Final Volume:	2.00 ml	0.00

Hold Time

Analytical :	40 days from date Prep
Preparation :	7 days from date Sampled

Require Confirmation?	YES
AFCEE ?	NO

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
1-Bromo-2-nitrobenzene	INT_ST				0.5-2.0		-		ng/L	2		577-19-5
1-Bromo-2-nitrobenzene {2}	INT_ST				0.5-2.0		-		ng/L	2		CASID39207
1-Bromo-2-nitrobenzene {3}	INT_ST				0.5-2.0		-		ng/L	2		CASID39208
1-Bromo-2-nitrobenzene {4}	INT_ST				0.5-2.0		-		ng/L	2		CASID39209
1-Bromo-2-nitrobenzene {5}	INT_ST				0.5-2.0		-		ng/L	2		CASID39210
2,4'-DDD	MS		0.068	0.5	70-130.0	30	16-143	30	ng/L	5		53-19-0
2,4'-DDE	MS		0.12	0.5	70-130	30	29-123	30	ng/L	5		3424-82-6
2,4'-DDT	MS		0.13	0.5	70-130	30	28-137	30	ng/L	5		789-02-6
4,4'-DDD	MS		0.21	0.5	22-141	30	38-140	30	ng/L	5		72-54-8
4,4'-DDE	MS		0.19	0.5	21-139	30	36-137	30	ng/L	5		72-55-9
4,4'-DDT	MS		0.17	0.5	30-143	30	45-146	30	ng/L	5		50-29-3
Aldrin	MS		0.11	0.5	11-124	30	10-125	30	ng/L	5		309-00-2
alpha-BHC	MS		0.21	0.5	30-126	30	43-127	30	ng/L	5		319-84-6
alpha-Chlordane	MS		0.27	0.5	25-134	30	48-119	30	ng/L	5		5103-71-9
beta-BHC	MS		0.41	0.5	28-121	30	41-129	30	ng/L	5		319-85-7
Chlordane	TRG		2.0	10	11-147	30	34-156	30	ng/L			57-74-9
Chlordane {1}	MULTI		2.0	10	11-147	30	34-156	30	ng/L	100		CASID30272
Chlordane {2}	MULTI		2.0	10	11-147	30	34-156	30	ng/L	100		CASID30273
Chlordane {3}	MULTI		2.0	10	11-147	30	34-156	30	ng/L	100		CASID30274
Chlordane {4}	MULTI		2.0	10	11-147	30	34-156	30	ng/L	100		CASID30275
Chlordane {5}	MULTI		2.0	10	11-147	30	34-156	30	ng/L	100		CASID30276
Chlordane {6}	MULTI		2.0	10	11-147	30	34-156	30	ng/L	100		CASID30277
Chlorpyrifos	MS		0.13	0.5	70-130	30	10-124	30	ng/L	5		2921-88-2
cis-Nonachlor	MS		0.14	0.5	70-130	30	12-161	30	ng/L	5		5103-73-1
Decachlorobiphenyl	SURR				17-150		-		PERCE	5		2051-24-3
delta-BHC	MS		0.14	0.5	35-138	30	47-141	30	ng/L	5		319-86-8
Dieldrin	MS		0.37	0.5	25-134	30	50-120	30	ng/L	5		60-57-1
Endosulfan I	MS		0.25	0.5	23-109	30	30-115	30	ng/L	5		959-98-8

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Method: 8081A
Prep: EPA 3535
Matrix: WATER
Title: OC Pest in UL-Water IS

Source: SW-846
Revision:
Level: LOW

Method ID: MJ958
Last User: AKamawal
Last Date: 06/04/2009 16:25

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
Endosulfan II	MS		0.35	0.5	23-119	30	32-123	30	ng/L	5		33213-65-9
Endosulfan Sulfate	MS		0.28	0.5	25-130	30	46-120	30	ng/L	5		1031-07-8
Endrin	MS		0.49	0.5	35-137	30	53-132	30	ng/L	5		72-20-8
Endrin Aldehyde	MS		0.21	0.5	10-126	30	30-114	30	ng/L	5		7421-93-4
Endrin Ketone	MS		0.32	0.5	38-117	30	45-127	30	ng/L	5		53494-70-5
gamma-BHC (Lindane)	MS		0.47	0.5	30-126	30	42-128	30	ng/L	5		58-89-9
gamma-Chlordane	MS		0.31	0.5	35-121	30	48-119	30	ng/L	5		5103-74-2
Heptachlor	MS		0.18	0.5	21-127	30	34-126	30	ng/L	5		76-44-8
Heptachlor Epoxide	MS		0.21	0.5	24-132	30	45-124	30	ng/L	5		1024-57-3
Hexachlorobenzene	MS		0.27	0.5	10-124	30	23-97	30	ng/L	5		118-74-1
Hexachlorobutadiene	MS		0.095	0.5	70-130	30	10-109	30	ng/L	5		87-68-3
Hexachloroethane	MS		0.080	0.5	70-130	30	11-99	30	ng/L	5		67-72-1
Isodrin	MS		0.15	0.5	12-123	30	10-138	30	ng/L	5		465-73-6
Methoxychlor	MS		0.28	0.5	38-134	30	48-140	30	ng/L	5		72-43-5
Mirex	MS		0.19	0.5	70-130	30	24-147	30	ng/L	5		2385-85-5
Oxychlordane	MS		0.069	0.5	70-130	30	22-146	30	ng/L	5		27304-13-8
Tetrachloro-m-xylene	SURR				10-121		-		PERCE	5		877-09-8
Toxaphene	TRG		9.0	25	25-134	30	37-142	30	ng/L			8001-35-2
Toxaphene {1}	MULTI		9.0	25	25-134	30	37-142	30	ng/L	100		CASID30266
Toxaphene {2}	MULTI		9.0	25	25-134	30	37-142	30	ng/L	100		CASID30267
Toxaphene {3}	MULTI		9.0	25	25-134	30	37-142	30	ng/L	100		CASID30268
Toxaphene {4}	MULTI		9.0	25	25-134	30	37-142	30	ng/L	100		CASID30269
Toxaphene {5}	MULTI		9.0	25	25-134	30	37-142	30	ng/L	100		CASID30270
Toxaphene {6}	MULTI		9.0	25	25-134	30	37-142	30	ng/L	100		CASID30271
trans-Nonachlor	MS		0.11	0.5	70-130	30	27-145	30	ng/L	5		39765-80-5

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Method Information

Method: 8081A
Prep: EPA 3541
Matrix: SOIL
Title: Reg level OC Pest in Soil By 3541 IS

Source: SW-846
Revision:
Level: LOW

Method ID: MJ1016
Last User: AKamawal
Last Date: 05/06/2009 13:41

Optimal Prep

Sample Amount:	10.00 g	0.00
Final Volume:	10.00 ml	0.00

Hold Time

Analytical :	40 days from date Prep
Preparation :	14 days from date Sampled

Require Confirmation?	YES
AFCEE ?	NO

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
1-Bromo-2-nitrobenzene	INT_ST				0.5-2.0		-					577-19-5
1-Bromo-2-nitrobenzene {2}	INT_ST				0.5-2.0		-					CASID39207
1-Bromo-2-nitrobenzene {3}	INT_ST				0.5-2.0		-					CASID39208
1-Bromo-2-nitrobenzene {4}	INT_ST				0.5-2.0		-					CASID39209
1-Bromo-2-nitrobenzene {5}	INT_ST				0.5-2.0		-					CASID39210
2,4'-DDD	MS		1.4	5	10-184	40	36-148	40	ug/Kg	100		53-19-0
2,4'-DDE	MS		0.94	5	17-172	40	36-145	40	ug/Kg	100		3424-82-6
2,4'-DDT	MS		0.50	5	10-180	40	34-148	40	ug/Kg	100		789-02-6
4,4'-DDD	MS		1.00	5	26-161	40	51-152	40	ug/Kg	100		72-54-8
4,4'-DDE	MS		1.60	5	32-156	40	51-149	40	ug/Kg	100		72-55-9
4,4'-DDT	MS		0.85	5	22-174	40	59-151	40	ug/Kg	100		50-29-3
Aldrin	MS		0.34	5	37-143	40	43-141	40	ug/Kg	100		309-00-2
alpha-BHC	MS		0.35	5	36-145	40	45-150	40	ug/Kg	100		319-84-6
alpha-Chlordane	MS		1.20	5	33-141	40	42-145	40	ug/Kg	100		5103-71-9
beta-BHC	MS		0.50	5	38-148	40	47-149	40	ug/Kg	100		319-85-7
Chlordane	TRG		22.00	100	41-153	40	61-142	40	ug/Kg			57-74-9
Chlordane {1}	MULTI		22.00	100	41-153	40	61-142	40	ug/Kg			CASID30272
Chlordane {2}	MULTI		22.00	100	41-153	40	61-142	40	ug/Kg			CASID30273
Chlordane {3}	MULTI		22.00	100	41-153	40	61-142	40	ug/Kg			CASID30274
Chlordane {4}	MULTI		22.00	100	41-153	40	61-142	40	ug/Kg			CASID30275
Chlordane {5}	MULTI		22.00	100	41-153	40	61-142	40	ug/Kg			CASID30276
Chlordane {6}	MULTI		22.00	100	41-153	40	61-142	40	ug/Kg			CASID30277
Chlorpyrifos	MS		0.66	5	70-130	40	70-130	40	ug/Kg	100		2921-88-2
cis-Nonachlor	MS		0.57	5	10-174	40	44-137	40	ug/Kg	100		5103-73-1
Decachlorobiphenyl	SURR				22-142		-		PERCE	100		2051-24-3
delta-BHC	MS		0.37	5	40-164	40	59-162	40	ug/Kg	100		319-86-8
Dieldrin	MS		0.48	5	37-146	40	50-142	40	ug/Kg	100		60-57-1
Endosulfan I	MS		1.70	5	18-133	40	36-124	40	ug/Kg	100		959-98-8

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Method: 8081A
Prep: EPA 3541
Matrix: SOIL
Title: Reg level OC Pest in Soil By 3541 IS

Source: SW-846
Revision:
Level: LOW

Method ID: MJ1016
Last User: AKamawal
Last Date: 05/06/2009 13:41

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
Endosulfan II	MS		0.86	5	19-147	40	42-130	40	ug/Kg	100		33213-65-9
Endosulfan Sulfate	MS		0.57	5	28-149	40	48-143	40	ug/Kg	100		1031-07-8
Endrin	MS		0.45	5	34-161	40	54-155	40	ug/Kg	100		72-20-8
Endrin Aldehyde	MS		1.40	5	11-147	40	31-139	40	ug/Kg	100		7421-93-4
Endrin Ketone	MS		0.55	5	36-149	40	41-158	40	ug/Kg	100		53494-70-5
gamma-BHC (Lindane)	MS		0.45	5	33-154	40	48-146	40	ug/Kg	100		58-89-9
gamma-Chlordane	MS		0.60	5	27-149	40	42-145	40	ug/Kg	100		5103-74-2
Heptachlor	MS		0.83	5	38-145	40	47-142	40	ug/Kg	100		76-44-8
Heptachlor Epoxide	MS		0.39	5	29-150	40	48-140	40	ug/Kg	100		1024-57-3
Hexachlorobenzene	MS		0.47	5	15-139	40	18-122	40	ug/Kg	100		118-74-1
Hexachlorobutadiene	MS		0.53	5	10-119	40	13-141	40	ug/Kg	100		87-68-3
Hexachloroethane	MS		0.47	5	10-99	40	19-130	40	ug/Kg	100		67-72-1
Isodrin	MS		0.30	5	70-130	40	29-153	40	ug/Kg	100		465-73-6
Methoxychlor	MS		0.61	5	37-162	40	55-153	40	ug/Kg	100		72-43-5
Mirex	MS		0.68	5	10-168	40	46-128	40	ug/Kg	100		2385-85-5
Oxychlordane	MS		0.68	5	16-144	40	39-126	40	ug/Kg	100		27304-13-8
Permethrin	MS		10	10	70-130	40	70-130	40	ug/Kg	100		52645-53-1
Perthane	MS		5	5	70-130	40	70-130	40	ug/Kg	100		72-56-0
Tetrachloro-m-xylene	SURR				25-125		-		PERCE	100		877-09-8
Toxaphene	TRG		37.00	250	10-184	40	37-155	40	ug/Kg			8001-35-2
Toxaphene {1}	MULTI		37.00	250	10-184	40	37-155	40	ug/Kg			CASID30266
Toxaphene {2}	MULTI		37.00	250	10-184	40	37-155	40	ug/Kg			CASID30267
Toxaphene {3}	MULTI		37.00	250	10-184	40	37-155	40	ug/Kg			CASID30268
Toxaphene {4}	MULTI		37.00	250	10-184	40	37-155	40	ug/Kg			CASID30269
Toxaphene {5}	MULTI		37.00	250	10-184	40	37-155	40	ug/Kg			CASID30270
Toxaphene {6}	MULTI		37.00	250	10-184	40	37-155	40	ug/Kg			CASID30271
trans-Nonachlor	MS		0.53	5	10-149	40	42-132	40	ug/Kg	100		39765-80-5

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Method Information

Method: 8081A
Prep: EPA 3541
Matrix: SEDIMENT
Title: OC Pest in LL-Sediment/Soil IS

Source: SW-846
Revision:
Level: LOW

Method ID: MJ1014
Last User: AKamawal
Last Date: 05/15/2009 11:58

Optimal Prep

Sample Amount:	20.00 g	0.00
Final Volume:	4.00 ml	0.00

Hold Time

Analytical :	40 days from date Prep
Preparation :	14 days from date Sampled

Require Confirmation?	YES
AFCEE ?	NO

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
1-Bromo-2-nitrobenzene	INT_ST				0.5-2.0		-					577-19-5
1-Bromo-2-nitrobenzene {2}	INT_ST				0.5-2.0		-					CASID39207
1-Bromo-2-nitrobenzene {3}	INT_ST				0.5-2.0		-					CASID39208
1-Bromo-2-nitrobenzene {4}	INT_ST				0.5-2.0		-					CASID39209
1-Bromo-2-nitrobenzene {5}	INT_ST				0.5-2.0		-					CASID39210
2,2',4,4',5,5'-Hexabromobiphenyl	SURR				70-130		-		PERCE	100		59080-40-9
2,4'-DDD	MS		0.13	1.0	10-184	40	36-148	40	ug/Kg	100		53-19-0
2,4'-DDE	MS		0.16	1.0	17-172	40	36-145	40	ug/Kg	100		3424-82-6
2,4'-DDT	MS		0.058	1.0	10-180	40	34-148	40	ug/Kg	100		789-02-6
4,4'-DDD	MS		0.11	1.0	26-161	40	51-152	40	ug/Kg	100		72-54-8
4,4'-DDE	MS		0.11	1.0	32-156	40	51-149	40	ug/Kg	100		72-55-9
4,4'-DDT	MS		0.17	1.0	22-174	40	59-151	40	ug/Kg	100		50-29-3
Aldrin	MS		0.16	1.0	37-143	40	43-141	40	ug/Kg	100		309-00-2
alpha-BHC	MS		0.11	1.0	36-145	40	45-150	40	ug/Kg	100		319-84-6
alpha-Chlordane	MS		0.10	1.0	33-141	40	42-145	40	ug/Kg	100		5103-71-9
beta-BHC	MS		0.18	1.0	38-148	40	47-149	40	ug/Kg	100		319-85-7
Chlordane	TRG		1.90	10	41-153	40	61-142	40	ug/Kg			57-74-9
Chlordane {1}	MULTI		1.90	10	41-153	40	61-142	40	ug/Kg			CASID30272
Chlordane {2}	MULTI		1.90	10	41-153	40	61-142	40	ug/Kg			CASID30273
Chlordane {3}	MULTI		1.90	10	41-153	40	61-142	40	ug/Kg			CASID30274
Chlordane {4}	MULTI		1.90	10	41-153	40	61-142	40	ug/Kg			CASID30275
Chlordane {5}	MULTI		1.90	10	41-153	40	61-142	40	ug/Kg			CASID30276
Chlordane {6}	MULTI		1.90	10	41-153	40	61-142	40	ug/Kg			CASID30277
Chlorpyrifos	MS		0.15	1.0	70-130	40	70-130	40	ug/Kg	100		2921-88-2
cis-Nonachlor	MS		0.12	1.0	10-174	40	44-137	40	ug/Kg	100		5103-73-1
Decachlorobiphenyl	SURR				22-142		-		PERCE	100		2051-24-3
delta-BHC	MS		0.074	1.0	40-164	40	59-162	40	ug/Kg	100		319-86-8
Dieldrin	MS		0.14	1.0	37-146	40	50-142	40	ug/Kg	100		60-57-1

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Method: 8081A
Prep: EPA 3541
Matrix: SEDIMENT
Title: OC Pest in LL-Sediment/Soil IS

Source: SW-846
Revision:
Level: LOW

Method ID: MJ1014
Last User: AKamawal
Last Date: 05/15/2009 11:58

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
Endosulfan I	MS		0.063	1.0	18-133	40	36-124	40	ug/Kg	100		959-98-8
Endosulfan II	MS		0.14	1.0	19-147	40	42-130	40	ug/Kg	100		33213-65-9
Endosulfan Sulfate	MS		0.11	1.0	28-149	40	48-143	40	ug/Kg	100		1031-07-8
Endrin	MS		0.094	1.0	34-161	40	54-155	40	ug/Kg	100		72-20-8
Endrin Aldehyde	MS		0.12	1.0	11-147	40	31-139	40	ug/Kg	100		7421-93-4
Endrin Ketone	MS		0.093	1.0	36-149	40	41-158	40	ug/Kg	100		53494-70-5
gamma-BHC (Lindane)	MS		0.080	1.0	33-154	40	48-146	40	ug/Kg	100		58-89-9
gamma-Chlordane	MS		0.090	1.0	27-149	40	42-145	40	ug/Kg	100		5103-74-2
Heptachlor	MS		0.12	1.0	38-145	40	47-142	40	ug/Kg	100		76-44-8
Heptachlor Epoxide	MS		0.084	1.0	29-150	40	48-140	40	ug/Kg	100		1024-57-3
Hexachlorobenzene	MS		0.20	1.0	15-139	40	18-122	40	ug/Kg	100		118-74-1
Hexachlorobutadiene	MS		0.21	1.0	10-119	40	13-141	40	ug/Kg	100		87-68-3
Hexachlorocyclopentadiene	MS		1.0	1.0	70-130	40	70-130	40	ug/Kg	100		77-47-4
Hexachloroethane	MS		0.33	1.0	10-99	40	19-130	40	ug/Kg	100		67-72-1
Isodrin	MS		0.17	1.0	70-130	40	29-153	40	ug/Kg	100		465-73-6
Methoxychlor	MS		0.19	1.0	37-162	40	55-153	40	ug/Kg	100		72-43-5
Mirex	MS		0.099	1.0	10-168	40	46-128	40	ug/Kg	100		2385-85-5
Oxychlordane	MS		0.085	1.0	16-144	40	39-126	40	ug/Kg	100		27304-13-8
Tetrachloro-m-xylene	SURR				25-125		-		PERCE	100		877-09-8
Toxaphene	TRG		4.80	50	10-184	40	37-155	40	ug/Kg			8001-35-2
Toxaphene {1}	MULTI		4.80	50	10-184	40	37-155	40	ug/Kg			CASID30266
Toxaphene {2}	MULTI		4.80	50	10-184	40	37-155	40	ug/Kg			CASID30267
Toxaphene {3}	MULTI		4.80	50	10-184	40	37-155	40	ug/Kg			CASID30268
Toxaphene {4}	MULTI		4.80	50	10-184	40	37-155	40	ug/Kg			CASID30269
Toxaphene {5}	MULTI		4.80	50	10-184	40	37-155	40	ug/Kg			CASID30270
Toxaphene {6}	MULTI		4.80	50	10-184	40	37-155	40	ug/Kg			CASID30271
trans-Nonachlor	MS		0.087	1.0	10-149	40	42-132	40	ug/Kg	100		39765-80-5

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Method Information

Method: 8081A
Prep: EPA 3541
Matrix: TISSUE
Title: OC Pest in Tissues IS

Source: SW-846
Revision:
Level: LOW

Method ID: MJ1026
Last User: AKamawal
Last Date: 05/05/2009 13:28

Optimal Prep

Sample Amount:	20.00 g	0.00
Final Volume:	4.00 ml	0.00

Hold Time

Analytical :	40 days from date Prep
Preparation :	365 days from date Sample

Require Confirmation?	YES
AFCEE ?	NO

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
1-Bromo-2-nitrobenzene	INT_ST				0.5-2.0		-					577-19-5
1-Bromo-2-nitrobenzene {2}	INT_ST				0.5-2.0		-					CASID39207
1-Bromo-2-nitrobenzene {3}	INT_ST				0.5-2.0		-					CASID39208
1-Bromo-2-nitrobenzene {4}	INT_ST				0.5-2.0		-					CASID39209
1-Bromo-2-nitrobenzene {5}	INT_ST				0.5-2.0		-					CASID39210
2,2',4,4',5,5'-Hexabromobiphenyl	SURR				70-130		-		PERCE	100		59080-40-9
2,4'-DDD	MS		0.73	1.0	34-141	40	45-131	40	ug/Kg	100		53-19-0
2,4'-DDE	MS		0.37	1.0	34-157	40	42-131	40	ug/Kg	100		3424-82-6
2,4'-DDT	MS		0.16	1.0	43-127	40	45-129	40	ug/Kg	100		789-02-6
4,4'-DDD	MS		0.55	1.0	46-122	40	33-139	40	ug/Kg	100		72-54-8
4,4'-DDE	MS		0.45	1.0	32-138	40	32-143	40	ug/Kg	100		72-55-9
4,4'-DDMU	MS		0.28	1.0	70-130	40	70-130	40	ug/Kg	100		1022-22-6
4,4'-DDT	MS		0.49	1.0	32-131	40	51-136	40	ug/Kg	100		50-29-3
Aldrin	MS		0.74	1.0	39-118	40	45-122	40	ug/Kg	100		309-00-2
alpha-BHC	MS		0.16	1.0	36-121	40	55-123	40	ug/Kg	100		319-84-6
alpha-Chlordane	MS		0.25	1.0	36-123	40	34-128	40	ug/Kg	100		5103-71-9
beta-BHC	MS		0.41	1.0	20-153	40	52-123	40	ug/Kg	100		319-85-7
Chlordane	TRG		3.3	10	70-130	40	36-117	40	ug/Kg			57-74-9
Chlordane {1}	MULTI		3.3	10	70-130	40	36-117	40	ug/Kg		CASID30	CASID30272
Chlordane {2}	MULTI		3.3	10	70-130	40	36-117	40	ug/Kg		CASID30	CASID30273
Chlordane {3}	MULTI		3.3	10	70-130	40	36-117	40	ug/Kg		CASID30	CASID30274
Chlordane {4}	MULTI		3.3	10	70-130	40	36-117	40	ug/Kg		CASID30	CASID30275
Chlordane {5}	MULTI		3.3	10	70-130	40	36-117	40	ug/Kg		CASID30	CASID30276
Chlordane {6}	MULTI		3.3	10	70-130	40	36-117	40	ug/Kg		CASID30	CASID30277
Chlorpyrifos	MS		0.23	1.0	70-130	40	10-110	40	ug/Kg	100		2921-88-2
cis-Nonachlor	MS		0.29	1.0	70-130	40	25-148	40	ug/Kg	100		5103-73-1
Decachlorobiphenyl	SURR				21-128		-		PERCE	100		2051-24-3
delta-BHC	MS		0.20	1.0	39-134	40	49-146	40	ug/Kg	100		319-86-8

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Method: 8081A
Prep: EPA 3541
Matrix: TISSUE
Title: OC Pest in Tissues IS

Source: SW-846
Revision:
Level: LOW

Method ID: MJ1026
Last User: AKamawal
Last Date: 05/05/2009 13:28

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
Dieldrin	MS		0.20	1.0	38-121	40	36-128	40	ug/Kg	100		60-57-1
Endosulfan I	MS		0.22	1.0	22-119	40	21-119	40	ug/Kg	100		959-98-8
Endosulfan II	MS		0.24	1.0	30-122	40	33-113	40	ug/Kg	100		33213-65-9
Endosulfan Sulfate	MS		0.53	1.0	39-124	40	35-134	40	ug/Kg	100		1031-07-8
Endrin	MS		0.28	1.0	46-128	40	42-136	40	ug/Kg	100		72-20-8
Endrin Aldehyde	MS		0.62	1.0	10-113	40	10-114	40	ug/Kg	100		7421-93-4
Endrin Ketone	MS		0.39	1.0	39-129	40	36-133	40	ug/Kg	100		53494-70-5
gamma-BHC (Lindane)	MS		0.21	1.0	47-118	40	56-122	40	ug/Kg	100		58-89-9
gamma-Chlordane	MS		0.26	1.0	33-125	40	33-128	40	ug/Kg	100		5103-74-2
Heptachlor	MS		0.27	1.0	34-126	40	42-127	40	ug/Kg	100		76-44-8
Heptachlor Epoxide	MS		0.18	1.0	32-121	40	38-130	40	ug/Kg	100		1024-57-3
Hexachlorobenzene	MS		0.37	1.0	52-103	40	10-127	40	ug/Kg	100		118-74-1
Hexachlorobutadiene	MS		0.30	1.0	70-130	40	10-159	40	ug/Kg	100		87-68-3
Hexachloroethane	MS		0.32	1.0	70-130	40	10-137	40	ug/Kg	100		67-72-1
Isodrin	MS		0.28	1.0	70-130	40	10-140	40	ug/Kg	100		465-73-6
Methoxychlor	MS		0.48	1.0	26-152	40	41-145	40	ug/Kg	100		72-43-5
Mirex	MS		0.26	1.0	35-127	40	21-149	40	ug/Kg	100		2385-85-5
Oxychlordane	MS		0.39	1.0	39-102	40	20-143	40	ug/Kg	100		27304-13-8
PCB 112	SURR				70-130	40	70-130	40	ug/Kg	100		74472-36-9
PCB 34	SURR				70-130	40	70-130	40	ug/Kg	100		37680-68-5
Tetrachloro-m-xylene	SURR				40-115		-		PERCE	100		877-09-8
Toxaphene	TRG		13	50	29-137	40	41-125	40	ug/Kg			8001-35-2
Toxaphene {1}	MULTI		13	50	29-137	40	41-125	40	ug/Kg			CASID30266
Toxaphene {2}	MULTI		13	50	29-137	40	41-125	40	ug/Kg			CASID30267
Toxaphene {3}	MULTI		13	50	29-137	40	41-125	40	ug/Kg			CASID30268
Toxaphene {4}	MULTI		13	50	29-137	40	41-125	40	ug/Kg			CASID30269
Toxaphene {5}	MULTI		13	50	29-137	40	41-125	40	ug/Kg			CASID30270
Toxaphene {6}	MULTI		13	50	29-137	40	41-125	40	ug/Kg			CASID30271
trans-Nonachlor	MS		0.27	1.0	39-115	40	21-152	40	ug/Kg	100		39765-80-5

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STANDARD OPERATING PROCEDURE

PCBS AS AROCLORS

SOC-8082Ar
Revision 13
June 25, 2009

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Approved By: _____ Supervisor — 6/29/09 Date

Julie Fish — QA Manager — 6/29/09 Date

_____ Laboratory Manager — 6/29/09 Date

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1317 South 13th Avenue
Kelso, Washington 98626

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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL
NUMBER: _____
Initials: _____ Date: _____

PCBS AS AROCLORS

1. SCOPE AND APPLICATION

This procedure is used to determine the concentrations of PCBs as Aroclors using EPA Method 8082. This procedure is typically applied to water, sediment, and soil matrices but may also be applicable to tissue or various miscellaneous waste samples. Table 1 lists the analytes that are determined by this procedure and lists the method reporting limits (MRLs) for each compound in water and soil. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The reported MRL may be adjusted if required for specific project requirements; however, the capability of achieving other reported MRLs must be demonstrated. Method Detection Limits that have been achieved are given in Attachment A. MDLs may change as repeat studies are conducted.

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2. METHOD SUMMARY

2.1. This procedure provides gas chromatographic conditions for the detection of parts-per-billion (ppb) levels of PCBs. The target PCBs are extracted from samples using the appropriate procedure for the sample matrix (see applicable SOP), analyzed, and reported as Aroclors. Liquid samples are extracted using continuous liquid-liquid extraction (Method 3520, CAS SOP EXT-3520) or when allowed, using solid phase extraction (Method 3535, CAS SOP EXT-3535). Soil/sediment samples are extracted using Soxhlet (Method 3540, CAS SOP EXT-3540) or automated Soxhlet extraction (Method 3541, CAS SOP EXT-3541). An aliquot of the extract is injected into the gas chromatograph (GC). The compounds are separated on a fused silica capillary column. Compounds of interest are detected by an electron capture detector. Identification of the analytes of interest is performed by comparing the retention times of the analytes with the respective retention times of an authentic standard and by comparison of elution patterns to those of Aroclor standards. Quantitative analysis is performed by using the authentic standard to produce a calibration factor or calibration curve, and using the calibration data to determine the concentration of an analyte in the extract. The concentration in the sample is calculated using the sample weight or volume and the extract volume.

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2.2. The sensitivity of this method usually depends on the level of interferences rather than on instrument limitations. If interferences prevent detection of the analytes, GPC, florisil column cleanup, sulfur cleanup, or concentrated sulfuric acid cleanups are used to eliminate interferences in the analysis. Refer to section 4.2 for cleanup procedure references.

3. DEFINITIONS

Analysis Sequence - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample extracts interspersed with calibration standards. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

Initial (or Independent) Calibration Verification (ICV) - Initial calibration verification standards which are analyzed after initial calibration with newly prepared standards but prior to sample analysis, in order to verify the validity of the standards used in calibration. Once it is determined there is no systematic error in preparation of the calibration standards, they are considered valid for subsequent calibrations (as methods and expiration dates allow). The ICV standards are prepared from a materials obtained from a source different from that used to prepare calibration standards.

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Matrix Spike/Duplicate Matrix Spike Analysis - In the matrix spike analysis, predetermined quantities of stock solutions of selected Aroclors are added to a sample matrix prior to sample extraction and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, spiked, and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. The concentration of the spike should be at 5 to 10 times the MRL or at levels specified by a project analysis plan.

Standard Curve - A standard curve is a calibration curve which plots concentrations of a known analyte standard versus the instrument response to the analyte.

Surrogate - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples, and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

Method Blank - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

Continuing Calibration Verification Standard (CCV) - A standard injected into the instrument at specified intervals and is used to verify the initial calibration.

Instrument Blank (CCB) - The instrument blank (also called continuing calibration blank) is a volume of clean solvent analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the

3. DEFINITIONS

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Matrix Spike/Duplicate Matrix Spike Analysis - In the matrix spike analysis, predetermined quantities of stock solutions of selected Aroclors are added to a sample matrix prior to sample extraction and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, spiked, and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. The concentration of the spike should be at 5 to 10 times the MRL or at levels specified by a project analysis plan.

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Standard Curve - A standard curve is a calibration curve which plots concentrations of a known analyte standard versus the instrument response to the analyte.

Surrogate - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples, and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

Method Blank - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

Continuing Calibration Verification Standard (CCV) - A standard injected into the instrument at specified intervals and is used to verify the initial calibration.

Instrument Blank (CCB) - The instrument blank (also called continuing calibration blank) is a volume of clean solvent analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the

instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples into other analyses.

4. INTERFERENCES

4.1. 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, late-eluting peaks, especially in the 15% and 50% fractions from the florisil cleanup. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Phthalate contamination is not usually a problem in our laboratory operation.

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4.2. Co-extractables such as lipids, waxes, etc., can be removed via GPC cleanup (CAS SOP SOC-3640A). Certain fractionization cleanups can be used to selectively remove organochlorine pesticides, aiding in Aroclor determination (CAS SOP SOC-3665). The presence of elemental sulfur will result in interferences for most Aroclors. If GPC cleanup is insufficient, cleanup via Method 3660 (CAS SOP SOC-3660) may be used for the removal of sulfur.

5. SAFETY

5.1. The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level.

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5.2. Follow all applicable safety procedures as described in the CAS Safety Manual. A reference file of material safety data sheets is available to all personnel involved in these analyses. CAS also maintains a file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Containers used to collect samples should be purchased pre-cleaned containers. Alternatively, containers may be soap and water washed followed by methanol (or isopropanol) rinsing. The sample containers should be of glass or teflon and have screw-top covers with teflon liners. In situations where teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may <u>not</u> be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic.

6.2. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples

should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g., if an automatic sampler is used), run reagent water through the sampler and use the rinseate as a field blank.

6.3. Samples should be tested for residual chlorine at the time of sampling. For aqueous samples with residual chlorine present, add 3-mL 10% sodium thiosulfate solution per gallon (0.008%).

6.4. Water and soil samples must be iced or refrigerated at 4 ± 2°C from time of collection until extraction. Tissue samples should be stored in accordance with project requirements, typically refrigerated or frozen.

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6.5. Water samples must be extracted within 7 days and the extracts analyzed within 40 days. Soil samples must be extracted within 14 days and the extract analyzed within 40 days. Extracts are stored under refrigeration until analysis.

7. APPARATUS AND EQUIPMENT

7.1. Gas Chromatograph (GC)

7.1.1. Analytical system complete with gas chromatograph suitable for splitless or on-column automated injection into a wide bore capillary column with an electron capture detector (ECD). Use of Large Volume Injection (LVI) is optional. Helium is used as the carrier gas; argon/methane mixture is used for the detector makeup gas (auxiliary gas). Current instrumental systems are identified as follows:

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Instrument I.D.	Analytical System	Routine Matrix
GC09	H-P 5890	Water/Soil/Tissue
GC22	Agilent 6890	Water-LL/Tissue
GC32	Agilent 6890	Water/Soil/Tissue

7.1.2. GC Autosampler: The GC system should be configured with a compatible autosampler for automated injection of standards, samples, and QC samples.

7.1.3. GC Columns - fused silica capillary columns
Column 1: DB-35MS, 30-m x 0.53mm, 1.0um film thickness, or equivalent.
Column 2: DB-XLB, 30-m x 0.53mm, 1.5um film thickness, or equivalent.

Note: Column diameter and film thickness varies depending on the column. Refer to the instrument maintenance logbook for the column used for a specific instrument configuration.

7.1.4. Data System - A computer data system must be interfaced to the GC/ECD. The system allows the continuous acquisition and storage on machine-readable media of chromatographic data obtained throughout the duration of the analysis program.

The computer must have software that includes automated calibration, identification, and quantitation routines. The software must also be capable of integrating the chromatographic peaks abundances. The current version of the manufacturer's software is preferred (Target or HP Chemstation/Enviroquant).

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

8.1. Solvents: Hexane, acetone, methylene chloride, isooctane, and methanol. Pesticide grade or equivalent.

8.2. Standards

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8.2.1. The following commercially prepared stock standards and ICV stock standards are purchased from various reputable vendors and must be certified by the manufacturer. The ICV stock standards are obtained from a different source from the initial calibration. Transfer stock standard solutions into Teflon-sealed screw-cap bottles. Stock standard solutions are stored at -10° C, or at ambient temperature as recommended by the vendor, and protected from light. The expiration date for unopened ampules is the manufacturer's assigned expiration date. If the manufacturer does not assign a date, an expiration date of 1 year from receipt is assigned. Check stock standards frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

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Standard	Concentration	Use
8081/8082 Surrogate	200 ug/mL	ICAL
Aroclor 1016	1000 ug/mL	ICAL
Aroclor 1221	1000 ug/mL	ICAL
Aroclor 1232	1000 ug/mL	ICAL
Aroclor 1242	1000 ug/mL	ICAL
Aroclor 1248	1000 ug/mL	ICAL
Aroclor 1254	1000 ug/mL	ICAL
Aroclor 1260	1000 ug/mL	ICAL
Aroclor 1262	1000 ug/mL	ICAL
Aroclor 1268	1000 ug/mL	ICAL
Aroclor 1016	100 ug/mL	ICV
Aroclor 1221	100 ug/mL	ICV
Aroclor 1232	100 ug/mL	ICV
Aroclor 1242	100 ug/mL	ICV
Aroclor 1248	100 ug/mL	ICV
Aroclor 1254	100 ug/mL	ICV
Aroclor 1260	100 ug/mL	ICV
Aroclor 1262	100 ug/mL	ICV
Aroclor 1268	100 ug/mL	ICV

8.2.2. Intermediate Aroclor calibration standard solutions are made by diluting 1000 ug/mL stock standards 1:20 in hexane. An intermediate surrogate standard is prepared at 5ug/mL by diluting the stock 1:40 in hexane. No ICV intermediates are prepared.

8.2.3. Prepare calibration standards at a minimum of five concentration levels containing equal concentrations of both Aroclors 1016 and 1260 by dilution of the intermediate standards with hexane. One of the concentration levels should be at or below a concentration representing the method reporting limit (MRL). The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. See Table 3 for preparation and concentrations, including standards designated as CCVs. A calibration standard of mid-range concentration is used for the CCV.

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8.2.4. The independent calibration verification (ICV) standards are prepared from stock solutions from a different source from the initial calibration as listed in Table 3. Expiration periods are the same as for equivalent stock and calibration standards.

8.2.5. Standards of the other Aroclors are prepared for use as retention time/pattern standards and to establish calibration factors for these Aroclors (see Table 3).

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8.2.6. Calibration standard solutions are stored at 4 ± 2°C, or at ambient temperature as recommended by the vendor, and must be replaced after six months, or sooner, if comparison with check standards indicate a problem.

8.2.7. A surrogate spiking solution is prepared at 2 ug/mL by making a 1:100 dilution of the surrogate stock standard in acetone. The surrogate solution is stored in the refrigerator for up to six months.

8.2.8. Matrix spike solution: Prepare a spiking solution at 40 ug/mL containing both Aroclor 1016 and 1260 by diluting the 1000 ug/mL stock standards 1:25 with acetone.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument.

9.2. Carrier gas - Inline purifiers or scubbers should be in place for all sources of carrier gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3. Gas Chromatograph

9.3.1. Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance includes changing the injection port liner, seal, washer, o-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chomatographic performance. In some cases liners and seals may be cleaned and re-used.

9.3.2. Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column cutting tool.

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9.3.3. Over time, the column will exhibit poorer overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

10. RESPONSIBILITIES

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10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in 8082, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. Water samples (1L) are extracted at a pH of 5-9 with methylene chloride, using Method 3520 (CAS SOP EXT-3520) or when allowed, using solid phase extraction (CAS SOP EXT-3535). Refer to the applicable extraction SOP. For extraction by 3535, acidification of the sample prior to extraction may be allowable if project objectives and performance requirements of methods 3535 and 8081 are met. An ultra low-level water option may be used, where a 1L sample

amount and a final extract volume of 2mL is used. Large Volume Injectors are typically used with this option.

Note: Project-specific or regulation-specific extraction methods may apply. For projects originating from South Carolina and under the SC DHEC lab certification, use the 3520 extraction method only.

11.1.2. Soil/sediment samples are extracted using either EPA Method 3540 (CAS SOP EXT-3540) or method 3541 (CAS SOP EXT-3541). Refer to the applicable extraction SOP. A low-level sediment option may be used where the sample weight of 40g (20g dry weight) and a final extract volume of 4mL is used.

11.1.3. Additional sample cleanup procedures may be employed as appropriate for the samples. Refer to the section on interferences and the appropriate CAS SOP.

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11.2. Calibration

11.2.1. The default calibration procedures are given in the following sections and, at the time of this revision, follow Method 8000B. The analyst should also refer to the CAS SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOC-CAL), where calibration procedures and policies are described. The calibration procedure(s) and options chosen must follow the CAS protocols.

Note: Certain state or program protocols have specific procedures for calibration. This may include all or part of Method 8000C. The analyst must ensure that the correct procedures are used. Known exceptions to 8000B are as follows:

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- Arizona DHS requires the use of Method 8000C calibration procedures for projects originating in Arizona and under the AZ DHS lab licensure. Refer to the AZDHS calibration guidance document at the following online location: http://www.azdhs.gov/lab/license/tech/8000cmethod.pdf.

Note: The use of 'averaging' of %RSDs or CCV %D (a.k.a grand mean) is not allowed for projects under the DoD QSM protocols.

Note: The use of quadratic regression calibration is not allowed for projects (samples) originating from South Carolina and under the SC DHEC lab certification.

11.2.2. Prepare a minimum of 5 calibration standards containing equal concentrations of both Aroclor 1016 and 1260 by dilution of the stock standard(s) with isooctane or hexane. Single standards of each of the other target Aroclors are required to aid the analyst in pattern recognition. Once the linearity of the detector has been demonstrated using Aroclor 1016/1260 standards, the single standards of the remaining target Aroclors are also used to determine the calibration factor for each

Aroclor. Prepare a standard for each of the other Aroclors. The concentrations should correspond to the mid-point of the linear range of the detector.

A minimum of 3 peaks must be chosen for each Aroclor, and preferably 6 peaks. The peaks must be characteristic of the Aroclor in question. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of 3 to 6 peaks should include at least one peak that is unique to that Aroclor. Use at least five peaks for the Aroclor 1016/1260 mixture, none of which should be found in both of these Aroclors. Establish the retention time window position using the mid point of the ICAL range before processing the calibration curve.

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11.2.3. Calibrate the system immediately prior to conducting any analyses. Refer to Table 2 for instrument conditions. Starting with the standard of lowest concentration, analyze each 1016/1260 calibration standard and tabulate response (peak area) versus the concentration in the standard. Refer to Table 2 for instrument conditions. Calculate the ratio of the response to the amount injected the (calibration factor) for each analyte at each standard concentration. For 1016/1260 and DCB, the Relative Standard Deviation (RSD) must be less than 20% when average response factor is used.

11.2.4. Analyze each of the single-point calibration standards of the other target Aroclors. Calculate the calibration factor (CF) for each analyte at each standard concentration.

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11.2.5. Each calibration of each Aroclor is verified by an independent source. Prepare an independent calibration verification standard (ICV) by dilution of a stock solution purchased from a different vendor and analyze immediately after each initial calibration. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. Evaluate the ICV as described in the SOP for Calibration of Instruments for Organics Chromatographic Analyses.

Note: DoD ICV acceptance criteria is ± 20% from the analytes assigned value.

11.3. Calibration Verification

11.3.1. The working calibration curve or calibration factor must be verified on each analytical sequence by the analysis of one or more mid-range calibration standards (CCV). A standard (CCV) must be injected at the start of each sequence and after each set of sample extracts (every 10 samples or every 12 hours, whichever is first) in the analysis sequence.

Note: DoD projects require a CCV analysis every 10 field samples.

11.3.2. The acceptance criterion for all analytes in the CCV analysis is a response (RF or concentration) within ±15% D of the expected value, as compared to the initial calibration. Refer to the CAS protocols for organics analyses calibration for CCV evaluation protocols.

Note: DoD CCV acceptance criteria is ± 20% from the analytes assigned value

11.4. Retention Time Windows

11.4.1. Pattern recognition/matching and retention times are used for the identification of PCBs as Aroclors.

11.4.2. Establish retention time windows for the peaks used for quantitation with the GC system in acceptable operating condition. Make three injections of all analytes throughout the course of a 72-hour period. Serial injections over less than a 72-hour period may result in retention time windows that are too tight. Using retention times from these analyses, calculate retention time windows. Refer to EPA Method 8000B for detailed instructions.

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11.4.3. Plus or minus three times the standard deviation of the absolute retention times for each standard will be used to define the retention time window; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms. In those cases where the standard deviation for a particular standard is zero, the laboratory may use a default window of ± 0.03 minutes. If the peak width is > 0.06 minutes, use a default window of 0.1 minutes.

11.4.4. Calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. Retain this data in the method file.

11.5. Gas Chromatography

11.5.1. Set up an analytical sequence for the standards and samples to be analyzed. Calibrate the system as described in Section 11.2. Refer to Table 2 for typical instrument operating conditions. The same conditions must be used for samples as for calibration and QC analyses. Ensure that the instrument configuration is correct and that any necessary maintenance has been performed. Figure 1 shows a typical analysis sequence.

Note: For DoD projects, the CCB must be analyzed following the CCV. Instrument blanks/CCBs may not be analyzed prior to QC samples or standards.

11.5.2. Evaluate the CCVs as indicated in Section 11.3. Use the standards interspersed throughout the sample analysis sequence to evaluate the qualitative performance of the GC system including positioning of the retention time window. If any retention time shift which would impede analyte identification is evident (as shown by Aroclor pattern irregularities or the surrogate falling outside of the retention

time window), evaluate the chromatogram for possible causes such as carryover from a highly contaminated sample. If a problem related to GC system has been determined to be the cause of retention time shift, perform whatever maintenance is necessary before reanalyzing the CCV or recalibrating and proceeding with sample analysis. All samples that were injected after the sample exceeded the criteria must be reinjected if initial analysis indicated the presence of any analytes of interest.

FIGURE 1

Analysis Sequence

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Initial Calibration Blank	ICB
Standard 1	1221 midpoint
Standard 2	1232 midpoint
Standard 3	1242 midpoint
Standard 4	1248 midpoint
Standard 5	1254 midpoint
Standard 6	1262 midpoint
Standard 7	1268 midpoint
Standards 8-12	1016/1260 ICAL standards
Standards 13-20	ICVs for 1016, 1221, 1232, 1242, 1254, 1260, 1262, 1268
Continuing Calibration Blank	CCB
Continuing Calibration Verification	CCV
Method Blank	
Laboratory Control Sample	LCS
Sample 1	
Sample 2	
Sample 3	
Sample 4	
Sample 5	
Sample 6	
Sample 7	
Sample 8	
Matrix Spike	
Duplicate Matrix Spike	
Continuing Calibration Blank	CCB
Continuing Calibration Verification	CCV
Sample 9	
Sample 10	
etc.	
etc.	

Continuing Calibration Blank	CCB
Continuing Calibration Verification	CCV

Note: If a valid 1016/1260 initial calibration is in place, a new set of initial calibration standards may not be required. In this case, CCV standards are analyzed at the beginning of the sequences in place of initial calibration standards.

Note: For DoD projects, the CCB must be analyzed following the CCV. Instrument blanks and CCBs may not be analyzed prior to QC samples or standards.

11.6. Troubleshooting

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11.6.1. Initial calibration – If the initial calibration fails to meet the criteria, or the ICV fails (indicating a calibration problem), the following steps may be taken, depending on nature of the problem.

- Recheck the information entered into the software used for calibration and quantitation. Verify the standard values are correct and datafiles are correct. If incorrect, repeat the calibration with the correct information.
- Recheck standards preparation to ensure that standards are correct. Re-prepare and reanalyze if needed.
- Ensure that proper preventive maintenance was performed. Repeat the preventive maintenance if necessary and reanalyze the calibration.
- If calibration problems persist or more substantial calibration problems exist, corrective maintenance or repair may be needed. This includes such measures as column changes, detector maintenance, or GC repair. This will depend on the nature of the problem. Following any such maintenance, repeat the calibration.

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11.6.2. Continuing calibration – If the CCV analysis fails to meet the criteria, the following steps may be taken, depending on nature of the problem.

- Recheck the information entered into the software used for calibration and quantitation. Verify the standard values are correct and datafiles are correct. If incorrect, repeat the calibration with the correct information.
- Recheck standards preparation to ensure that standards are correct and that the correct standard is used as the CCV. Re-prepare and reanalyze if needed. Note that NELAC and DoD requirements apply when multiple CCVs are analyzed.
- Ensure that proper preventive maintenance was performed. Repeat the preventive maintenance if necessary and reanalyze the CCV.
- If calibration problems persist or more substantial calibration problems exist, corrective maintenance or repair may be needed. This includes such measures as column changes, detector maintenance, or GC repair. This

will depend on the nature of the problem. Following any such maintenance, repeat analysis of the CCV and necessary samples. Major maintenance will require recalibration. Note that some samples may quickly deteriorate the system to the point that closing CCVs will not pass. This should be verified through a second run of the samples and documented.

12. QA/QC REQUIREMENTS

12.1. Refer to Section 8.0 of Method 8082 for general QC protocol. In addition to instrument criteria for calibration, the ability of each analyst/instrument to generate acceptable accuracy and precision must be documented prior to sample analysis (IPR study). This must be validated before analyses of samples begin, or whenever significant changes to the procedures have been made. To do this, four reagent water samples are spiked with each target analyte, extracted, and analyzed. Refer to Method 8082 for this requirement and acceptance criteria.

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12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates with a MDL spiking solution (at a level below the MRL) for each target analyte, extract, and analyze. The MDL studies should be done for each matrix, prep method, and instrument. Refer to the CAS *SOP for The Determination of Method Detection Limits*.

12.2.2. Calculate the average concentration found (x) in the sample concentration, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study should be done annually.

12.3. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the *SOP for Sample Batches*. The Quality Assurance Manual (Section 12) provides guidance for interpretation of QC results. Ongoing QC samples include:

12.3.1. Method blank - A method blank is extracted and analyzed with every batch of 20 or fewer samples to demonstrate that there are no method interferences. The method blank must demonstrate that interferences from the analytical and preparation steps minimized. No target analytes should be detected above the MRL in the method blank.

Note: DoD projects require no analytes detected > ½ the RL or > 1/10 the regulatory limit.

12.3.2. A lab control sample (LCS) must be extracted and analyzed with every batch of 20 samples. The water LCS is prepared by adding 50 μL of the matrix spike solution to 1L of reagent water, resulting in a concentration of 2.0 ug/L. The soil LCS is prepared by adding 100 μL of spike solution to 20g of sand, resulting in a concentration of 200 ug/kg. For project-specific low-level extractions, spiking amounts can be adjusted accordingly. Calculate percent recovery (%R) as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

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Acceptance criteria for lab control samples are listed in Attachment A. Project-specific or program-specific acceptance criteria may supersede CAS criteria. For example, for samples requiring South Carolina DHEC certification the acceptance criteria are 70-130 % recovery. If the lab control sample (LCS) fails acceptance limits for any of the compounds, the analyst must evaluate the system and calibration. If no problems are found, corrective action must be taken. The acceptance criteria listed are current criteria, but are subject to change as control limits are updated.

12.3.3. A matrix spike/duplicate matrix spike (MS/DMS) must be extracted and analyzed with every batch of 20 or fewer samples. The MS/DMS is prepared by adding the same volume of the matrix spike solution to the sample as listed for the LCS, then proceeding with the entire extraction and analysis. Calculate percent recovery (%R) as follows:

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$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

Calculate Relative Percent Difference (RPD) as:

$$RPD = \frac{R1 - R2}{(R1 + R2)/2} \times 100$$

Where R1 = % recovery of the MS
R2 = % recovery of the DMS

Acceptance criteria for matrix spikes are listed in Attachment A. If the MS/DMS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken. The acceptance criteria listed are current criteria, but are subject to change as control limits are updated.

12.3.4. Surrogate spike is added to every sample, blank and spike prior to extraction. Two surrogate standards (tetrachloro-m-xylene and decachlorobiphenyl) are added to each sample. For water, 100μL of the surrogate spike is added to 1L, resulting in 0.2 ug/L. For soil, 200μL of the surrogate spike is added to 20g, resulting in 20 ug/kg. Calculate surrogate percent recovery (%R) as:

$$\%R = S/V \times 100$$

Where S = The amount of surrogate recovered
V = The amount spiked/final volume

The acceptance limits for the surrogates are given in Attachment A. Both surrogate recoveries must be within the acceptance limits. If either (or both) surrogate is outside of acceptance limits for reasons other than matrix interferences, corrective action must be taken. Corrective actions include recalculation, reanalysis, or reextraction and reanalysis. The acceptance criteria listed are current criteria, but are subject to change as control limits are updated.

12.3.5. Additional QA/QC measures include control charting of QC sample results.

12.4. Troubleshooting

12.4.1. Method blanks – If the method blank fails to meet the criteria, the sample data in the associated batch should be examined. If all samples and QC have hits for the analyte, samples and QC should be re-extracted and reanalyzed as necessary (samples with higher level hits may not need reanalysis). It should be verified through the analysis of instrument blanks that the problem is isolated to either the GC of the sample preparation. If the problem is isolated to the MB, the data may be flagged and narrated. Also refer to Section 12 of the QA Manual for flowcharts.

The source of MB contamination should be isolated and corrected as soon as possible to prevent further failures.

12.4.2. Surrogate, LCS and MS QC Data – Refer to Section 12 of the QA Manual for flowcharts addressing data interpretation and corrective actions.

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13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. Identification of PCBs as Aroclors

13.1.1. To identify Aroclors, compare the chromatographic pattern of the sample to known Aroclor standards. Tentative identification of PCBs as Aroclors is made when the pattern of peaks in the sample chromatogram matches the pattern of peaks in the Aroclor standard itself. There also needs to be agreement between the retention times and response ratios of the 3-6 selected quantitation peaks in the sample chromatogram and the Aroclor standard.

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13.1.2. Tentative identification of analytes must be confirmed using a second GC column of dissimilar phase. Identify the Aroclor by comparing the chromatographic pattern of the sample to known Aroclor standards analyzed on the same column. Confirmation of the Aroclor is made when the sample chromatogram matches the pattern of peaks in the Aroclor being confirmed. Quantitations for the 2 columns must agree ($\leq$ 40%RPD) to confirm the identification. If interferences or other sample anomalies make the RPD value >40% but the analyst makes a positive identification, the basis of the identification must be documented and the data user notified of the discrepancy (see section 13.2).

13.2. Sample matrix difficulties

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13.2.1. Weathering of PCBs in the environment and changes resulting from waste treatment processes may alter the pattern of a specific Aroclor so it does not closely match an Aroclor standard. The earlier eluting peaks will often diminish in comparison to the later eluting peaks. If this is observed, alternate peaks may be selected to aid identification to reduce quantitation bias.

13.2.2. Metabolism by organisms may also alter the pattern since individual PCB congeners are metabolized at different rates. When working with tissue samples, the 40% RPD criteria for confirmation may not be met.

13.2.3. Samples may also include mixtures of two or more Aroclors. To the extent possible, identify and quantify each Aroclor.

13.2.4. High amounts of organochlorine pesticides in the sample may interfere with identification. If this is observed, alternate peaks may be selected to aid identification to reduce quantitation bias. Certain fractionization cleanups can be used to selectively remove organochlorine pesticides, aiding in Aroclor determination (Acid cleanup, CAS SOP SOC-3665).

13.2.5. For all of these reasons a high level of analyst expertise is required to interpret complex chromatograms.

13.3. Quantitation of PCBs as Aroclors:

13.3.1. The quantitation of PCBs as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard or standards. All calibration acceptance criteria as described in section 11must be met before reporting any results. Sample results should then be reported according to the organics confirmation Sop (SOC-CONF). Results may be reported from either column if all calibration acceptance criteria as described in section 11are met.

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13.3.2. Once the Aroclor pattern has been identified, compare the responses of 3 to 6 major peaks in the calibration standard of that Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the individual calibration factor for each of the 3 to 6 peaks and the calibration model selected in section 11. The concentration is determined using the 3 to 6 characteristic peaks and then the concentrations are averaged to determine the concentration of the Aroclor. If there are interfering peaks with the 3 to 6 quantitation peaks that cause Aroclor average to be falsely overstated, then that interference peak is Q-deleted using the data system and the average is recalculated so that the average more truly represents the concentration in the sample. This often occurs when there are more than one Aroclor in a sample extract or if pesticides are present. Quantitation of mixed Aroclors will require the selection of peaks that are not shared in common by both Aroclors.

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13.3.3. For samples with severe matrix interferences, the quantitation may be performed by measuring the total area of the PCB pattern and quantifying on the basis of the Aroclor standard that is most similar to the sample. Any peaks that are not identifiable as PCBs should be subtracted from the total area. When the quantitation option is used, the sample problems should be described for the data user and quantification procedure documented.

13.3.4. Using the data system, calculate the concentration in the extract using the calibration model chosen for calibration (see the SOP SOC-CAL).

13.3.5. Using the data system, calculate the concentration of each analyte in the sample extract (Cex) μg/ml units using the calibration factor or calibration curve (Section 11). The sample concentration computed using the following equations:

Aqueous Samples:

$$Concentration\ (\mu g / L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$$

Where Cex = Concentration in extract in μg/ml
Vf = Final volume of extract in ml
D = Dilution factor
Vs = Volume of sample extracted, liters

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Nonaqueous Samples:

$$Concentration\ (mg / Kg) = \frac{(Cex)\ (Vf)\ (D) \times 1{,}000}{(W) \times 1{,}000}$$

Where Cex = Concentration in extract in μg/ml
Vf = Final volume of extract in ml
D = Dilution factor
W = Weight of sample extracted. The wet or dry weight may be used, depending upon the specific client requirements.

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13.4. Sample concentrations are reported when all QC criteria for the analysis has been met or the results are qualified with a footnote.

13.5. Data Review

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP for Laboratory Data Review Process for details.

13.6. Reporting

13.6.1. Reports are generated using the STEALTH Data Reporting System which compiles the SMO login information. This compilation is then transferred to a file, which STEALTH uses to generate a report. The forms generated may be CAS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.6.2. As an alternative, reports are generated using Excel© templates located in R:\SVG\forms. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The detected analytes, surrogate and matrix spikes are then transferred, by hand or electronically, to the templates.

13.6.3. Sample concentrations are reported when all QC criteria for the analysis have been met or the results are qualified with an appropriate footnote. For Arizona projects the appropriate Arizona qualifier must be used.

14. CONTENGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

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Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the SOP for Nonconformity and Corrective Action for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for Report Generation or in project-specific requirements.

15. METHOD PERFORMANCE

15.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

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15.2. The method detection limit (MDL) is established using the procedure described in the SOP for The Determination of Method Detection Limits (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16. TRAINING

16.1. Training outline

16.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

16.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst for a period of 3 months. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

16.1.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

16.2. Training is documented following the SOP for Documentation of Technical Personnel Training.

16.2.1. NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

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17. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

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18. WASTE MANAGEMENT

The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the laboratory Safety Manual, Sections 6 and 7.

19. REFERENCES

18.1. Polychlorinated Biphenyls (PCBs) as Aroclors, Method 8082, Revision 0, EPA Test Methods for Evaluating Solid Waste, SW-846, Final Update III, December 1996.

18.2. EPASW846, Test Methods For Evaluating Solid Waste, Third Edition, Update III, December 1996, Method 8000B, Revision 2.

18.3. 8000C Method criteria, Arizona DHS, 2/13/2007. Available online at http://www.azdhs.gov/lab/license/tech/8000cmethod.pdf

18.4. *DoD Quality Systems Manual for Environmental Laboratories* Version 4.1 4/22/09.

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20. CHANGES SINCE THE LAST REVISION

20.1. Sec 11.2 Removed SC and method 8000C reference.
20.2. Sec 7.1.1 Removed HTRW prep requirements.
20.3. Sec 11.2 Added DoD grand mean prohibition.
20.4. Sec 11.2.2 Added TR window positioning step.
20.5. Sec 11.2.5 Added DoD ICV criteria.
20.6. Sec 11.3.2 Added DoD acceptance criteria.
20.7. Sec 11.5.2 Added RT window positioning to CCV.
20.8. Sec 12.3 Added DoD MB criteria.
20.9. Sec 11.1.1 Changed ULL optimal amount form 2 L to 1 L.

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TABLE 1

Target Analytes, Method Reporting Limits, and Method Detection Limits

Aroclor	Water MRL (ug/L)	Water (low level) MRL (ug/L)	Water (ultra low level) MRL (ug/L)
Aroclor 1016	0.20	0.02	0.005
Aroclor 1221	0.40	0.04	0.01
Aroclor 1232	0.20	0.02	0.005
Aroclor 1242	0.20	0.02	0.005
Aroclor 1248	0.20	0.02	0.005
Aroclor 1254	0.20	0.02	0.005
Aroclor 1260	0.20	0.02	0.005

Aroclor	Soil/Sediment MRL (mg/kg)	Soil/Sediment (low level) MRL (ug/kg)
Aroclor 1016	0.10	10
Aroclor 1221	0.20	20
Aroclor 1232	0.10	10
Aroclor 1242	0.10	10
Aroclor 1248	0.10	10
Aroclor 1254	0.10	10
Aroclor 1260	0.10	10

Aroclor	Tissue MRL (ug/kg)
Aroclor 1016	10
Aroclor 1221	20
Aroclor 1232	10
Aroclor 1242	10
Aroclor 1248	10
Aroclor 1254	10
Aroclor 1260	10

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TABLE 2

Gas Chromatograph Operating Conditions*

Gas Chromatograph:	Hewlett-Packard Model 5890 or equivalent w/ECD
Injection Port Temperature:	300°C
Oven Temperature Program:	160°C for 0.5 min., 30°/min ramp to 210°C, then 10°/min. to 300°C, hold 11.03 min.
Detector Temperature:	350°C
Injection Volume:	1 μL
Column:	30 m, DB-35MS and 30 m DB-XLB*
Carrier Gas:	Helium
Auxillary Gas:	Argon/Methane
Data System:	HP Chemstation (acquisition) and Target (data)

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* The instrument temperatures may be modified depending on the instrument used. Also, the GC column diameter and film thickness depend on the instrument used. All conditions must be the same for initial calibration, continuing calibration, sample, and QC analyses.

TABLE 3

CALIBRATION STANDARD PREPARATION

1016/1260 Initial Calibration Standards (prepared in hexane)

Aroclor 1016 50 ug/mL Intermediate	Aroclor 1260 50 ug/mL Intermediate	Surrogate 5 ug/mL Intermediate	Final Volume	Final Concentration Aroclors	Final Concentration Surrogates
12.5 uL	12.5 uL	12.5 uL	25 mL	25 ug/L	2.5 ug/L
25 uL	25 uL	25 uL	25 mL	50 ug/L	5.0 ug/L
250 uL	250 uL	250 uL	25 mL	500 ug/L	50 ug/L
500 uL	500 uL	500 uL	25 mL	1000 ug/L*	100 ug/L*
1000 uL	1000 uL	1000 uL	25 mL	2000 ug/L	200 ug/L
2500 uL	2500 uL	2500 uL	25 mL	5000 ug/L	500 ug/L

* CCV Standard

Single-Point Calibration Standards

Intermediate Standard (1000 ug/mL)	Aliquot	Final Volume	Solvent	Final Concentration
Aroclor 1221	25 uL	25 mL	Hexane	1000 ug/L
Aroclor 1232	25 uL	25 mL	↓	1000 ug/L
Aroclor 1242	25 uL	25 mL	↓	1000 ug/L
Aroclor 1248	25 uL	25 mL	↓	1000 ug/L
Aroclor 1254	25 uL	25 mL	↓	1000 ug/L
Aroclor 1262	25 uL	25 mL	↓	1000 ug/L
Aroclor 1268	25 uL	25 mL	↓	1000 ug/L

ICV Standards

ICV Stock Standard (100 ug/mL)	Aliquot	Final Volume	Solvent	Final Concentration
Aroclor 1016	250 uL	25 mL	Hexane	1000 ug/L
Aroclor 1221	250 uL	25 mL	↓	1000 ug/L
Aroclor 1232	250 uL	25 mL	↓	1000 ug/L
Aroclor 1242	250 uL	25 mL	↓	1000 ug/L
Aroclor 1248	250 uL	25 mL	↓	1000 ug/L
Aroclor 1254	250 uL	25 mL	↓	1000 ug/L
Aroclor 1260	250 uL	25 mL	↓	1000 ug/L
Aroclor 1262	250 uL	25 mL	↓	1000 ug/L
Aroclor 1268	250 uL	25 mL	↓	1000 ug/L

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ATTACHMENT A

QC Acceptance Criteria
Method Reporting Limits
Method Detection Limits

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Method Information

Method: 8082
Prep: EPA 3535
Matrix: WATER
Title: PCB Aroclors in Water

Source: SW-846
Revision:
Level: LOW

Method ID: MJ579
Last User: AKamawal
Last Date: 06/18/2009 11:30

Optimal Prep

Sample Amount:	1,000.00 m	0.00
Final Volume:	2.00 ml	0.00

Hold Time

Analytical :	40 days from date Prep
Preparation :	7 days from date Sampled

Require Confirmation?	YES
AFCEE ?	NO

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
2,2',4,4',5,5'-Hexabromobiphenyl	SURR				70-130		-		PERCE	100		59080-40-9
Aroclor 1016	MS		0.0094	0.2	10-161	30	32-115	30	ug/L	1000		12674-11-2
Aroclor 1016 {1}	MULTI		0.0094	0.2	10-161	30	32-115	30	ug/L	1000		CASID30221
Aroclor 1016 {2}	MULTI		0.0094	0.2	10-161	30	32-115	30	ug/L	1000		CASID30222
Aroclor 1016 {3}	MULTI		0.0094	0.2	10-161	30	32-115	30	ug/L	1000		CASID30223
Aroclor 1016 {4}	MULTI		0.0094	0.2	10-161	30	32-115	30	ug/L	1000		CASID30224
Aroclor 1016 {5}	MULTI		0.0094	0.2	10-161	30	32-115	30	ug/L	1000		CASID30225
Aroclor 1221	TRG		0.020	0.4	-		-		ug/L			11104-28-2
Aroclor 1221 {1}	MULTI		0.020	0.4	-		-		ug/L			CASID30226
Aroclor 1221 {2}	MULTI		0.020	0.4	-		-		ug/L			CASID30227
Aroclor 1221 {3}	MULTI		0.020	0.4	-		-		ug/L			CASID30228
Aroclor 1221 {4}	MULTI		0.020	0.4	-		-		ug/L			CASID30229
Aroclor 1221 {5}	MULTI		0.020	0.4	-		-		ug/L			CASID30230
Aroclor 1232	TRG		0.023	0.2	-		-		ug/L			11141-16-5
Aroclor 1232 {1}	MULTI		0.023	0.2	-		-		ug/L			CASID30231
Aroclor 1232 {2}	MULTI		0.023	0.2	-		-		ug/L			CASID30232
Aroclor 1232 {3}	MULTI		0.023	0.2	-		-		ug/L			CASID30233
Aroclor 1232 {4}	MULTI		0.023	0.2	-		-		ug/L			CASID30234
Aroclor 1232 {5}	MULTI		0.023	0.2	-		-		ug/L			CASID30235
Aroclor 1242	TRG		0.013	0.2	-		-		ug/L			53469-21-9
Aroclor 1242 {1}	MULTI		0.013	0.2	-		-		ug/L			CASID30236
Aroclor 1242 {2}	MULTI		0.013	0.2	-		-		ug/L			CASID30237
Aroclor 1242 {3}	MULTI		0.013	0.2	-		-		ug/L			CASID30238
Aroclor 1242 {4}	MULTI		0.013	0.2	-		-		ug/L			CASID30239
Aroclor 1242 {5}	MULTI		0.013	0.2	-		-		ug/L			CASID30240
Aroclor 1248	TRG		0.0054	0.2	-		-		ug/L			12672-29-6
Aroclor 1248 {1}	MULTI		0.0054	0.2	-		-		ug/L			CASID30241
Aroclor 1248 {2}	MULTI		0.0054	0.2	-		-		ug/L			CASID30242

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Method: 8082
Prep: EPA 3535
Matrix: WATER
Title: PCB Aroclors in Water

Source: SW-846
Revision:
Level: LOW

Method ID: MJ579
Last User: AKamawal
Last Date: 06/18/2009 11:30

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
Aroclor 1248 {3}	MULTI		0.0054	0.2	-		-		ug/L			CASID30243
Aroclor 1248 {4}	MULTI		0.0054	0.2	-		-		ug/L			CASID30244
Aroclor 1248 {5}	MULTI		0.0054	0.2	-		-		ug/L			CASID30245
Aroclor 1254	TRG		0.0070	0.2	-		-		ug/L			11097-69-1
Aroclor 1254 {1}	MULTI		0.0070	0.2	-		-		ug/L			CASID30246
Aroclor 1254 {2}	MULTI		0.0070	0.2	-		-		ug/L			CASID30247
Aroclor 1254 {3}	MULTI		0.0070	0.2	-		-		ug/L			CASID30248
Aroclor 1254 {4}	MULTI		0.0070	0.2	-		-		ug/L			CASID30249
Aroclor 1254 {5}	MULTI		0.0070	0.2	-		-		ug/L			CASID30250
Aroclor 1260	MS		0.0034	0.2	29-123	30	36-115	30	ug/L	1000		11096-82-5
Aroclor 1260 {1}	MULTI		0.0034	0.2	29-123	30	36-115	30	ug/L	1000		CASID30251
Aroclor 1260 {2}	MULTI		0.0034	0.2	29-123	30	36-115	30	ug/L	1000		CASID30252
Aroclor 1260 {3}	MULTI		0.0034	0.2	29-123	30	36-115	30	ug/L	1000		CASID30253
Aroclor 1260 {4}	MULTI		0.0034	0.2	29-123	30	36-115	30	ug/L	1000		CASID30254
Aroclor 1260 {5}	MULTI		0.0034	0.2	29-123	30	36-115	30	ug/L	1000		CASID30255
Aroclor 1262	TRG		0.0048	0.2	-		-		ug/L			37324-23-5
Aroclor 1262 {1}	MULTI		0.0048	0.2	-		-		ug/L			CASID30256
Aroclor 1262 {2}	MULTI		0.0048	0.2	-		-		ug/L			CASID30257
Aroclor 1262 {3}	MULTI		0.0048	0.2	-		-		ug/L			CASID30258
Aroclor 1262 {4}	MULTI		0.0048	0.2	-		-		ug/L			CASID30259
Aroclor 1262 {5}	MULTI		0.0048	0.2	-		-		ug/L			CASID30260
Aroclor 1268	TRG		0.0065	0.2	-		-		ug/L			11100-14-4
Aroclor 1268 {1}	MULTI		0.0065	0.2	-		-		ug/L			CASID30261
Aroclor 1268 {2}	MULTI		0.0065	0.2	-		-		ug/L			CASID30262
Aroclor 1268 {3}	MULTI		0.0065	0.2	-		-		ug/L			CASID30263
Aroclor 1268 {4}	MULTI		0.0065	0.2	-		-		ug/L			CASID30264
Aroclor 1268 {5}	MULTI		0.0065	0.2	-		-		ug/L			CASID30265
Decachlorobiphenyl	SURR				17-140		-		PERCE	100		2051-24-3
Tetrachloro-m-xylene	SURR				10-109		-		PERCE	100		877-09-8

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Method Information

Method: 8082
Prep: EPA 3535
Matrix: WATER
Title: PCB-LL Aroclors in Water

Source: SW-846
Revision:
Level: LOW

Method ID: MJ706
Last User: AKamawal
Last Date: 02/24/2009 14:44

Optimal Prep

Sample Amount:	1,000.00 m	0.00
Final Volume:	2.00 ml	0.00

Hold Time

Analytical :	40 days from date Prep
Preparation :	7 days from date Sampled

Require Confirmation?	YES
AFCEE ?	NO

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
2,2',4,4',5,5'-Hexabromobiphenyl	SURR				70-130		-		PERCE	5		59080-40-9
Aroclor 1016	MS		0.0041	0.02	10-161	30	32-115	30	ug/L	100		12674-11-2
Aroclor 1016 {1}	MULTI		0.0041	0.02	10-161	30	32-115	30	ug/L	100		CASID30221
Aroclor 1016 {2}	MULTI		0.0041	0.02	10-161	30	32-115	30	ug/L	100		CASID30222
Aroclor 1016 {3}	MULTI		0.0041	0.02	10-161	30	32-115	30	ug/L	100		CASID30223
Aroclor 1016 {4}	MULTI		0.0041	0.02	10-161	30	32-115	30	ug/L	100		CASID30224
Aroclor 1016 {5}	MULTI		0.0041	0.02	10-161	30	32-115	30	ug/L	100		CASID30225
Aroclor 1221	TRG		0.0041	0.04	-		-		ug/L			11104-28-2
Aroclor 1221 {1}	MULTI		0.0041	0.04	-		-		ug/L			CASID30226
Aroclor 1221 {2}	MULTI		0.0041	0.04	-		-		ug/L			CASID30227
Aroclor 1221 {3}	MULTI		0.0041	0.04	-		-		ug/L			CASID30228
Aroclor 1221 {4}	MULTI		0.0041	0.04	-		-		ug/L			CASID30229
Aroclor 1221 {5}	MULTI		0.0041	0.04	-		-		ug/L			CASID30230
Aroclor 1232	TRG		0.0041	0.02	-		-		ug/L			11141-16-5
Aroclor 1232 {1}	MULTI		0.0041	0.02	-		-		ug/L			CASID30231
Aroclor 1232 {2}	MULTI		0.0041	0.02	-		-		ug/L			CASID30232
Aroclor 1232 {3}	MULTI		0.0041	0.02	-		-		ug/L			CASID30233
Aroclor 1232 {4}	MULTI		0.0041	0.02	-		-		ug/L			CASID30234
Aroclor 1232 {5}	MULTI		0.0041	0.02	-		-		ug/L			CASID30235
Aroclor 1242	TRG		0.0041	0.02	-		-		ug/L			53469-21-9
Aroclor 1242 {1}	MULTI		0.0041	0.02	-		-		ug/L			CASID30236
Aroclor 1242 {2}	MULTI		0.0041	0.02	-		-		ug/L			CASID30237
Aroclor 1242 {3}	MULTI		0.0041	0.02	-		-		ug/L			CASID30238
Aroclor 1242 {4}	MULTI		0.0041	0.02	-		-		ug/L			CASID30239
Aroclor 1242 {5}	MULTI		0.0041	0.02	-		-		ug/L			CASID30240
Aroclor 1248	TRG		0.0041	0.02	-		-		ug/L			12672-29-6
Aroclor 1248 {1}	MULTI		0.0041	0.02	-		-		ug/L			CASID30241
Aroclor 1248 {2}	MULTI		0.0041	0.02	-		-		ug/L			CASID30242

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Method: 8082
Prep: EPA 3535
Matrix: WATER
Title: PCB-LL Aroclors in Water

Source: SW-846
Revision:
Level: LOW

Method ID: MJ706
Last User: AKamawal
Last Date: 02/24/2009 14:44

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
Aroclor 1248 {3}	MULTI		0.0041	0.02	-		-		ug/L			CASID30243
Aroclor 1248 {4}	MULTI		0.0041	0.02	-		-		ug/L			CASID30244
Aroclor 1248 {5}	MULTI		0.0041	0.02	-		-		ug/L			CASID30245
Aroclor 1254	TRG		0.0041	0.02	-		-		ug/L			11097-69-1
Aroclor 1254 {1}	MULTI		0.0041	0.02	-		-		ug/L			CASID30246
Aroclor 1254 {2}	MULTI		0.0041	0.02	-		-		ug/L			CASID30247
Aroclor 1254 {3}	MULTI		0.0041	0.02	-		-		ug/L			CASID30248
Aroclor 1254 {4}	MULTI		0.0041	0.02	-		-		ug/L			CASID30249
Aroclor 1254 {5}	MULTI		0.0041	0.02	-		-		ug/L			CASID30250
Aroclor 1260	MS		0.0041	0.02	29-123	30	36-115	30	ug/L	100		11096-82-5
Aroclor 1260 {1}	MULTI		0.0041	0.02	29-123	30	36-115	30	ug/L	100		CASID30251
Aroclor 1260 {2}	MULTI		0.0041	0.02	29-123	30	36-115	30	ug/L	100		CASID30252
Aroclor 1260 {3}	MULTI		0.0041	0.02	29-123	30	36-115	30	ug/L	100		CASID30253
Aroclor 1260 {4}	MULTI		0.0041	0.02	29-123	30	36-115	30	ug/L	100		CASID30254
Aroclor 1260 {5}	MULTI		0.0041	0.02	29-123	30	36-115	30	ug/L	100		CASID30255
Aroclor 1262	TRG		0.0041	0.02	-		-		ug/L			37324-23-5
Aroclor 1262 {1}	MULTI		0.0041	0.02	-		-		ug/L			CASID30256
Aroclor 1262 {2}	MULTI		0.0041	0.02	-		-		ug/L			CASID30257
Aroclor 1262 {3}	MULTI		0.0041	0.02	-		-		ug/L			CASID30258
Aroclor 1262 {4}	MULTI		0.0041	0.02	-		-		ug/L			CASID30259
Aroclor 1262 {5}	MULTI		0.0041	0.02	-		-		ug/L			CASID30260
Aroclor 1268	TRG		0.0041	0.02	-		-		ug/L			11100-14-4
Aroclor 1268 {1}	MULTI		0.0041	0.02	-		-		ug/L			CASID30261
Aroclor 1268 {2}	MULTI		0.0041	0.02	-		-		ug/L			CASID30262
Aroclor 1268 {3}	MULTI		0.0041	0.02	-		-		ug/L			CASID30263
Aroclor 1268 {4}	MULTI		0.0041	0.02	-		-		ug/L			CASID30264
Aroclor 1268 {5}	MULTI		0.0041	0.02	-		-		ug/L			CASID30265
Decachlorobiphenyl	SURR				17-140		-		PERCE	5		2051-24-3
PCBs, Total	MS				-		-		ug/L	200		1336-36-3
Tetrachloro-m-xylene	SURR				10-109		-		PERCE	5		877-09-8

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Method Information

Method: 8082
Prep: EPA 3535
Matrix: WATER
Title: PCB Aroclors in UL-Water

Source: SW-846
Revision:
Level: LOW

Method ID: MJ702
Last User: AKamawal
Last Date: 02/24/2009 14:44

Optimal Prep

Sample Amount:	1,000.00 m	0.00
Final Volume:	2.00 ml	0.00

Hold Time

Analytical :	40 days from date Prep
Preparation :	7 days from date Sampled

Require Confirmation?	YES
AFCEE ?	NO

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
2,2',4,4',5,5'-Hexabromobiphenyl	SURR				70-130		-		PERCE	5		59080-40-9
Aroclor 1016	MS		0.0041	0.005	10-161	30	32-115	30	ug/L	100		12674-11-2
Aroclor 1016 {1}	MULTI		0.0041	0.005	10-161	30	32-115	30	ug/L	100		CASID30221
Aroclor 1016 {2}	MULTI		0.0041	0.005	10-161	30	32-115	30	ug/L	100		CASID30222
Aroclor 1016 {3}	MULTI		0.0041	0.005	10-161	30	32-115	30	ug/L	100		CASID30223
Aroclor 1016 {4}	MULTI		0.0041	0.005	10-161	30	32-115	30	ug/L	100		CASID30224
Aroclor 1016 {5}	MULTI		0.0041	0.005	10-161	30	32-115	30	ug/L	100		CASID30225
Aroclor 1221	TRG		0.0041	0.010	-	30	-	30	ug/L			11104-28-2
Aroclor 1221 {1}	MULTI		0.0041	0.010	-	30	-	30	ug/L			CASID30226
Aroclor 1221 {2}	MULTI		0.0041	0.010	-	30	-	30	ug/L			CASID30227
Aroclor 1221 {3}	MULTI		0.0041	0.010	-	30	-	30	ug/L			CASID30228
Aroclor 1221 {4}	MULTI		0.0041	0.010	-	30	-	30	ug/L			CASID30229
Aroclor 1221 {5}	MULTI		0.0041	0.010	-	30	-	30	ug/L			CASID30230
Aroclor 1232	TRG		0.0041	0.005	-	30	-	30	ug/L			11141-16-5
Aroclor 1232 {1}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30231
Aroclor 1232 {2}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30232
Aroclor 1232 {3}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30233
Aroclor 1232 {4}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30234
Aroclor 1232 {5}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30235
Aroclor 1242	TRG		0.0041	0.005	-	30	-	30	ug/L			53469-21-9
Aroclor 1242 {1}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30236
Aroclor 1242 {2}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30237
Aroclor 1242 {3}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30238
Aroclor 1242 {4}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30239
Aroclor 1242 {5}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30240
Aroclor 1248	TRG		0.0041	0.005	-	30	-	30	ug/L			12672-29-6
Aroclor 1248 {1}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30241
Aroclor 1248 {2}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30242

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Method: 8082
Prep: EPA 3535
Matrix: WATER
Title: PCB Aroclors in UL-Water

Source: SW-846
Revision:
Level: LOW

Method ID: MJ702
Last User: AKamawal
Last Date: 02/24/2009 14:44

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
Aroclor 1248 {3}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30243
Aroclor 1248 {4}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30244
Aroclor 1248 {5}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30245
Aroclor 1254	TRG		0.0041	0.005	-	30	-	30	ug/L			11097-69-1
Aroclor 1254 {1}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30246
Aroclor 1254 {2}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30247
Aroclor 1254 {3}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30248
Aroclor 1254 {4}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30249
Aroclor 1254 {5}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30250
Aroclor 1260	MS		0.0041	0.005	29-123	30	36-115	30	ug/L	100		11096-82-5
Aroclor 1260 {1}	MULTI		0.0041	0.005	29-123	30	36-115	30	ug/L	100		CASID30251
Aroclor 1260 {2}	MULTI		0.0041	0.005	29-123	30	36-115	30	ug/L	100		CASID30252
Aroclor 1260 {3}	MULTI		0.0041	0.005	29-123	30	36-115	30	ug/L	100		CASID30253
Aroclor 1260 {4}	MULTI		0.0041	0.005	29-123	30	36-115	30	ug/L	100		CASID30254
Aroclor 1260 {5}	MULTI		0.0041	0.005	29-123	30	36-115	30	ug/L	100		CASID30255
Aroclor 1262	TRG		0.0041	0.005	-	30	-	30	ug/L			37324-23-5
Aroclor 1262 {1}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30256
Aroclor 1262 {2}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30257
Aroclor 1262 {3}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30258
Aroclor 1262 {4}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30259
Aroclor 1262 {5}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30260
Aroclor 1268	TRG		0.0041	0.005	-	30	-	30	ug/L			11100-14-4
Aroclor 1268 {1}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30261
Aroclor 1268 {2}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30262
Aroclor 1268 {3}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30263
Aroclor 1268 {4}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30264
Aroclor 1268 {5}	MULTI		0.0041	0.005	-	30	-	30	ug/L			CASID30265
Decachlorobiphenyl	SURR				17-140		-		PERCE	5		2051-24-3
PCBs, Total	MS				-		-		ug/L			1336-36-3
Tetrachloro-m-xylene	SURR				10-109		-		PERCE	5		877-09-8

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Method Information

Method: 8082
Prep: EPA 3541
Matrix: SOIL
Title: Reg level 3541 PCB Aroclors in Soil

Source: SW-846
Revision:
Level: LOW

Method ID: MJ731
Last User: AKamawal
Last Date: 05/05/2009 15:00

Optimal Prep

Sample Amount:	10.00 g	0.00
Final Volume:	10.00 ml	0.00

Hold Time

Analytical :	40 days from date Prep
Preparation :	14 days from date Sampled

Require Confirmation?	YES
AFCEE ?	NO

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
2,2',4,4',5,5'-Hexabromobiphenyl	SURR				70-130		-			100		59080-40-9
Aroclor 1016	MS		0.019	0.10	24-168	40	44-130	40	mg/Kg	1000		12674-11-2
Aroclor 1016 {1}	MULTI		0.019	0.10	24-168	40	44-130	40	mg/Kg	1000		CASID30221
Aroclor 1016 {2}	MULTI		0.019	0.10	24-168	40	44-130	40	mg/Kg	1000		CASID30222
Aroclor 1016 {3}	MULTI		0.019	0.10	24-168	40	44-130	40	mg/Kg	1000		CASID30223
Aroclor 1016 {4}	MULTI		0.019	0.10	24-168	40	44-130	40	mg/Kg	1000		CASID30224
Aroclor 1016 {5}	MULTI		0.019	0.10	24-168	40	44-130	40	mg/Kg	1000		CASID30225
Aroclor 1221	TRG		0.012	0.20	-	40	-	40	mg/Kg			11104-28-2
Aroclor 1221 {1}	MULTI		0.012	0.20	-	40	-	40	mg/Kg			CASID30226
Aroclor 1221 {2}	MULTI		0.012	0.20	-	40	-	40	mg/Kg			CASID30227
Aroclor 1221 {3}	MULTI		0.012	0.20	-	40	-	40	mg/Kg			CASID30228
Aroclor 1221 {4}	MULTI		0.012	0.20	-	40	-	40	mg/Kg			CASID30229
Aroclor 1221 {5}	MULTI		0.012	0.20	-	40	-	40	mg/Kg			CASID30230
Aroclor 1232	TRG		0.024	0.10	-	40	-	40	mg/Kg			11141-16-5
Aroclor 1232 {1}	MULTI		0.024	0.10	-	40	-	40	mg/Kg			CASID30231
Aroclor 1232 {2}	MULTI		0.024	0.10	-	40	-	40	mg/Kg			CASID30232
Aroclor 1232 {3}	MULTI		0.024	0.10	-	40	-	40	mg/Kg			CASID30233
Aroclor 1232 {4}	MULTI		0.024	0.10	-	40	-	40	mg/Kg			CASID30234
Aroclor 1232 {5}	MULTI		0.024	0.10	-	40	-	40	mg/Kg			CASID30235
Aroclor 1242	TRG		0.012	0.10	-	40	-	40	mg/Kg			53469-21-9
Aroclor 1242 {1}	MULTI		0.012	0.10	-	40	-	40	mg/Kg			CASID30236
Aroclor 1242 {2}	MULTI		0.012	0.10	-	40	-	40	mg/Kg			CASID30237
Aroclor 1242 {3}	MULTI		0.012	0.10	-	40	-	40	mg/Kg			CASID30238
Aroclor 1242 {4}	MULTI		0.012	0.10	-	40	-	40	mg/Kg			CASID30239
Aroclor 1242 {5}	MULTI		0.012	0.10	-	40	-	40	mg/Kg			CASID30240
Aroclor 1248	TRG		0.0072	0.10	-	40	-	40	mg/Kg			12672-29-6
Aroclor 1248 {1}	MULTI		0.0072	0.10	-	40	-	40	mg/Kg			CASID30241
Aroclor 1248 {2}	MULTI		0.0072	0.10	-	40	-	40	mg/Kg			CASID30242

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Method: 8082
Prep: EPA 3541
Matrix: SOIL
Title: Reg level 3541 PCB Aroclors in Soil

Source: SW-846
Revision:
Level: LOW

Method ID: MJ731
Last User: AKamawal
Last Date: 05/05/2009 15:00

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
Aroclor 1248 {3}	MULTI		0.0072	0.10	-	40	-	40	mg/Kg			CASID30243
Aroclor 1248 {4}	MULTI		0.0072	0.10	-	40	-	40	mg/Kg			CASID30244
Aroclor 1248 {5}	MULTI		0.0072	0.10	-	40	-	40	mg/Kg			CASID30245
Aroclor 1254	TRG		0.0088	0.10	-	40	-	40	mg/Kg			11097-69-1
Aroclor 1254 {1}	MULTI		0.0088	0.10	-	40	-	40	mg/Kg			CASID30246
Aroclor 1254 {2}	MULTI		0.0088	0.10	-	40	-	40	mg/Kg			CASID30247
Aroclor 1254 {3}	MULTI		0.0088	0.10	-	40	-	40	mg/Kg			CASID30248
Aroclor 1254 {4}	MULTI		0.0088	0.10	-	40	-	40	mg/Kg			CASID30249
Aroclor 1254 {5}	MULTI		0.0088	0.10	-	40	-	40	mg/Kg			CASID30250
Aroclor 1260	MS		0.013	0.10	24-163	40	52-127	40	mg/Kg	1000		11096-82-5
Aroclor 1260 {1}	MULTI		0.013	0.10	24-163	40	52-127	40	mg/Kg	1000		CASID30251
Aroclor 1260 {2}	MULTI		0.013	0.10	24-163	40	52-127	40	mg/Kg	1000		CASID30252
Aroclor 1260 {3}	MULTI		0.013	0.10	24-163	40	52-127	40	mg/Kg	1000		CASID30253
Aroclor 1260 {4}	MULTI		0.013	0.10	24-163	40	52-127	40	mg/Kg	1000		CASID30254
Aroclor 1260 {5}	MULTI		0.013	0.10	24-163	40	52-127	40	mg/Kg	1000		CASID30255
Aroclor 1262	TRG		0.020	0.10	-	40	-	40	mg/Kg			37324-23-5
Aroclor 1262 {1}	MULTI		0.020	0.10	-	40	-	40	mg/Kg			CASID30256
Aroclor 1262 {2}	MULTI		0.020	0.10	-	40	-	40	mg/Kg			CASID30257
Aroclor 1262 {3}	MULTI		0.020	0.10	-	40	-	40	mg/Kg			CASID30258
Aroclor 1262 {4}	MULTI		0.020	0.10	-	40	-	40	mg/Kg			CASID30259
Aroclor 1262 {5}	MULTI		0.020	0.10	-	40	-	40	mg/Kg			CASID30260
Aroclor 1268	TRG		0.0066	0.10	-	40	-	40	mg/Kg			11100-14-4
Aroclor 1268 {1}	MULTI		0.0066	0.10	-	40	-	40	mg/Kg			CASID30261
Aroclor 1268 {2}	MULTI		0.0066	0.10	-	40	-	40	mg/Kg			CASID30262
Aroclor 1268 {3}	MULTI		0.0066	0.10	-	40	-	40	mg/Kg			CASID30263
Aroclor 1268 {4}	MULTI		0.0066	0.10	-	40	-	40	mg/Kg			CASID30264
Aroclor 1268 {5}	MULTI		0.0066	0.10	-	40	-	40	mg/Kg			CASID30265
Decachlorobiphenyl	SURR				38-144		-		PERCE	100		2051-24-3
Tetrachloro-m-xylene	SURR				22-130		-		PERCE	100		877-09-8

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Method Information

Method: 8082
Prep: EPA 3541
Matrix: SOIL
Title: PCB Aroclors in LL-Sediment/Soil

Source: SW-846
Revision:
Level: LOW

Method ID: MJ696
Last User: AKamawal
Last Date: 04/03/2008 17:42

Optimal Prep

Sample Amount:	20.00 g	0.00
Final Volume:	4.00 ml	0.00

Hold Time

Analytical :	40 days from date Prep
Preparation :	14 days from date Sampled

Require Confirmation?	YES
AFCEE ?	NO

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
2,2',4,4',6,6'-Hexabromobiphenyl	SURR				70-130		-		PERCE	100		59261-08-4
Aroclor 1016	MS		2.1	10	24-168	40	44-130	40	ug/Kg	1000		12674-11-2
Aroclor 1016 {1}	MULTI		2.1	10	24-168	40	44-130	40	ug/Kg	1000		CASID30221
Aroclor 1016 {2}	MULTI		2.1	10	24-168	40	44-130	40	ug/Kg	1000		CASID30222
Aroclor 1016 {3}	MULTI		2.1	10	24-168	40	44-130	40	ug/Kg	1000		CASID30223
Aroclor 1016 {4}	MULTI		2.1	10	24-168	40	44-130	40	ug/Kg	1000		CASID30224
Aroclor 1016 {5}	MULTI		2.1	10	24-168	40	44-130	40	ug/Kg	1000		CASID30225
Aroclor 1221	TRG		2.1	20	-	40	-		ug/Kg			11104-28-2
Aroclor 1221 {1}	MULTI		2.1	20	-	40	-		ug/Kg			CASID30226
Aroclor 1221 {2}	MULTI		2.1	20	-	40	-		ug/Kg			CASID30227
Aroclor 1221 {3}	MULTI		2.1	20	-	40	-		ug/Kg			CASID30228
Aroclor 1221 {4}	MULTI		2.1	20	-	40	-		ug/Kg			CASID30229
Aroclor 1221 {5}	MULTI		2.1	20	-	40	-		ug/Kg			CASID30230
Aroclor 1232	TRG		2.1	10	-	40	-		ug/Kg			11141-16-5
Aroclor 1232 {1}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30231
Aroclor 1232 {2}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30232
Aroclor 1232 {3}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30233
Aroclor 1232 {4}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30234
Aroclor 1232 {5}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30235
Aroclor 1242	TRG		2.1	10	-	40	-		ug/Kg			53469-21-9
Aroclor 1242 {1}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30236
Aroclor 1242 {2}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30237
Aroclor 1242 {3}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30238
Aroclor 1242 {4}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30239
Aroclor 1242 {5}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30240
Aroclor 1248	TRG		2.1	10	-	40	-		ug/Kg			12672-29-6
Aroclor 1248 {1}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30241
Aroclor 1248 {2}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30242

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Method: 8082
Prep: EPA 3541
Matrix: SOIL
Title: PCB Aroclors in LL-Sediment/Soil

Source: SW-846
Revision:
Level: LOW

Method ID: MJ696
Last User: AKamawal
Last Date: 04/03/2008 17:42

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
Aroclor 1248 {3}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30243
Aroclor 1248 {4}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30244
Aroclor 1248 {5}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30245
Aroclor 1254	TRG		2.1	10	-	40	-		ug/Kg			11097-69-1
Aroclor 1254 {1}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30246
Aroclor 1254 {2}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30247
Aroclor 1254 {3}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30248
Aroclor 1254 {4}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30249
Aroclor 1254 {5}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30250
Aroclor 1260	MS		2.1	10	24-163	40	52-127	40	ug/Kg	1000		11096-82-5
Aroclor 1260 {1}	MULTI		2.1	10	24-163	40	52-127	40	ug/Kg	1000		CASID30251
Aroclor 1260 {2}	MULTI		2.1	10	24-163	40	52-127	40	ug/Kg	1000		CASID30252
Aroclor 1260 {3}	MULTI		2.1	10	24-163	40	52-127	40	ug/Kg	1000		CASID30253
Aroclor 1260 {4}	MULTI		2.1	10	24-163	40	52-127	40	ug/Kg	1000		CASID30254
Aroclor 1260 {5}	MULTI		2.1	10	24-163	40	52-127	40	ug/Kg	1000		CASID30255
Aroclor 1262	TRG		2.1	10	-	40	-		ug/Kg			37324-23-5
Aroclor 1262 {1}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30256
Aroclor 1262 {2}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30257
Aroclor 1262 {3}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30258
Aroclor 1262 {4}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30259
Aroclor 1262 {5}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30260
Aroclor 1268	TRG		2.1	10	-	40	-		ug/Kg			11100-14-4
Aroclor 1268 {1}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30261
Aroclor 1268 {2}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30262
Aroclor 1268 {3}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30263
Aroclor 1268 {4}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30264
Aroclor 1268 {5}	MULTI		2.1	10	-	40	-		ug/Kg			CASID30265
Decachlorobiphenyl	SURR				38-144		-		PERCE	100		2051-24-3
PCBs, Total	MS				-		-		ug/Kg	2000		1336-36-3
Tetrachloro-m-xylene	SURR				22-130		-		PERCE	100		877-09-8

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Method Information

Method: 8082
Prep: EPA 3541
Matrix: TISSUE
Title: EPS PCB Aroclors in Tissue

Source: SW-846
Revision:
Level: LOW

Method ID: MJ923
Last User: AKamawal
Last Date: 04/03/2008 17:51

Optimal Prep

Sample Amount:	10.00 g	0.00
Final Volume:	2.00 ml	0.00

Hold Time

Analytical :	40 days from date Prep
Preparation :	365 days from date Sample

Require Confirmation?	YES
AFCEE ?	NO

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
2,2',4,4',5,5'-Hexabromobiphenyl	SURR				70-130		-		PERCE	100		59080-40-9
2,2',4,4',6,6'-Hexabromobiphenyl	SURR				70-130		-		PERCE	100		59261-08-4
Aroclor 1016	MS		1.7	10	10-165	40	42-128	40	ug/Kg	1000		12674-11-2
Aroclor 1016 {1}	MULTI		1.7	10	10-165	40	42-128	40	ug/Kg	1000		CASID30221
Aroclor 1016 {2}	MULTI		1.7	10	10-165	40	42-128	40	ug/Kg	1000		CASID30222
Aroclor 1016 {3}	MULTI		1.7	10	10-165	40	42-128	40	ug/Kg	1000		CASID30223
Aroclor 1016 {4}	MULTI		1.7	10	10-165	40	42-128	40	ug/Kg	1000		CASID30224
Aroclor 1016 {5}	MULTI		1.7	10	10-165	40	42-128	40	ug/Kg	1000		CASID30225
Aroclor 1221	TRG		1.7	20	-	40	-	40	ug/Kg			11104-28-2
Aroclor 1221 {1}	MULTI		1.7	20	-	40	-	40	ug/Kg		CASID30	CASID30226
Aroclor 1221 {2}	MULTI		1.7	20	-	40	-	40	ug/Kg		CASID30	CASID30227
Aroclor 1221 {3}	MULTI		1.7	20	-	40	-	40	ug/Kg		CASID30	CASID30228
Aroclor 1221 {4}	MULTI		1.7	20	-	40	-	40	ug/Kg		CASID30	CASID30229
Aroclor 1221 {5}	MULTI		1.7	20	-	40	-	40	ug/Kg		CASID30	CASID30230
Aroclor 1232	TRG		1.7	10	-	40	-	40	ug/Kg			11141-16-5
Aroclor 1232 {1}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30231
Aroclor 1232 {2}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30232
Aroclor 1232 {3}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30233
Aroclor 1232 {4}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30234
Aroclor 1232 {5}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30235
Aroclor 1242	TRG		1.7	10	-	40	-	40	ug/Kg			53469-21-9
Aroclor 1242 {1}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30236
Aroclor 1242 {2}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30237
Aroclor 1242 {3}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30238
Aroclor 1242 {4}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30239
Aroclor 1242 {5}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30240
Aroclor 1248	TRG		1.7	10	-	40	-	40	ug/Kg			12672-29-6
Aroclor 1248 {1}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30241

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Method: 8082
Prep: EPA 3541
Matrix: TISSUE
Title: EPS PCB Aroclors in Tissue

Source: SW-846
Revision:
Level: LOW

Method ID: MJ923
Last User: AKamawal
Last Date: 04/03/2008 17:51

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Spike Level	Reg Limit	Analyte ID
Aroclor 1248 {2}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30242
Aroclor 1248 {3}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30243
Aroclor 1248 {4}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30244
Aroclor 1248 {5}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30245
Aroclor 1254	TRG		1.7	10	-	40	-	40	ug/Kg			11097-69-1
Aroclor 1254 {1}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30246
Aroclor 1254 {2}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30247
Aroclor 1254 {3}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30248
Aroclor 1254 {4}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30249
Aroclor 1254 {5}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30250
Aroclor 1260	MS		1.7	10	10-185	40	46-131	40	ug/Kg	1000		11096-82-5
Aroclor 1260 {1}	MULTI		1.7	10	10-185	40	46-131	40	ug/Kg	1000		CASID30251
Aroclor 1260 {2}	MULTI		1.7	10	10-185	40	46-131	40	ug/Kg	1000		CASID30252
Aroclor 1260 {3}	MULTI		1.7	10	10-185	40	46-131	40	ug/Kg	1000		CASID30253
Aroclor 1260 {4}	MULTI		1.7	10	10-185	40	46-131	40	ug/Kg	1000		CASID30254
Aroclor 1260 {5}	MULTI		1.7	10	10-185	40	46-131	40	ug/Kg	1000		CASID30255
Aroclor 1262	TRG		1.7	10	-	40	-	40	ug/Kg			37324-23-5
Aroclor 1262 {1}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30256
Aroclor 1262 {2}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30257
Aroclor 1262 {3}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30258
Aroclor 1262 {4}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30259
Aroclor 1262 {5}	MULTI		1.7	10	-	40	-	40	ug/Kg		CASID30	CASID30260
Aroclor 1268	MS		1.7	10	70-130	40	70-130	40	ug/Kg	1000		11100-14-4
Aroclor 1268 {1}	MULTI		1.7	10	-	40	-	40	ug/Kg	1000	CASID30	CASID30261
Aroclor 1268 {2}	MULTI		1.7	10	-	40	-	40	ug/Kg	1000	CASID30	CASID30262
Aroclor 1268 {3}	MULTI		1.7	10	-	40	-	40	ug/Kg	1000	CASID30	CASID30263
Aroclor 1268 {4}	MULTI		1.7	10	-	40	-	40	ug/Kg	1000	CASID30	CASID30264
Aroclor 1268 {5}	MULTI		1.7	10	-	40	-	40	ug/Kg	1000	CASID30	CASID30265
Decachlorobiphenyl	SURR				10-202		-		PERCE	100		2051-24-3
Tetrachloro-m-xylene	SURR				17-118		-		PERCE	100		877-09-8

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STANDARD OPERATING PROCEDURE

SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS

EPA Method 8270C

SOC-8270C
Revision 12
June 26, 2009

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Approved By: _____ Supervisor 7/1/09 Date

_____ QA Manager 7/9/09 Date

_____ Laboratory Manager 7/9/09 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

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Annual review of this SOP has been performed and the SOP still reflects current practice.
Initials: _____ Date: _____
Initials: _____ Date: _____
Initials: _____ Date: _____

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS
Method 8270C

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine the concentrations of Semi-Volatile Organic Compounds in water and soil using EPA Method 8270C. This procedure may also be applicable to various miscellaneous waste samples. Tables 1 and 1A indicate compounds that may be determined by this method and lists their method reporting limits (MRLs) in water and soil. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. The reported MRL may be adjusted if required for specific project requirements; however, the capability of achieving other reported MRLs must be demonstated. The Method Detection Limits (MDLs) will vary depending on the instrument used and preparation method and may also change slightly as MDL studies are updated.

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1.2. This procedure can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone phase. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols. Other compounds than those listed in Tables 1 and 1A may be analyzed. Refer to Section 1 of method 8270C.

2. METHOD SUMMARY

2.1. This method provides Gas Chromatography/Mass Spectrometry (GC/MS) conditions for the detection of Semi-volatile Organic Compounds. Prior to the use of this method, an appropriate sample preparation method must be used to recover the analytes of interest. A 1.0 µL aliquot of the extract is injected into the gas chromatograph (GC). The compounds are separated on a fused silica capillary column. Compounds of interest are detected by a mass selective detector. Identification of the analytes of interest is performed by comparing the retention times of the analytes with the respective retention times of an authentic standard, and by comparing mass spectra of analytes with mass spectra of reference materials. Quantitative analysis is performed by using the authentic standard to produce a response factor and calibration curve, and using the calibration data to determine the concentration of an analyte in the extract. The concentration in the sample is calculated using the sample weight or volume and the extract volume.

2.2. The following compounds may require special treatment when being determined by this method. Benzidine can be subject to oxidative losses during solvent concentration and the

chromatography for this compound is poor. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, to a chemical reaction in acetone, and can undergo photochemical decomposition. N-nitroso-dimethylamine is difficult to separate from the solvent under the chromatographic conditions described. N-nitroso-diphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. Pentachlorophenol, 2,4-dinitrophenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

3. DEFINITIONS

3.1. **Analysis Sequence** - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with injection of Decafluorotriphenylphosphine (DFTPP) followed by initial calibration standard(s). Once calibrated, a CCV is evaluated and extracts can be run. The sequence ends after 12 hours based on the DFTPP injection time.

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3.2. **Matrix Spike/Duplicate Matrix Spike Analysis** - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample extraction and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. Samples are split into duplicates, then spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

COPY

3.3. **Standard Curve** - A standard curve is a calibration curve which plots concentrations of a known analyte standard versus the instrument response to the analyte.

3.4. **Surrogate** - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

3.5. **Method Blank** - The method blank is an artificial sample designed to monitor introduction of artifacts into the process. The method blank is carried through the entire analytical procedure.

3.6. **Continuing Calibration Verification Standard (CCV)** - A mid-level standard injected into the instrument at specified intervals and is used to verify the validity of the initial calibration.

3.7. **Independent Calibration Verification Standard (ICV)** - A mid-level standard injected into the instrument after the calibration curve from a different source than the standards in the curve and is used to verify the validity of the initial calibration.

4. INTERFERENCES

4.1. Raw 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. Corrective action should be taken to eliminate the interferences.

4.2. Accurate determination of phthalate esters can pose difficulties when using this methodology. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware may occur when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalates can best be minimized by avoiding contact with any plastic materials. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

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4.3. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

5. SAFETY

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5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personal protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the CAS safety policies, approved methods and in MSDSs where available. Refer to the CAS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. This method uses Methylene Chloride, a known human carcinogen. Viton brand gloves should be used while rinsing, pouring or transferring the solvent

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Containers used to collect samples should be purchased pre-cleaned. Alternatively, containers used to collect samples for the determination of semivolatile organic compounds may be soap and water washed followed by methanol (or isopropanol) rinsing. The sample containers should be of glass or Teflon and have screw-top covers with Teflon liners. In situations where Teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum

foil, causing eventual contamination of the sample. Plastic containers or lids may not be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic.

6.2. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g., if an automatic sampler is used), run reagent water through the sampler and use the rinseate as a field blank.

6.3. Water and soil samples must be iced or refrigerated at 4 ± 2°C from time of collection until extraction.

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6.4. Water samples must be extracted within 7 days and the extracts analyzed within 40 days following extraction. Soil samples must be extracted within 14 days and the extract analyzed within 40 days following extraction. Extracts are stored at -10°C.

7. APPARATUS AND MATERIALS

7.1. Gas Chromatograph/Mass Spectrometer System

7.1.1. Gas Chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

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7.1.2. Column: ZB-5MS with Guardian - 30 m x 0.25 mm ID x 0.25 µm film thickness silicone-coated fused-silica capillary column. Catalog #7HG-G010-11-GGA, or equivalent.

7.1.3. Mass Spectrometer - Capable of scanning from 35 to 500 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria in Table 2 when 1.0 µL of the GC/MS tuning standard is injected through the GC (50 ng of DFTPP).

7.1.4. GC/MS Interface - Any GC-to-MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria may be used.

7.1.5. Data System - A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic

program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

7.2. Appropriate analytical balance (0.0001 g), volumetric flasks, syringes, vials, and bottles for standards preparation.

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

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8.1. Solvents: Acetone, methylene chloride, methanol, and other appropriate solvents. Solvents must be of sufficient purity to permit usage without lessening the accuracy of the determination or introducing interferences.

8.2. Stock Standard Solutions (See Table 3)

8.2.1. Commercially prepared stock standards are typically used when available at a concentration of 1000 ug/ml or more. They must be A2LA or ISO9000 certified by the manufacturer. Standard concentrations can be verified by comparison versus an independently prepared standard. Alternatively, prepare stock standard solutions at a concentration of 1000 µg/ml by dissolving 0.0100 g of reference material in methylene chloride or other suitable solvent and diluting to volume in a 10mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard.

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8.2.2. Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at -10°C and protect from light, or store as recommended by the manufacturer. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

8.2.3. Stock standard solutions must be replaced after one year, or sooner, if comparison with check standards or samples indicates a problem.

8.3. Internal Standard Solutions (See Table 3) - The internal standards are 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12} (See Table 4 for corresponding compounds). The nominal concentration of the standard is 4000 ng/µL. Each 1 ml of sample extract undergoing analysis should be spiked with 10 µL of the internal standard solution, resulting in a concentration of 40 ng/µL of each internal standard. Store at -10°C or less when not being used. When using premixed certified solutions, store according to the manufacturer's recommendations.

8.4. GC/MS Tuning Standard (See Table 3) - A methylene chloride solution containing 50 ng/µL of decafluorotriphenylphosphine (DFTPP). The standard should also contain 50 ng/µL of benzidine, DDT, and pentachlorophenol, to verify injection port inertness and GC column performance. Store at -10°C or less when not being used, or store according to the manufacturer's recommendations.

8.5. Calibration Standards (See Table 3)

8.5.1. A minimum of five initial calibration standards should be prepared from stock solutions. One of the calibration standards should be at a concentration at or below the method reporting limit; the others should correspond to the range of concentrations found in real samples, but should not exceed the working range of the GC/MS system. At least one calibration standard must be at a concentration corresponding to a sample concentration meeting project-specific data quality objectives. Each standard should contain each analyte for detection by this method. Each 1 ml aliquot of calibration standards should be spiked with 10 µL of the internal standard solution prior to analysis. All calibration standards should be stored at -10°C or less and should be freshly prepared once a year, or sooner if check standards indicate a problem.

8.5.2. The daily calibration standard (CCV) is prepared at a nominal 50 ng/µL concentration from stock solutions. The CCV is prepared weekly and can be stored at 4°C ± 2°C, or as recommended by the manufacturer. The DFTPP standard may be combined with this standard (maintaining 50 ng/µL concentration) providing tuning verification and calibration verification can be done without interferences.

8.6. QC Standards (See Table 4)

8.6.1. Surrogates: Prepare a working solution in methanol containing 2-fluorophenol, phenol-d6, and 2,4,6-tribromophenol at 150 ng/µL and nitrobenzene-d5, 2-fluorobiphenyl, and terphenyl-d14 at 100 ng/µL. Aliquots of the solution are spiked into all extracted samples, blanks, and QC samples according to the extraction SOP used.

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8.6.2. Matrix Spike Standards: Prepare a working solution in methanol containing all target analytes to be reported ("full list spike") at 100 ng/µL. Aliquots of the solution are spiked into the selected QC aliquots according to the extraction SOP used.

Note: The spiking level of surrogate and spike may need to be adjusted according to project requirements, if dilutions are expected due to high levels of extracted components, or if a lower calibration range is used.

9. PREVENTIVE MAINTENANCE

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9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument.

9.2. Carrier gas - Inline purifiers or scrubbers should be in place for all sources of carrier gas. These are selected to remove water, oxygen, and hydrocarbons. Purifiers should be changed as recommended by the supplier.

9.3. Gas Chromatograph

9.3.1. Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column. Injection port maintenance includes changing the injection port liner, seal, washer, o-ring, septum, column ferrule, and autosampler syringe as needed. Liners and seals should be changed when recent sample analyses predict a problem with chomatographic performance. In some cases liners and seals may be cleaned and re-used.

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9.3.2. Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and "clean" (uniform, without fragmentation) by using the proper column cutting tool.

9.3.3. Over time, the column will exhibit poorer overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced. This is especially true when evident in conjunction with calibration difficulties.

9.4. Mass Spectrometer

9.4.1. Tune the MS as needed to result in consistent and acceptable performance while meeting the required ion abundance criteria given in section 11.

9.4.2. For units under service contract, certain maintenance is performed by instrument service staff, including pump oil changed, vacuuming boards, etc., as recommended by the manufacturer.

9.4.3. MS source cleaning should be performed as needed, depending on the performance of the unit. This may be done by the analyst or by instrument service staff.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

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10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the Sop for Documentation of Training, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

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11.1. Sample Preparation

11.1.1. Water samples

11.1.1.1. Water samples are prepared using continuous liquid-liquid extraction and EPA method 3520C. Refer to the CAS SOP EXT-3520. In some circumstances, such as rush samples or for TCLP leachates, samples may be prepared using separatory funnel procedures (EPA 3510C). Refer to the CAS SOP EXT-3510.

11.1.1.2. Perform the extraction on a 1000mL aliquot of sample. For TCLP leachates, use 100mL of sample.

11.1.2. Soil, sediment, and solid samples are prepared using the either automated soxhlet extraction (SOP EXT-3541) or ultrasonic extraction (SOP EXT-3550). The nominal sample size is 30g. Sample amounts may be decreased in the case of high-concentration waste samples.

11.1.3. Product samples are prepared using EPA method 3580.

11.1.4. Extracts may be screened by GC/FID (SOP SOC-SCR). Cleanup by GPC is optional.

11.1.5. Following sample preparation, sample extracts are then transferred to the extract cold storage unit. Extracts must be analyzed within 40 days of extraction.

11.2. The recommended GC/MS operating conditions are listed below. The GC conditions may be modified to accommodate specific instrument models and configurations.

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Mass range:	35-500 amu		
Scan Time:	1 sec/scan		
Initial temperature:	40°C, hold for 4 minutes		
Temperature program:	40-270°C at 10°C/min		
Final temperature:	300°C, hold until benzo[g,h,i]perylene has eluted		
Injector temperature:	250-300°C		
Detector interface temp:	300°C		
Injector:	Atas Optic 2, split method		
Final time:	32.0 min.		
Equilibration time:	0.20 min.		
Temperature ramp:	Initial temp	150°C	
	Ramp rate	8°C/sec.	
	Final temp	300°C	
Pressure profile:	Transfer pressure	10 psi	
	Transfer time	1.50 min.	
	Initial pressure	8.83 psi	
	Final pressure	38.50 psi	
Split flow:	3.0mL/min. at initial		
Sample volume:	1.0 µL		
Carrier gas:	helium at 35 cm/sec		

NOTE: The default calibration procedures are given in the following sections and, at the time of this revision, follow Method 8000B due to inconsistent implementation of 8000C across various states/agencies. The analyst should also refer to the CAS *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOC-CAL)*, where calibration procedures and policies are described. The calibration procedure(s) and options chosen must follow the CAS protocols.

NOTE: Certain state or program protocols have specific procedures for calibration. This may include all or part of Method 8000C. The analyst must ensure that the correct procedures are used. Known exceptions to 8000B are as follows:

- Arizona DHS requires the use of Method 8000C calibration procedures for projects originating in Arizona and under the AZ DHS lab licensure. Refer to the AZDHS calibration guidance document at the following online location: http://www.azdhs.gov/lab/license/tech/8000cmethod.pdf.
- The use of quadratic regression calibration is not allowed for projects (samples) originating from South Carolina and under the SC DHEC lab certification.

Note: The use of 'averaging' of %RSDs or CCV %D (a.k.a grand mean) is not allowed for projects under the DoD QSM protocols. When working on these projects all analytes must pass the %RSD criteria as stated in the method and the average %RSD cannot be used.

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11.3. Initial Calibration

11.3.1. Prior to calibration, analyze the GC/MS tuning standard using instrument conditions used for calibration. Obtain the spectrum for evaluation using one of the following options:

- Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or part of any other closely eluting peak.

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- Use one scan at the apex of the peak. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or part of any other closely eluting peak.

- Use one scan either directly preceding or following the apex of the peak. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or part of any other closely eluting peak.

- Use the average across the entire peak up to a total of 5 scans. Peak integration must be consistent with standard operating procedure. If the peak is wider than 5 scans, the tune will consist of the peak apex scan and the two scans immediately preceding and following the apex. Background subtraction is required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or part of any other closely eluting peak.

11.3.2. Evaluate the spectrum obtained for DFTPP against the tuning criteria in Table 2 (see 8270C, Section 7.3.1 for guidance). The GC/MS must meet the DFTPP ion abundance criteria prior to further analyses. To assess column performance and injection port inertness, pentachlorophenol and benzidine should be present at an acceptable level and peak tailing should not be excessive. DDT degradation should not exceed 20%. If excessive tailing, poor chromatography, or degradation of >20% is noted, the injection port may require cleaning. It may also be necessary to remove the first 15-30 cm of the GC column. If hardware tuning criteria can not be met, the source may need cleaning, filaments replaced or other maintenance.

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11.3.3. The internal standards should permit most of the components of interest in the chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Refer to Table 5 for internal standards and corresponding analytes assigned for quantitation. Use the base peak ion from the specific internal standard as the primary ion for quantitation (See Table 1 of EPA 8270C). If interferences are noted, use the next most intense ion as the quantitation ion (i.e. for 1,4-dichlorobenzene-d_4, use 152 m/z for quantitation).

11.3.4. Analyze 1.0 µL of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each compound (as indicated in Table 1 of EPA 8270C).

11.3.5. Establish the retention time window position for each analyte and surrogate using the midpoint standard of the initial calibration curve.

11.3.6. Calculate response factors (RFs) for each compound relative to one of the internal standards as follows:

$$RF = (A_x C_{is})/(A_{is} C_x)$$

where:

A_x = Area of the characteristic ion for compound being measured.
A_{is} = Area of the characteristic ion for specific internal standard.
C_{is} = Concentration of the specific internal standard (ng/µL).
C_x = Concentration of the compound being measured (ng/µL).

11.3.7. A system performance check must be performed to ensure that minimum average RFs are met before the calibration curve is used. For semivolatiles, the System Performance Check Compound (SPCCs) are: N-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol. The minimum acceptable average RF for these compounds is 0.050. The SPCCs typically have very low RFs (0.1-0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated. If they are not acceptable, perform GC maintenance (see section 9.3).

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11.3.8. The percent relative standard deviation (%RSD) should be less than 15% for each compound. However, the %RSD for each individual Calibration Check Compound (CCC) (see below) must be less than 30%. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units.

$$\%RSD = \frac{SD}{\overline{RF}} \times 100$$

where:

RSD = relative standard deviation.

$\overline{RF}$ = mean of 5 initial RFs for a compound.

SD = standard deviation of average RFs for a compound.

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$$SD = \sqrt{\sum_{i=1}^{N} \frac{(RF_i - \overline{RF})^2}{N - 1}}$$

where:

RF_i = RF for each of the 5 calibration levels

N = Number of RF values (i.e., 5)

Calibration Check Compounds (CCC):

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
(N-Nitroso)-diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

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11.3.9. If the % RSD of any CCC is 30% or greater, then the chromatographic system is too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure.

11.3.10. Linearity - If the % RSD of any compound is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

11.3.11. In those instances where the %RSD for one or more analytes exceeds 15%, the initial calibration may still be acceptable if the following conditions are met:

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- The mean of the RSD values for all analytes in the calibration is < 15%.
- The mean RSD criteria applies to all target analytes in the calibration standards, regardless of whether or not they are of interest for a specific project.
- The data user must be supplied with an initial calibration summary indicating the compounds which exceed 15% RSD and the result of the mean RSD calculation.

Note: The use of 'averaging' of %RSDs or CCV %D (a.k.a grand mean) is not allowed for projects under the DoD QSM protocols. When working on these projects all analytes must pass the %RSD criteria as stated in the method and the average %RSD cannot be used.

11.3.12. If all of the conditions in Section 11.3.9 are met, then the average response factor may be used to determine sample concentrations as described in Section 11.3.8.

11.3.13. Following initial calibration, analyze an ICV standard. The ICV solution must contain all analytes in the calibration standards. Calculate the concentration using the typical procedure used for quantitation. Calculate the percent difference (%D) from the ICV true value. Evaluate the ICV as described in the CAS Organics Calibration SOP.

Note: DoD project acceptance criteria is ±20% of true value.

11.4. Continuing Calibration

11.4.1. A calibration standard, or standards, at mid-concentration (See Table 3) containing all semivolatile analytes, DFTPP, and all required surrogates, must be analyzed every 12 hours during analysis.

11.4.2. The DFTPP must result in a mass spectrum (see 8270C, Section 7.3.1 for guidance) that meets the criteria given in Table 2. These criteria must be demonstrated during each 12 hour shift. Obtain the DFTPP spectrum as described in section 11.3.1.

Note: When analyzing samples subject to Wisconsin DNR regulations, a second CCV must be analyzed when second order (quadratic) calibrations are used. One will be analyzed at the lower end of the calibration range and one at a point where the curve can no longer be characterized as first order.

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11.4.3. The retention time may be updated for all analytes and surrogates using the CCV standard to account for minor fluctuations or after routine system maintenance.

11.4.4. System Performance Check Compounds (SPCCs): For each daily calibration, a system performance check must be made. For each SPCC compound in the daily calibration standard, a minimum response factor of 0.050 must be obtained. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. The minimum RF for semivolatile SPCCs is 0.050. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

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11.4.5. Calibration Check Compounds (CCCs): After the system performance check, CCCs listed in Section 11.3.6 are used to check the validity of the initial calibration.

Calculate the percent drift using:

$$\% \, Drift = \frac{C_t - C_m}{C_t} \, x \, 100$$

where:
C_t = Calibration Check Compound standard concentration.
C_m = Measured concentration using selected quantitation method.

If the percent drift for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. If the criterion is not met (> 20% drift) for any one CCC, corrective action must be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration must be generated. This criterion must be met before sample analysis begins. If the % RSD for non-CCC compounds exceeds 30%, the analyst must determine if the response is sufficient to attain MRL for that analyte and any hits for that analyte must be rerun for quantitation.

Note: DoD acceptance criteria is ± 20% from true value for all target compounds and surrogates in the CCV.

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11.4.6. The internal standard responses and retention times in the calibration check standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the midpoint standard of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrective action identified, as required. If the EICP area for any of the internal standards changes by a factor of two (50% to 200%) from that in the midpoint standard of the most recent initial calibration sequence, the chromatographic system must be inspected for malfunctions and corrective action identified, as appropriate. When corrective action is taken, reanalysis of samples analyzed while the system was malfunctioning is required. Update the reference spectra and retention times in the quantitation database for the instrument method or ID file. The initial calibration average RF or calibration curve is then used in the quantitation of subsequent analyses.

11.4.7. A blank (method blank, GPC blank, or solvent blank) should be analyzed after the CCV to prove the system is free of contaminants. If contaminants are found in a method blank or GPC blank, then a solvent blank should be ran to help isolate the source of contamination.

11.5. GC/MS Analysis

11.5.1. Evaluate FID screen and make proper dilution (See FID screening SOP).

11.5.2. Spike the 1 ml extract obtained from sample preparation with 10 µL of the internal standard solution just prior to analysis. Use the same operating conditions as were used for initial calibration.

11.5.3. If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution must take place. Additional internal standard must be added to the diluted extract to maintain the required 40ng/µL of each internal standard in the extracted volume. The diluted extract must be reanalyzed.

11.5.4. Store the extracts at -10°C or less, protected from light in vials equipped with unpierced Teflon lined septa. Archive extracts in freezer for 3 months after analysis in the instrument/date specific storage boxes.

12. QA/QC REQUIREMENTS

12.1. Refer to Section 8.0 of Method 8270C for general QC protocol. In addition to instrument criteria for calibration, the ability of each analyst/instrument to generate acceptable accuracy and precision must be documented prior to sample analysis (IPR study). This must be validated before analyses of samples begin, or whenever significant changes to the procedures have been made. To do this, four tap water samples are spiked with each target analyte, extracted, and analyzed. Refer to Method 8270C Section 8.3 for this requirement and acceptance criteria.

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12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates with a MDL spiking solution (at a level below the MRL) for each target analyte, extract, and analyze. The MDL studies should be done for each matrix, prep method, and instrument. Refer to the CAS SOP *for The Determination of Method Detection Limits and Limits of Detection.*

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12.2.2. Calculate the average concentration found (x) in the *sample concentration*, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study should be done annually.

12.3. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the SOP for *Sample Batches*. In general, these include:

12.3.1. Method blank - A method blank is extracted and analyzed with every batch of 20 or fewer samples to demonstrate that there are no method interferences. The method blank must demonstrate that interferences from the analytical and preparation steps minimized. No target analytes should be detected above the MRL in the method blank. For some project specific needs, exceptions may be noted and method blank results above the MRL may be reported for common lab contaminants (phthalate esters, etc.).

Note: DoD requires no analytes detected at > ½ the RL or 1/10 the regulatory limit, which ever is grater. For common laboratory contaminates there should be no detection > the RL.

12.3.2. A lab control sample (LCS) must be extracted and analyzed with every batch of 20 or fewer samples. The LCS is prepared by spiking a blank with the matrix

spike solution, and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

Acceptance criteria for lab control samples are listed in Attachment A. The accuracy of the analysis is controlled on a subset of target analytes. If the project analyte list is fewer than 20 analytes, all are considered control analytes. Analytes which are used for control analytes are listed in Table 6. For DoD projects all project target analytes are considered control analytes. If the LCS recovery for any control analyte fails acceptance limits, corrective action is required. If instrument corrective action is not applicable or ineffective, re-extraction of the associated samples is required. If any other analyte fails the advisory acceptance limits, the analyst must evaluate the impact on data quality and take any necessary corrective action, which may include re-extraction of the associated samples. Project-specific requirements may require all compounds to be treated as control analytes, or dictate use of project acceptance criteria.

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12.3.3. A matrix spike/duplicate matrix spike (MS/DMS) must be extracted and analyzed with every batch of 20 or fewer samples. The MS is prepared by spiking a sample aliquot with the matrix spike solution, and going through the entire extraction and analysis. Calculate percent recovery (%R) as follows:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

Calculate Relative Percent Difference (RPD) as:

$$\%RPD = \frac{R1 - R2}{(R1 + R2)/2} \times 100$$

Where R1 = recovered concentration in the MS
R2 = recovered concentration in the DMS

The acceptance limits for the MS/DMS recovery are given in Attachment A. If the MS/DMS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken. The RPD acceptance limits are 30% for water and 40% for soils, sediments, and solids. Project-specific requirements may dictate the use of project acceptance criteria.

12.3.4. The acceptance limits for the surrogates are given in Attachment A. If any surrogate recovery is outside acceptance criteria, the sample data must be closely evaluated for possible matrix interferences. If none are present, then corrective action must be taken. The sample should be re-analyzed if instrument factors (calibration, injection port) are suspected. If not, re-extraction and re-analysis is required, except in cases of high recovery and no positive hits in the sample for the analyte class represented by the particular surrogate.

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12.3.5. Additional QA/QC measures include control charting of QC sample results.

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. Qualitative Analysis - The qualitative identification of compounds determined by this procedure is based on retention time, and comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the instrument and conditions used for the sample analysis. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds should be identified as present when the criteria below are met.

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13.1.1. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

13.1.2. The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

13.1.3. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

13.1.4. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is <25% of the sum of the 2 peak heights. Otherwise, structural isomers are identified as isomeric pairs.

13.1.5. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks appear to represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification. When analytes coelute, the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

13.2. For samples containing components not associated with the calibration standards, a library search may be made of the purpose of tentative identification. Refer to method 8270C for guidance on tentatively identified compound (TIC) identification and quantification.

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13.3. Quantitation and Calculations

13.3.1. The GC/MS data stations, in current use, all use the H-P RTE Integrator to generate the raw data used to calculate the standards $\overline{RF_x}$ values, the sample amounts, and the spike values. The software does three passes through each data file. The first two identify and integrate each internal standard and surrogate. The third pass uses the time-drift information from the first two passes to search for all method analytes in the proper retention times and with the proper characteristic quantitation ions.

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When $\overline{RF_x}$ is used, calculate the extract concentration as follows:

$$C_{ex} = \frac{(Resp_x)(Amt_{ISTD})}{(Resp_{ISTD})(\overline{RF_x})}$$

Where:
C_{ex} = the concentration in the sample extract (ppm);
$Resp_x$ = the peak area of the analytes of interest;
$Resp_{ISTD}$ = the peak area of the associated internal standard;
Amt_{ISTD} = the amount, in ppm, of internal standard added
$\overline{RF_x}$ = the average response from the initial calibration.

13.3.2. The concentration of analytes in the original sample is computed using the following equations:

Aqueous Samples: $$Concentration\ (\mu g / L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$$

Where	Cex	=	Concentration in extract in μg/mL
	Vf	=	Final volume of extract in mL
	D	=	Dilution factor
	Vs	=	Volume of sample extracted, liters

Nonaqueous Samples: $$Concentration\ (mg / Kg) = \frac{(Cex)\ (Vf)\ (D)}{(W)}$$

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Where	Cex	=	Concentration in extract in μg/mL
	Vf	=	Final volume of extract in mL
	D	=	Dilution factor
	W	=	Weight of sample extracted in grams.

13.4. Data Review

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP for *Laboratory Data Review Process* for details.

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13.5. Reporting

13.5.1. Reports are generated in the CAS LIMS by compiling the SMO login, sample prep database, instrument, date, and client-specified report requirements (when specified). This compilation is then transferred to a file that the Stealth reporting system uses to generate a report. The forms generated may be CAS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.5.2. As an alternative, reports are generated using Excel© templates located in R:\SVM\forms. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The detected analytes, surrogate and matrix spikes are then transferred, by hand or electronically, to the templates.

13.5.3. Sample concentrations are reported when all QC criteria for the analysis have been met or the results are qualified with an appropriate footnote. For Arizona projects the appropriate Arizona qualifier must be used.

14. CONTINGENCIES FOR HANDLING OUT-OF- CONTROL OR UNACCEPTABLE DATA

Corrective action measures applicable to specific analysis steps are discussed in the applicable section of this (and other applicable) SOP(s). Also, refer to the SOP for *Nonconformity and Corrective Action* for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for *Report Generation* or in project-specific requirements.

15. METHOD PERFORMANCE

15.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2. The method detection limit (MDL) is established using the procedure described in the SOP for *the Determination of Method Detection Limits and Limits of Detection* (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

16. POLLUTION PREVENTION

It is the laboratory's practice to minimize the amount of solvents and reagents used to perform this method wherever technically sound, feasibly possible, and within method requirements. Standards are prepared in volumes consistent with laboratory use in order to minimize the volume of expired standards to be disposed of. The threat to the environment from solvents and/or reagents used in this method may be minimized when recycled or disposed of properly.

17. WASTE MANAGEMENT

17.1. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS EH&S Manual.

17.2. This method uses Methylene Chloride and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and recycled off site.

17.3. This method uses non-halogenated solvents and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and disposed of in accordance with Federal and State regulations.

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18. TRAINING

18.1. Training outline

18.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

18.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst for a period of 3 months. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

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18.1.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

18.2. Training is documented following the SOP *for Documentation of Technical Personnel Training*.

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18.2.1. NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

19. REFERENCES

19.1. Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry, Method 8270C, EPA Test Methods for Evaluating Solid Waste, SW-846, Final Update III, December 1996.

19.2. 8000C Method criteria, Arizona DHS, 2/13/2007. Available online at http://www.azdhs.gov/lab/license/tech/8000cmethod.pdf

19.3. *DoD Quality Systems Manual for Environmental Laboratories* Version 4.1 4/22/09.

CHANGES SINCE THE LAST REVISION

19.4. Sec 11.2 Removed reference to 8000C and SC.
19.5. Sec 11.3.9 Added DoD calibration requirement not allowing use of grand mean in calibrations.
19.6. Sec 11.3.13 Added DoD ICV acceptance criteria.
19.7. Sec 11.4.5 Added DoD CCV acceptance criteria.
19.8. Sec 12.3 Added DoD MB requirements.
19.9. Sec 18 Added training requirements.
19.10. Sec 19 Added DoD reference.
19.11. Sec 11.3.5 Added retention window positioning steps during ICAL.
19.12. Sec 11.4.3 Added retention window positioning step using CCV.

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TABLE 1

SEMI-VOLATILE ORGANIC COMPOUNDS - STANDARD ANALYTE LIST	METHOD REPORTING LIMITS	
Compound	**Water (μg/L)**	**Soil (mg/Kg)**
N-Nitrosodimethylamine	25	2.0
Aniline	25	1.0
Bis(2-chloroethyl) Ether	10	0.33
1,2-Dichlorobenzene	10	0.33
1,3-Dichlorobenzene	10	0.33
1,4-Dichlorobenzene	10	0.33
Bis(2-chloroisopropyl) Ether	10	0.33
N-Nitrosodi-n-propylamine	10	0.33
Hexachloroethane	10	0.33
Nitrobenzene	10	0.33
Isophorone	10	0.33
Bis(2-chloroethoxy)methane	10	0.33
1,2,4-Trichlorobenzene	10	0.33
Naphthalene	10	0.33
4-Chloroaniline	10	0.33
Hexachlorobutadiene	10	0.33
2-Methylnaphthalene	10	0.33
Hexachlorocyclopentadiene	10	0.33
2-Chloronaphthalene	10	0.33
2-Nitroaniline	25	2.0
Dimethyl Phthalate	10	0.33
Acenaphthylene	10	0.33
3-Nitroaniline	25	2.0
Acenaphthene	10	0.33
Dibenzofuran	10	0.33
2,4-Dinitrotoluene	10	0.33
2,6-Dinitrotoluene	10	0.33
Diethyl Phthalate	10	0.33

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TABLE 1 (continued)

SEMI-VOLATILE ORGANIC COMPOUNDS - STANDARD ANALYTE LIST	METHOD REPORTING LIMITS	
Compound	**Water (µg/L)**	**Soil (mg/Kg)**
4-Chlorophenyl Phenyl Ether	10	0.33
Fluorene	10	0.33
4-Nitroaniline	25	2.0
N-Nitrosodiphenylamine	10	0.33
4-Bromophenyl Phenyl Ether	10	0.33
Hexachlorobenzene	10	0.33
Phenanthrene	10	0.33
Anthracene	10	0.33
Di-n-butyl Phthalate	10	0.33
Fluoranthene	10	0.33
Pyrene	10	0.33
Butylbenzyl Phthalate	10	0.33
3,3'-Dichlorobenzidine	25	2.0
Benz(a)anthracene	10	0.33
Bis(2-ethylhexyl) Phthalate	10	0.33
Chrysene	10	0.33
Di-n-octyl Phthalate	10	0.33
Benzo(b)fluoranthene	10	0.33
Benzo(k)fluoranthene	10	0.33
Benzo(a)pyrene	10	0.33
Indeno(1,2,3-c,d)pyrene	10	0.33
Dibenz(a,h)anthracene	10	0.33
Benzo(g,h,i)perylene	10	0.33
Phenol	10	0.33
2-Chlorophenol	10	0.33
Benzyl Alcohol	10	0.33
2-Methylphenol	10	0.33
3- and 4-Methylphenol (coeluting cpds)	10	0.33

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TABLE 1 (continued)

SEMI-VOLATILE ORGANIC COMPOUNDS - STANDARD ANALYTE LIST	METHOD REPORTING LIMITS	
Compound	**Water (μg/L)**	**Soil (mg/Kg)**
2-Nitrophenol	10	0.33
2,4-Dimethylphenol	10	0.33
Benzoic Acid	25	2.0
2,4-Dichlorophenol	10	0.33
4-Chloro-3-methylphenol	10	0.33
2,4,6-Trichlorophenol	10	0.33
2,4,5-Trichlorophenol	10	0.33
2,4-Dinitrophenol	25	2.0
4-Nitrophenol	25	2.0
2-Methyl-4,6-dinitrophenol	25	2.0
Pentachlorophenol	25	2.0

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TABLE 1A

SEMI-VOLATILE ORGANIC COMPOUNDS - ADDITIONAL ANALYTES	METHOD REPORTING LIMITS	
Compound	**Water (µg/L)**	**Soil (mg/Kg)**
Pyridine	25	0.33
2-Picoline	10	0.60
N-Nitrosodiethylamine	10	0.33
Methyl Methanesulfonate	10	0.33
N-Nitrosoethylmethylamine	10	0.33
Pentachloroethane	10	1.0
Acetophenone	10	0.33
N-Nitrosopyrrolidine	10	0.33
N-Nitrosomorpholine	10	0.33
N-Nitrosopiperidine	10	0.33
O,O,O-Triethyl Phosphorothioate	10	0.33
2,6-Dichlorophenol	10	0.33
Hexachloropropene	10	0.33
N-Nitrosodi-*n*-butylamine	10	0.33
p-Phenylenediamine	10	0.33
Safrole	10	0.33
1,2,4,5-Tetrachlorobenzene	10	0.33
Isosafrole	10	2.0
1,3-Dinitrobenzene	10	0.33
Pentachlorobenzene	10	0.33
1-Naphthylamine	10	0.33
2-Naphthylamine	10	0.33
2,3,4,6-Tetrachlorophenol	10	0.33
Diphenylamine	10	0.33
1,3,5-Trinitrobenzene	25	2.0
Phenacetin	50	2.0
4-Aminobiphenyl	10	0.33
4-Nitroquinoline N-Oxide	10	2.0

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TABLE 1A continued

SEMI-VOLATILE ORGANIC COMPOUNDS - ADDITIONAL ANALYTES	METHOD REPORTING LIMITS	
Compound	**Water (µg/L)**	**Soil (mg/Kg)**
Total Aramite	50	3.0
3,3'-Dimethylbenzidine	25	2.0
7,12-Dimethylbenz(a)anthracene	10	0.33
Hexachlorophene	150	5.0
3-Methylcholanthrene	10	0.33
N,N-Dimethyl-1-phenethylamine	10	20
2-Acetylaminofluorene	10	0.33
o-Toluidine	10	0.33
Ethyl Methanesulfonate	10	0.33
1,4-Naphthoquinone	10	0.33
5-Nitro-*o*-toluidine	10	0.33
p-Dimethylaminoazobenzene	10	0.33
Pentachloronitrobenzene	50	2.0
Methapyrilene	100	4.0
Chlorobenzilate	10	0.33
2-sec-Butyl-4,6-Dinitrophenol (Dinoseb)	25	1.0
Diallate	10	0.33
Dimethoate	10	0.33
Disulfoton	10	0.33
Famphur	10	0.33
Isodrin	10	0.33
Kepone	100	4.0
Methyl Parathion	10	0.33
Parathion	10	0.33
Phorate	10	0.33
Pronamide	10	0.33
Thionazine	25	2.0

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TABLE 2
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

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Alternate tuning criteria (from Method 525.2 or CLP OLM03.1) may be used provided that method performance is not adversely affected and that method performance criteria is met. The criteria used must be the same for **all** ion abundance criteria checks associated with a given analysis. For example, initial calibration, continuing calibration(s), QC, and sample analyses for a given sample must all use the same criteria.

TABLE 3
8270 STANDARDS

CALIBRATION
Recommended: Supelco catalog #(or equivalent from other vendors*):

Equity CLP SVM Mix, 1000 ppm
Equity Cal Mix 4, 2000 ppm
Equity N-Nitrosodiphenylamine, 5000ppm
Equity Benzidines mix, 2000ppm

Accustandard 8270 surrogate mix, 4000ppm

Prepare 1 ml of each calibration point from a new unopened ampule.
Calibration curve: 5 ppm, 10 ppm, 20 ppm, 50 ppm, 80 ppm, 120 ppm, 160 ppm, and 200 ppm.
Add 10ul internal standard when curve is prepared.
Place ~ 100 µl in autosample vial insert, run, and discard after verifying data is acceptable.
Store in 5 ml amber mininert vials (2-5 ml vials for 10 ml) at -10°C. Expiration is 1 year from date prepared.
Order more solutions when down to one unopened ampule.

ICV
The ICV standard is prepared from the following Accustandard stock standards or equivalent from other vendors*:
Accustandard CLP-HC-BNR (2000ppm), CLP-HC-AR (2000ppm), Z-014ER3 (2000ppm), Z-014F (2000ppm), M-8270-SS (4000ppm), and App-9-114-10x (1000ppm).
Add 10 µl internal standard (Z-014J) for each 1 ml of ICV prepared.
Place in autosampler vial, analyze, recap, and refrigerate.
Expiration is 1 year after ICV was prepared or the expiration date of the stock standard whichever is earliest.

CCV & TUNE
Use the same solutions that were used for the calibration curve and the following:

AccuStandard (recommended) catalog #(or equivalent from other vendors*):

M-625C-3-1000X 1 ml 25 mg/ml DFTPP

Prepare 1 ml of 50 ppm 8270 CCV standard, place in autosample vial and cap with red crimp top. 50ppm is the nominal concentration.
Expiration date is 1 week after CCV was prepared.

RECAP AND STORE IMMEDIATELY AFTER INJECTING
Store remaining stock solutions in 1 ml amber mininert vial. Expiration date is six months after ampule is opened. Order when down to one unopened ampule.

* Vendor must be A2LA and/or ISO9000 certified.

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TABLE 4
QC Standards

Supelco Parent	Initial Concentration	Dilution (mixed)*	Final Conc.
8270 Surrogate			
B/N Surrogate Mix (cat no. 86-1377)	5000 ug/mL	20mL to 1000mL in MeOH	100 ug/mL
Acid Surrogate Mix (cat no 86-1376)	10000 ug/mL	15mL to 1000mL in MeOH	150 ug/mL
8270 Matrix Spike (mixed solution)			
CLP Semivolatiles Mix (cat no. 5S-06508)	1000 ug/mL	10mL to 100mL in MeOH	100 ug/mL
Benzidines Mix (cat no. 48467)	2000 ug/mL	5mL to 100mL in MeOH	100 ug/mL
N-Nitroso-diphenylamine (cat no. 46702-U)	5000 ug/mL	2mL to 100mL in MeOH	100 ug/mL
Misc mix (cat no. 86-1148)	2000 ug/mL	5mL to 100mL in MeOH	100 ug/mL
2,3,4,5-Tetrachlorophenol (cat no. 79131)	1000 ug/mL	10mL to 100mL in MeOH	100 ug/mL
1-Methylnaphthalene (cat no. 48162)	2000 ug/mL	5mL to 100mL in MeOH	100 ug/mL
Appendix 9 Mix (cat. no. App-9-186-20x)	2000 ug/mL	5mL to 100mL in MeOH	100 ug/mL

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Each time the QC standards are prepared, the supervisor should be made aware so that re-ordering can occur to maintain an adequate backup supply.

* For surrogate solution, split the total volume made into 4 bottles for storage and use. To avoid waste, the quantity made can be varied as anticipated for workload. For spike solution, split the total volume into 2 bottles for storage and use.

Standards Expiration: Unopened = 6 months from preparation date. Opened = 3 months from opening, or original 6 months, whichever is first. When breaking the seal on an unopened bottle, write the new expiration date on the bottle and in the standards logbook, with initial and date. Absolutely no expired standards are to be kept in the refrigerator.

TABLE 5
SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

1,4-Dichlorobenzene-d4 Internal Standard		
N-Nitrosodimethylamine	1,2-Dichlorobenzene	2-Methylphenol
Aniline	1,3-Dichlorobenzene	3- and 4-Methylphenol (coeluting cpds)
2-Fluorophenol (surrogate)	1,4-Dichlorobenzene	2-Picoline
Bis(2-chloroethyl) Ether	N-Nitrosodi-n-propylamine	Bis(2-chloroisopropyl) Ether
Phenol-d5 (surrogate)	Hexachloroethane	N-Nitrosopyrrolidine
Phenol	Methyl Methanesulfonate	N-Nitrosomorpholine
2-Chloropheol	N-Nitrosoethylmethylamine	*o*-Toluidine
Benzyl Alcohol	Acetophenone	Ethyl Methanesulfonate
Pyridine	N-Nitrosodiethylamine	Pentachloroethane
Naphthalene-d8 Internal Standard		
Nitrobenzene-d5(surrogate)	Hexachlorobutadiene	N-Nitrosodi-*n*-butylamine
Nitrobenzene	2-Methylnaphthalene	N-Nitrosopiperidine
Isophorone	2-Nitrophenol	N,N-Dimethyl-1-phenethylamine
Bis(2-chloroethoxy)methane	2,4-Dimethylphenol	O,O,O-Triethyl Phosphorothioate
1,2,4-Trichlorobenzene	Benzoic Acid	Hexachloropropene
Naphthalene	2,4-Dichlorophenol	*p*-Phenylenediamine
4-Chloroaniline	4-Chloro-3-methylphenol	Safrole
	2,6-Dichlorophenol	1,2,4,5-Tetrachlorobenzene
Acenaphthene-d10 Internal Standard		
2-Fluorobiphenyl (surrogate)	2,4-Dinitrotoluene	1-Naphthylamine
Hexachlorocyclopentadiene	2,6-Dinitrotoluene	2-Naphthylamine
2-Chloronaphthalene	Diethyl Phthalate	2,3,4,6-Tetrachlorophenol
2-Nitroaniline	4-Chlorophenyl Phenyl Ether	2,4,6-Tribromophenol (surrogate)
3-Nitroaniline	Fluorene	Pentachlorobenzene
4-Nitroaniline	4-Nitrophenol	1,3-Dinitrobenzene
Dimethyl Phthalate	2,4,6-Trichlorophenol	1,4-Naphthoquinone
Acenaphthylene	2,4,5-Trichlorophenol	5-Nitro-*o*-toluidine
Acenaphthene	2,4-Dinitrophenol	Thionazine
Dibenzofuran	Isosafrole	Diphenylamine

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TABLE 5 continued

Pnenanthrene-d10 Internal Standard		
N-Nitrosodiphenylamine	Pentachlorophenol	Pentachloronitrobenzene
4-Bromophenyl Phenyl Ether	1,3,5-Trinitrobenzene	Disulfoton
Hexachlorobenzene	Phorate	2-sec-Butyl-4,6-Dinitrophenol (Dinoseb)
Phenanthrene	Phenacetin	Methyl Parathion
Anthracene	Diallate	4-Nitroquinoline N-Oxide
Di-n-butyl Phthalate	Dimethoate	Parathion
Fluoranthene	4-Aminobiphenyl	Methapyrilene
2-Methyl-4,6-dinitrophenol	Pronamide	Isodrin
Chrysene-d12 Internal Standard		
Pyrene	Bis(2-ethylhexyl) Phthalate	Chlorobenzilate
Butylbenzyl Phthalate	Chrysene	Kepone
Benzidine	Terphenyl-d14 (surrogate)	3,3'-Dimethylbenzidine
3,3'-Dichlorobenzidine	Total Aramite	Famphur
Benz(a)anthracene	*p*-Dimethylaminoazobenzene	2-Acetylaminofluorene
Perylene-d12 Internal Standard		
Di-n-octyl Phthalate	Indeno(1,2,3-c,d)pyrene	Hexachlorophene
Benzo(b)fluoranthene	Dibenz(a,h)anthracene	3-Methylcholanthrene
Benzo(k)fluoranthene	Benzo(g,h,i)perylene	
Benzo(a)pyrene	7,12-Dimethylbenz(a)anthracene	

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TABLE 6
Control Analytes for Non-DoD Projects

1,2,4-Trichlorobenzene
1,4-Dichlorobenzene
2,4-Dinitrotoluene
2-Chloronaphthalene
2-Chlorophenol
4-Bromophenyl Phenyl Ether
4-Chloro-3-methylphenol
4-Nitrophenol
Acenaphthene
Benzo(a)pyrene
Diethyl Phthalate
Hexachloroethane
N-Nitrosodi-n-propylamine
Pentachlorophenol
Phenol
Pyrene

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ATTACHMENT A

Method Reporting Limits
Method Detection Limits
QC Acceptance Criteria

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Method Information

Method: 8270C
Prep: EPA 3541
Matrix: SOIL
Title: Base Neutral/Acid Semivolatile Organic Compounds

Source: SW-846
Revision: 7/8/2009

Method ID: MJ250

Optimal Prep
Sample Amount: 30.00 g
Final Volume: 1.00 ml

Hold Time
Analytical : 40 days from date Prep
Preparation : 14 days from date Sampled

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
1,4-Dichlorobenzene-d4	INT_STD				.5-2		-		mg/Kg	3855-82-1
Acenaphthene-d10	INT_STD				.5-2		-		mg/Kg	15067-26-2
Chrysene-d12	INT_STD				.5-2		-		mg/Kg	1719-03-5
Naphthalene-d8	INT_STD				.5-2		-		mg/Kg	1146-65-2
Perylene-d12	INT_STD				.5-2		-		mg/Kg	1520-96-3
Phenanthrene-d10	INT_STD				.5-2		-		mg/Kg	1517-22-2
1,2,4-Trichlorobenzene	MS		0.0110	0.33	28-105	40	42-91	40	mg/Kg	120-82-1
1,4-Dichlorobenzene	MS	CCC	0.0175	0.33	28-95	40	41-85	40	mg/Kg	106-46-7
2,4-Dinitrotoluene	MS		0.0149	0.33	36-127	40	48-119	40	mg/Kg	121-14-2
2-Chlorophenol	MS		0.0099	0.33	27-96	40	42-86	40	mg/Kg	95-57-8
4-Chloro-3-methylphenol	MS	CCC	0.0166	0.33	13-122	40	44-101	40	mg/Kg	59-50-7
4-Nitrophenol	MS	SPCC	0.146	2.0	12-141	40	41-121	40	mg/Kg	100-02-7
Acenaphthene	MS	CCC	0.0134	0.33	30-113	40	49-97	40	mg/Kg	83-32-9
N,N-Dimethylaniline	MS		0.0604	0.33	70-130	40	70-130	40	mg/Kg	121-69-7
N-Nitrosodi-n-propylamine	MS	SPCC	0.0191	0.33	32-107	40	41-96	40	mg/Kg	621-64-7
Pentachlorophenol	MS	CCC	0.125	2.0	10-135	40	39-112	40	mg/Kg	87-86-5
Phenol	MS	CCC	0.0195	0.33	18-106	40	40-87	40	mg/Kg	108-95-2
Pyrene	MS		0.0140	0.33	24-126	40	45-116	40	mg/Kg	129-00-0

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Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
2,4,6-Tribromophenol	SURR				20-123		-		PERCENT	118-79-6
2-Fluorobiphenyl	SURR				32-104		-		PERCENT	321-60-8
2-Fluorophenol	SURR				20-83		-		PERCENT	367-12-4
Nitrobenzene-d5	SURR				29-100		-		PERCENT	4165-60-0
Phenol-d6	SURR				23-90		-		PERCENT	13127-88-3
Terphenyl-d14	SURR				37-133		-		PERCENT	1718-51-0
1,2,3,4-Tetrachlorobenzene	TIC_TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	634-66-2
1,2,3,5-Tetrachlorobenzene	TIC_TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	634-90-2
1,2,3-Trichlorobenzene	TIC_TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	87-61-6
1,2,4,5-Tetrachlorobenzene	TIC_TRG		0.020	0.33	70-130	40	70-130	40	mg/Kg	95-94-3
1,3,5-Trichlorobenzene	TIC_TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	108-70-3
1-Methylnaphthalene	TIC_TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	90-12-0
2,3,7,8-Tetrachlorodibenzo-p-(	TIC_TRG		0.33	0.33	-		-		mg/Kg	1746-01-6
Acrylamide	TIC_TRG		50	50	70-130	40	70-130	40	mg/Kg	79-06-1
Diisobutyl Phthalate	TIC_TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	84-69-5
Diphenylamine	TIC_TRG		0.010	0.33	70-130	40	70-130	40	mg/Kg	122-39-4
Hexachlorocyclohexane	TIC_TRG		0.67	0.67	-		-		mg/Kg	608-73-1
HPMO	TIC_TRG		0.121	2.0	-		-		mg/Kg	3375-84-6
OPMO	TIC_TRG		0.0902	2.0	-		-		mg/Kg	CASID30031
Pentachlorobenzene	TIC_TRG		0.019	0.33	70-130	40	70-130	40	mg/Kg	608-93-5
1,2-Dichlorobenzene	TRG		0.0179	0.33	29-93	40	41-86	40	mg/Kg	95-50-1
1,2-Diphenylhydrazine	TRG		0.0146	0.33	44-107	40	45-111	40	mg/Kg	122-66-7
1,3,5-Trinitrobenzene	TRG		0.068	0.67	70-130	40	70-130	40	mg/Kg	99-35-4
1,3-Dichlorobenzene	TRG		0.0183	0.33	28-91	40	41-84	40	mg/Kg	541-73-1
1,3-Dinitrobenzene	TRG		0.073	0.33	70-130	40	70-130	40	mg/Kg	99-65-0
1,4-Dichlorobutane	TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	110-56-5
1,4-Dioxane	TRG		0.13	0.67	70-130	40	70-130	40	mg/Kg	123-91-1
1,4-Naphthoquinone	TRG		0.065	0.33	70-130	40	70-130	40	mg/Kg	130-15-4
1-Chloronaphthalene	TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	90-13-1
1-Methyl-2-pyrrolidinone	TRG		0.095	0.33	70-130	40	33-140	40	mg/Kg	872-50-4
1-Naphthylamine	TRG		0.036	0.33	70-130	40	70-130	40	mg/Kg	134-32-7
2,3,4,6-Tetrachlorophenol	TRG		0.046	1.0	45-110	40	45-110	40	mg/Kg	58-90-2
2,4,5-Trichlorophenol	TRG		0.0171	0.33	27-115	40	46-103	40	mg/Kg	95-95-4
2,4,6-Trichlorophenol	TRG	CCC	0.0143	0.33	24-119	40	46-101	40	mg/Kg	88-06-2
2,4-Dichlorophenol	TRG	CCC	0.0164	0.33	16-119	40	43-94	40	mg/Kg	120-83-2

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Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
2,4-Dimethylphenol	TRG		0.0151	0.33	10-117	40	16-89	40	mg/Kg	105-67-9
2,4-Dinitrophenol	TRG	SPCC	0.112	2.0	10-145	40	28-116	40	mg/Kg	51-28-5
2,6-Dichlorophenol	TRG		0.016	0.33	70-130	40	70-130	40	mg/Kg	87-65-0
2,6-Diisopropylnaphthalene	TRG		0.33	0.33	14-136	40	14-136	40	mg/Kg	24157-81-1
2,6-Dinitrotoluene	TRG		0.0156	0.33	36-125	40	49-114	40	mg/Kg	606-20-2
2-Acetylaminofluorene	TRG		0.027	4.0	70-130	40	70-130	40	mg/Kg	53-96-3
2-Chloronaphthalene	TRG		0.0100	0.33	26-114	40	44-95	40	mg/Kg	91-58-7
2-Methyl-4,6-dinitrophenol	TRG		0.1434	2.0	10-138	40	41-119	40	mg/Kg	534-52-1
2-Methylnaphthalene	TRG		0.0110	0.33	30-105	40	44-95	40	mg/Kg	91-57-6
2-Methylphenol	TRG		0.0167	0.33	14-103	40	35-92	40	mg/Kg	95-48-7
2-Naphthylamine	TRG		0.063	0.33	70-130	40	70-130	40	mg/Kg	91-59-8
2-Nitroaniline	TRG		0.0169	2.0	34-121	40	48-108	40	mg/Kg	88-74-4
2-Nitrophenol	TRG	CCC	0.0139	0.33	20-119	40	44-95	40	mg/Kg	88-75-5
2-Picoline	TRG		0.055	0.67	70-130	40	70-130	40	mg/Kg	109-06-8
3,3'-Dichlorobenzidine	TRG		0.0270	2.0	10-120	40	38-116	40	mg/Kg	91-94-1
3,3'-Dimethylbenzidine	TRG		1.2	2.0	70-130	40	70-130	40	mg/Kg	119-93-7
3-Methylcholanthrene	TRG		0.062	0.33	70-130	40	70-130	40	mg/Kg	56-49-5
3-Nitroaniline	TRG		0.175	2.0	15-123	40	46-111	40	mg/Kg	99-09-2
3-Nitrophenol	TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	554-84-7
4-Aminobiphenyl	TRG		0.065	0.33	70-130	40	70-130	40	mg/Kg	92-67-1
4-Bromophenyl Phenyl Ether	TRG		0.0122	0.33	32-127	40	49-110	40	mg/Kg	101-55-3
4-Chloroaniline	TRG		0.0144	0.33	11-98	40	37-96	40	mg/Kg	106-47-8
4-Chlorophenyl Phenyl Ether	TRG		0.0160	0.33	35-115	40	46-105	40	mg/Kg	7005-72-3
4-Methylphenol	TRG		0.0168	0.33	10-111	40	38-94	40	mg/Kg	106-44-5
4-Nitroaniline	TRG		0.179	2.0	10-137	40	39-120	40	mg/Kg	100-01-6
4-Nitroquinoline N-Oxide	TRG		0.068	3.0	70-130	40	70-130	40	mg/Kg	56-57-5
5-Nitro-o-toluidine	TRG		0.011	0.33	70-130	40	70-130	40	mg/Kg	99-55-8
7,12-Dimethylbenz(a)anthrace	TRG		0.015	0.33	70-130	40	70-130	40	mg/Kg	57-97-6
a,a-Dimethylphenethylamine	TRG		0.23	1.0	70-130	40	70-130	40	mg/Kg	122-09-8
Acenaphthylene	TRG		0.016	0.33	18-128	40	46-99	40	mg/Kg	208-96-8
Acetophenone	TRG		0.023	0.33	70-130	40	70-130	40	mg/Kg	98-86-2
Aniline	TRG		0.0216	1.0	10-92	40	24-92	40	mg/Kg	62-53-3
Anthracene	TRG		0.0139	0.33	21-134	40	50-108	40	mg/Kg	120-12-7
Aramite, Total	TRG		0.056	2.0	70-130	40	70-130	40	mg/Kg	140-57-8
Atrazine	TRG		0.017	0.33	70-130	40	70-130	40	mg/Kg	1912-24-9

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Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
Azobenzene	TRG		0.0146	0.33	70-130	40	70-130	40	mg/Kg	103-33-3
Benz(a)anthracene	TRG		0.0123	0.33	33-121	40	54-109	40	mg/Kg	56-55-3
Benzaldehyde	TRG		0.021	0.33	70-130	40	70-130	40	mg/Kg	100-52-7
Benzidine	TRG		0.42	2.0	10-81	40	10-81	40	mg/Kg	92-87-5
Benzo(a)pyrene	TRG	CCC	0.0198	0.33	31-122	40	51-114	40	mg/Kg	50-32-8
Benzo(b)fluoranthene	TRG		0.0172	0.33	33-124	40	53-110	40	mg/Kg	205-99-2
Benzo(g,h,i)perylene	TRG		0.0202	0.33	26-126	40	50-110	40	mg/Kg	191-24-2
Benzo(k)fluoranthene	TRG		0.0194	0.33	36-117	40	52-112	40	mg/Kg	207-08-9
Benzoic Acid	TRG		0.139	2.0	0-176	40	10-104	40	mg/Kg	65-85-0
Benzophenone	TRG		0.33	0.33	15-116	40	15-116	40	mg/Kg	119-61-9
Benzyl Alcohol	TRG		0.0168		25-106	40	35-96	40	mg/Kg	100-51-6
Biphenyl	TRG		0.009	0.33	70-130	40	70-130	40	mg/Kg	92-52-4
Bis(2-chloroethoxy)methane	TRG		0.0110	0.33	33-106	40	45-93	40	mg/Kg	111-91-1
Bis(2-chloroethyl) Ether	TRG		0.0117	0.33	29-103	40	42-88	40	mg/Kg	111-44-4
Bis(2-chloroisopropyl) Ether	TRG		0.0141	0.33	25-106	40	39-94	40	mg/Kg	39638-32-9
Bis(2-ethylhexyl) Phthalate	TRG		0.0186	0.33	35-126	40	51-115	40	mg/Kg	117-81-7
Butyl Benzyl Phthalate	TRG		0.0163	0.33	34-125	40	51-114	40	mg/Kg	85-68-7
Caprolactam	TRG		0.147	0.67	70-130	40	70-130	40	mg/Kg	105-60-2
Carbazole	TRG		0.0112	0.33	49-118	40	51-113	40	mg/Kg	86-74-8
Chlorobenzilate	TRG		0.020	0.33	70-130	40	70-130	40	mg/Kg	510-15-6
Chrysene	TRG		0.0118	0.33	33-121	40	53-109	40	mg/Kg	218-01-9
Diallate	TRG		0.022	0.33	70-130	40	70-130	40	mg/Kg	2303-16-4
Diazinon	TRG		0.33	0.33	22-135	40	22-135	40	mg/Kg	333-41-5
Dibenz(a,h)anthracene	TRG		0.0275	0.33	40-118	40	53-114	40	mg/Kg	53-70-3
Dibenzofuran	TRG		0.0118	0.33	35-112	40	47-101	40	mg/Kg	132-64-9
Dicyclopentadiene	TRG		0.032	0.33	70-130	40	70-130	40	mg/Kg	77-73-6
Diethyl Phthalate	TRG		0.0141	0.33	37-128	40	45-121	40	mg/Kg	84-66-2
Diethylene Glycol Dibenzoate	TRG		0.33	0.33	32-147	40	32-147	40	mg/Kg	120-55-8
Dimethoate	TRG		0.024	0.33	70-130	40	70-130	40	mg/Kg	60-51-5
Dimethyl Phthalate	TRG		0.0164	0.33	36-123	40	48-113	40	mg/Kg	131-11-3
Di-n-butyl Phthalate	TRG		0.0121	0.33	34-139	40	50-124	40	mg/Kg	84-74-2
Di-n-octyl Phthalate	TRG	CCC	0.0240	0.33	36-136	40	51-121	40	mg/Kg	117-84-0
Dinoseb	TRG		0.052	0.33	70-130	40	70-130	40	mg/Kg	88-85-7
Disulfoton	TRG		0.016	0.33	70-130	40	70-130	40	mg/Kg	298-04-4
Ethyl Methanesulfonate	TRG		0.089	0.33	70-130	40	70-130	40	mg/Kg	62-50-0

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Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
Ethylene Glycol Butyl Ether (E	TRG		0.0058	0.33	34-96	40	34-96	40	mg/Kg	111-76-2
Famphur	TRG		0.071	4.0	70-130	40	70-130	40	mg/Kg	52-85-7
Fluoranthene	TRG	CCC	0.0115	0.33	25-135	40	48-116	40	mg/Kg	206-44-0
Fluorene	TRG		0.0130	0.33	31-116	40	46-103	40	mg/Kg	86-73-7
Hexachlorobenzene	TRG		0.0147	0.33	37-119	40	50-109	40	mg/Kg	118-74-1
Hexachlorobutadiene	TRG	CCC	0.0141	0.33	27-107	40	42-91	40	mg/Kg	87-68-3
Hexachlorocyclopentadiene	TRG	SPCC	0.0125	0.33	10-79	40	10-68	40	mg/Kg	77-47-4
Hexachloroethane	TRG		0.0216	0.33	31-91	40	40-87	40	mg/Kg	67-72-1
Hexachlorophene	TRG		1.2	6.7	70-130	40	70-130	40	mg/Kg	70-30-4
Hexachloropropene	TRG		0.019	0.33	70-130	40	70-130	40	mg/Kg	1888-71-7
Indeno(1,2,3-cd)pyrene	TRG		0.0389	0.33	29-132	40	53-112	40	mg/Kg	193-39-5
Isodrin	TRG		0.0089	0.33	70-130	40	70-130	40	mg/Kg	465-73-6
Isophorone	TRG		0.0140	0.33	33-103	40	42-93	40	mg/Kg	78-59-1
Isosafrole	TRG		0.017	0.67	70-130	40	70-130	40	mg/Kg	120-58-1
Kepone	TRG		0.39	4.0	70-130	40	70-130	40	mg/Kg	143-50-0
Malathion	TRG		0.83	0.83	70-130	40	70-130	40	mg/Kg	121-75-5
Methapyrilene	TRG		0.091	1.0	70-130	40	70-130	40	mg/Kg	91-80-5
Methyl Methanesulfonate	TRG		0.089	0.33	70-130	40	70-130	40	mg/Kg	66-27-3
Methyl Parathion	TRG		0.013	0.33	70-130	40	70-130	40	mg/Kg	298-00-0
Naphthalene	TRG		0.0144	0.33	31-103	40	44-92	40	mg/Kg	91-20-3
n-Dodecane	TRG		0.33	0.33	26-95	40	26-95	40	mg/Kg	112-40-3
Nitrobenzene	TRG		0.0261	0.33	34-99	40	44-92	40	mg/Kg	98-95-3
N-Nitrosodiethylamine	TRG		0.029	0.33	70-130	40	70-130	40	mg/Kg	55-18-5
N-Nitrosodimethylamine	TRG		0.0251	2.0	20-98	40	34-93	40	mg/Kg	62-75-9
N-Nitrosodi-n-butylamine	TRG		0.11	0.33	70-130	40	70-130	40	mg/Kg	924-16-3
N-Nitrosodiphenylamine	TRG	CCC	0.018	0.33	14-143	40	44-113	40	mg/Kg	86-30-6
N-Nitrosomethylethylamine	TRG		0.099	0.33	70-130	40	70-130	40	mg/Kg	10595-95-6
N-Nitrosomorpholine	TRG		0.019	0.33	70-130	40	70-130	40	mg/Kg	59-89-2
N-Nitrosopiperidine	TRG		0.023	0.33	70-130	40	70-130	40	mg/Kg	100-75-4
N-Nitrosopyrrolidine	TRG		0.092	0.33	70-130	40	70-130	40	mg/Kg	930-55-2
O,O,O-Triethyl Phosphorothio	TRG		0.022	0.33	70-130	40	70-130	40	mg/Kg	126-68-1
o-Toluidine	TRG		0.017	0.33	70-130	40	70-130	40	mg/Kg	95-53-4
Parathion	TRG		0.084	0.33	70-130	40	70-130	40	mg/Kg	56-38-2
p-Dimethylaminoazobenzene	TRG		0.012	0.33	70-130	40	70-130	40	mg/Kg	60-11-7
Pentachloroethane	TRG		0.015	1.0	70-130	40	70-130	40	mg/Kg	76-01-7

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Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
Pentachloronitrobenzene	TRG		0.088	2.0	70-130	40	70-130	40	mg/Kg	82-68-8
PGMEA	TRG		0.036	1.0	70-130	40	70-130	40	mg/Kg	108-65-6
Phenacetin	TRG		0.013	2.0	70-130	40	70-130	40	mg/Kg	62-44-2
Phenanthrene	TRG		0.0100	0.33	35-119	40	52-108	40	mg/Kg	85-01-8
Phorate	TRG		0.026	0.33	70-130	40	70-130	40	mg/Kg	298-02-2
Picric Acid	TRG		3.3	3.3	70-130	40	70-130	40	mg/Kg	88-89-1
p-Phenylenediamine	TRG		0.79	2.0	70-130	40	70-130	40	mg/Kg	106-50-3
Pronamide	TRG		0.018	0.33	70-130	40	70-130	40	mg/Kg	23950-58-5
Pyridine	TRG		0.0199	0.33	10-153	40	10-153	40	mg/Kg	110-86-1
Quinoline	TRG		0.33	0.33	70-130	40	70-130	40	mg/Kg	91-22-5
Safrole	TRG		0.013	0.33	70-130	40	70-130	40	mg/Kg	94-59-7
Sulfotep	TRG		0.018	0.33	70-130	40	70-130	40	mg/Kg	3689-24-5
Thionazin	TRG		0.017	2.0	70-130	40	70-130	40	mg/Kg	297-97-2

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Method Information

Method: 8270C
Prep: EPA 3520C
Matrix: WATER
Title: Base Neutral/Acid Semivolatile Organic compounds

Source: SW-846
Revision: 7/8/2009

Method ID: MJ121

Optimal Prep
Sample Amount: 1,000.00 ml
Final Volume: 1.00 ml

Hold Time
Analytical : 40 days from date Prep
Preparation 7 days from date Sampled

Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
1,4-Dichlorobenzene-d4	INT_STD				.5-2		-		ug/L	3855-82-1
Acenaphthene-d10	INT_STD				.5-2		-		ug/L	15067-26-2
Chrysene-d12	INT_STD				.5-2		-		ug/L	1719-03-5
Naphthalene-d8	INT_STD				.5-2		-		ug/L	1146-65-2
Perylene-d12	INT_STD				.5-2		-		ug/L	1520-96-3
Phenanthrene-d10	INT_STD				.5-2		-		ug/L	1517-22-2
1,2,4-Trichlorobenzene	MS		0.355	10	49-97	30	51-103	30	ug/L	120-82-1
1,4-Dichlorobenzene	MS	CCC	0.317	10	47-93	30	54-96	30	ug/L	106-46-7
2,4-Dinitrotoluene	MS		0.274	10	62-123	30	61-126	30	ug/L	121-14-2
2-Chlorophenol	MS		0.311	10	49-100	30	59-101	30	ug/L	95-57-8
4-Chloro-3-methylphenol	MS	CCC	0.490	10	61-111	30	65-111	30	ug/L	59-50-7
4-Nitrophenol	MS	SPCC	1.92	25	57-119	30	55-126	30	ug/L	100-02-7
Acenaphthene	MS	CCC	0.281	10	57-107	30	61-110	30	ug/L	83-32-9
N,N-Dimethylaniline	MS		2.22	10	70-130	30	70-130	30	ug/L	121-69-7
N-Nitrosodi-n-propylamine	MS	SPCC	0.496	10	53-109	30	56-115	30	ug/L	621-64-7
Pentachlorophenol	MS	CCC	2.44	25	42-127	30	60-116	30	ug/L	87-86-5
Phenol	MS	CCC	0.324	10	47-101	30	48-105	30	ug/L	108-95-2
Pyrene	MS		0.731	10	52-121	30	53-124	30	ug/L	129-00-0

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Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
2,4,6-Tribromophenol	SURR				46-127		-		PERCENT	118-79-6
2-Fluorobiphenyl	SURR				48-114		-		PERCENT	321-60-8
2-Fluorophenol	SURR				39-103		-		PERCENT	367-12-4
Nitrobenzene-d5	SURR				46-115		-		PERCENT	4165-60-0
Phenol-d6	SURR				38-107		-		PERCENT	13127-88-3
Terphenyl-d14	SURR				22-146		-		PERCENT	1718-51-0
1-Methylnaphthalene	TIC_TRG		10	10	-		-		ug/L	90-12-0
2,3,7,8-Tetrachlorodibenzo-p-(	TIC_TRG		50	50	-		-		ug/L	1746-01-6
alpha-Terpineol	TIC_TRG		10	10	-		-		ug/L	10482-56-1
Chloroacetaldehyde	TIC_TRG		10	10	-		-		ug/L	107-20-0
HPMO	TIC_TRG		6.15	25	-		-		ug/L	3375-84-6
OPMO	TIC_TRG		3.86	25	-		-		ug/L	CASID30031
Paraldehyde	TIC_TRG		10	10	-		-		ug/L	123-63-7
Toluene diisocyanate	TIC_TRG		10	10	-		-		ug/L	CASID30165
1,2,4,5-Tetrachlorobenzene	TRG		0.26	10	70-130	30	70-130	30	ug/L	95-94-3
1,2-Dichlorobenzene	TRG		0.431	10	47-94	30	55-97	30	ug/L	95-50-1
1,2-Diphenylhydrazine	TRG		0.509	10	54-118	30	58-117	30	ug/L	122-66-7
1,3,5-Trinitrobenzene	TRG		0.38	25	70-130	30	70-130	30	ug/L	99-35-4
1,3-Dichlorobenzene	TRG		0.352	10	45-92	30	55-95	30	ug/L	541-73-1
1,3-Dinitrobenzene	TRG		0.52	10	70-130	30	70-130	30	ug/L	99-65-0
1,4-Dichlorobutane	TRG		1	10	70-130	30	70-130	30	ug/L	110-56-5
1,4-Dioxane	TRG		3.7	25	70-130	30	70-130	30	ug/L	123-91-1
1,4-Naphthoquinone	TRG		0.21	10	70-130	30	70-130	30	ug/L	130-15-4
1-Chloronaphthalene	TRG		1	10	70-130	30	70-130	30	ug/L	90-13-1
1-Methyl-2-pyrrolidinone	TRG		2.2	25	70-130	30	70-130	30	ug/L	872-50-4
1-Naphthylamine	TRG		0.70	10	70-130	30	70-130	30	ug/L	134-32-7
2,3,4,6-Tetrachlorophenol	TRG		0.55	10	56-124	30	56-124	30	ug/L	58-90-2
2,3,5,6-Tetrachlorophenol	TRG		0.62	10	70-130	30	70-130	30	ug/L	935-95-5
2,4,5-Trichlorophenol	TRG		0.381	10	60-112	30	62-117	30	ug/L	95-95-4
2,4,6-Trichlorophenol	TRG	CCC	0.203	10	60-113	30	63-117	30	ug/L	88-06-2
2,4-Dichlorophenol	TRG	CCC	0.297	10	55-105	30	59-107	30	ug/L	120-83-2
2,4-Dimethylphenol	TRG		0.264	10	22-116	30	32-100	30	ug/L	105-67-9
2,4-Dinitrophenol	TRG	SPCC	2.22	25	38-122	30	33-126	30	ug/L	51-28-5
2,6-Dichlorophenol	TRG		0.48	10	70-130	30	70-130	30	ug/L	87-65-0
2,6-Dinitrotoluene	TRG		0.349	10	69-113	30	65-122	30	ug/L	606-20-2

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Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
2-Acetylaminofluorene	TRG		0.23	100	70-130	30	70-130	30	ug/L	53-96-3
2-Chloronaphthalene	TRG		0.290	10	48-106	30	54-107	30	ug/L	91-58-7
2-Methyl-4,6-dinitrophenol	TRG		2.12	25	57-120	30	51-127	30	ug/L	534-52-1
2-Methylnaphthalene	TRG		0.239	10	57-100	30	57-106	30	ug/L	91-57-6
2-Methylphenol	TRG		0.328	10	52-100	30	51-108	30	ug/L	95-48-7
2-Naphthylamine	TRG		1.0	10	70-130	30	70-130	30	ug/L	91-59-8
2-Nitroaniline	TRG		0.336	25	23-138	30	62-117	30	ug/L	88-74-4
2-Nitrophenol	TRG	CCC	0.373	10	54-104	30	58-108	30	ug/L	88-75-5
2-Picoline	TRG		4.5	25	70-130	30	70-130	30	ug/L	109-06-8
3- and 4-Methylphenol Coeluti	TRG		0.478	10	-		-		ug/L	CASID30030
3,3'-Dichlorobenzidine	TRG		0.270	25	12-120	30	52-120	30	ug/L	91-94-1
3,3'-Dimethylbenzidine	TRG		5.4	20	70-130	30	70-130	30	ug/L	119-93-7
3-Methylcholanthrene	TRG		0.31	10	70-130	30	70-130	30	ug/L	56-49-5
3-Nitroaniline	TRG		3.25	25	57-119	30	63-120	30	ug/L	99-09-2
3-Nitrophenol	TRG		1	10	70-130	30	70-130	30	ug/L	554-84-7
4-Aminobiphenyl	TRG		1.4	10	70-130	30	70-130	30	ug/L	92-67-1
4-Bromophenyl Phenyl Ether	TRG		0.274	10	61-114	30	64-118	30	ug/L	101-55-3
4-Chloroaniline	TRG		0.375	10	42-117	30	57-111	30	ug/L	106-47-8
4-Chlorophenyl Phenyl Ether	TRG		0.278	10	56-116	30	59-113	30	ug/L	7005-72-3
4-Methylphenol	TRG		0.478	10	54-108	30	47-118	30	ug/L	106-44-5
4-Nitroaniline	TRG		4.03	25	37-130	30	53-127	30	ug/L	100-01-6
4-Nitroquinoline N-Oxide	TRG		4.6	100	70-130	30	70-130	30	ug/L	56-57-5
5-Nitro-o-toluidine	TRG		1.0	10	70-130	30	70-130	30	ug/L	99-55-8
7,12-Dimethylbenz(a)anthrace	TRG		0.32	10	70-130	30	70-130	30	ug/L	57-97-6
a,a-Dimethylphenethylamine	TRG		6.4	25	70-130	30	70-130	30	ug/L	122-09-8
Acenaphthylene	TRG		0.236	10	59-101	30	58-109	30	ug/L	208-96-8
Acetophenone	TRG		0.60	10	70-130	30	70-130	30	ug/L	98-86-2
Aniline	TRG		0.487	25	23-116	30	35-118	30	ug/L	62-53-3
Anthracene	TRG		0.612	10	59-112	30	62-116	30	ug/L	120-12-7
Aramite, Total	TRG		0.52	50	70-130	30	70-130	30	ug/L	140-57-8
Atrazine	TRG		0.46	10	70-130	30	70-130	30	ug/L	1912-24-9
Azobenzene	TRG		0.509	10	70-130	30	70-130	30	ug/L	103-33-3
Benz(a)anthracene	TRG		0.591	10	63-110	30	69-113	30	ug/L	56-55-3
Benzaldehyde	TRG		0.50	10	70-130	30	70-130	30	ug/L	100-52-7
Benzidine	TRG		19	50	10-181	30	10-181	30	ug/L	92-87-5

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Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
Benzo(a)pyrene	TRG		0.651	10	54-123	30	57-124	30	ug/L	50-32-8
Benzo(b)fluoranthene	TRG		0.584	10	59-117	30	66-117	30	ug/L	205-99-2
Benzo(g,h,i)perylene	TRG		0.812	10	61-116	30	68-116	30	ug/L	191-24-2
Benzo(k)fluoranthene	TRG		0.827	10	57-118	30	63-119	30	ug/L	207-08-9
Benzoic acid	TRG		5.819	25	10-136	30	10-109	30	ug/L	65-85-0
Benzyl alcohol	TRG		0.377	10	51-105	30	53-111	30	ug/L	100-51-6
Biphenyl	TRG		0.66	10	70-130	30	70-130	30	ug/L	92-52-4
Bis(2-chloroethoxy)methane	TRG		0.276	10	55-101	30	57-108	30	ug/L	111-91-1
Bis(2-chloroethyl) Ether	TRG		0.333	10	51-97	30	56-104	30	ug/L	111-44-4
Bis(2-chloroisopropyl) Ether	TRG		0.311	10	43-106	30	53-105	30	ug/L	39638-32-9
Bis(2-ethylhexyl) Phthalate	TRG		1.89	10	58-122	30	64-122	30	ug/L	117-81-7
Butyl Benzyl Phthalate	TRG		0.470	10	60-117	30	64-121	30	ug/L	85-68-7
Caprolactam	TRG		0.58	25	70-130	30	70-130	30	ug/L	105-60-2
Carbazole	TRG		0.237	10	63-122	30	63-122	30	ug/L	86-74-8
Chlorobenzilate	TRG		0.45	10	70-130	30	70-130	30	ug/L	510-15-6
Chrysene	TRG		0.787	10	61-113	30	68-114	30	ug/L	218-01-9
Diallate	TRG		0.47	10	70-130	30	70-130	30	ug/L	2303-16-4
Dibenz(a,h)anthracene	TRG		0.752	10	60-120	30	65-121	30	ug/L	53-70-3
Dibenzofuran	TRG		0.325	10	61-110	30	62-112	30	ug/L	132-64-9
Dicyclopentadiene	TRG		0.59	10	70-130	30	70-130	30	ug/L	77-73-6
Diethyl Phthalate	TRG		0.289	10	61-122	30	62-122	30	ug/L	84-66-2
Dimethoate	TRG		0.69	10	70-130	30	70-130	30	ug/L	60-51-5
Dimethyl Phthalate	TRG		0.254	10	69-110	30	64-119	30	ug/L	131-11-3
Di-n-butyl Phthalate	TRG		0.364	10	61-127	30	65-126	30	ug/L	84-74-2
Di-n-octyl Phthalate	TRG	CCC	0.626	10	57-129	30	60-131	30	ug/L	117-84-0
Dinoseb	TRG		0.42	10	70-130	30	70-130	30	ug/L	88-85-7
Diphenylamine	TRG		0.42	10	70-130	30	70-130	30	ug/L	122-39-4
Disulfoton	TRG		0.57	10	70-130	30	70-130	30	ug/L	298-04-4
Ethyl Methanesulfonate	TRG		0.28	10	70-130	30	70-130	30	ug/L	62-50-0
Famphur	TRG		0.27	10	70-130	30	70-130	30	ug/L	52-85-7
Fluoranthene	TRG	CCC	0.652	10	56-123	30	60-121	30	ug/L	206-44-0
Fluorene	TRG		0.323	10	58-112	30	59-112	30	ug/L	86-73-7
Hexachlorobenzene	TRG		0.628	10	56-115	30	63-117	30	ug/L	118-74-1
Hexachlorobutadiene	TRG	CCC	0.291	10	44-94	30	54-99	30	ug/L	87-68-3
Hexachlorocyclopentadiene	TRG	SPCC	1.21	10	10-58	30	10-60	30	ug/L	77-47-4

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Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
Hexachloroethane	TRG		0.289	10	40-101	30	50-100	30	ug/L	67-72-1
Hexachlorophene	TRG		44	150	70-130	30	70-130	30	ug/L	70-30-4
Hexachloropropene	TRG		0.19	10	70-130	30	70-130	30	ug/L	1888-71-7
Indeno(1,2,3-cd)pyrene	TRG		0.684	10	62-113	30	68-116	30	ug/L	193-39-5
Isodrin	TRG		0.36	10	70-130	30	70-130	30	ug/L	465-73-6
Isophorone	TRG		0.246	10	60-99	30	57-106	30	ug/L	78-59-1
Isosafrole	TRG		0.48	10	70-130	30	70-130	30	ug/L	120-58-1
Kepone	TRG		4.1	20	70-130	30	70-130	30	ug/L	143-50-0
Malathion	TRG		1	25	70-130	30	70-130	30	ug/L	121-75-5
Methapyrilene	TRG		9.3	100	70-130	30	70-130	30	ug/L	91-80-5
Methyl Methanesulfonate	TRG		0.31	10	70-130	30	70-130	30	ug/L	66-27-3
Methyl Parathion	TRG		0.51	10	70-130	30	70-130	30	ug/L	298-00-0
Naphthalene	TRG		0.365	10	51-99	30	51-106	30	ug/L	91-20-3
Nitrobenzene	TRG		0.567	10	31-141	30	56-108	30	ug/L	98-95-3
N-Nitrosodiethylamine	TRG		0.41	10	70-130	30	70-130	30	ug/L	55-18-5
N-Nitrosodimethylamine	TRG		0.479	25	47-100	30	53-109	30	ug/L	62-75-9
N-Nitrosodi-n-butylamine	TRG		0.57	10	70-130	30	64-105	30	ug/L	924-16-3
N-Nitrosodiphenylamine	TRG	CCC	0.48	10	10-135	30	58-121	30	ug/L	86-30-6
N-Nitrosomethylethylamine	TRG		4.6	25	70-130	30	70-130	30	ug/L	10595-95-6
N-Nitrosomorpholine	TRG		0.25	10	70-130	30	70-130	30	ug/L	59-89-2
N-Nitrosopiperidine	TRG		0.32	10	70-130	30	70-130	30	ug/L	100-75-4
N-Nitrosopyrrolidine	TRG		0.39	10	70-130	30	70-130	30	ug/L	930-55-2
O,O,O-Triethyl Phosphorothio	TRG		0.37	10	70-130	30	70-130	30	ug/L	126-68-1
o-Toluidine	TRG		1.4	10	70-130	30	70-130	30	ug/L	95-53-4
Parathion	TRG		0.51	10	70-130	30	70-130	30	ug/L	56-38-2
p-Dimethylaminoazobenzene	TRG		0.31	10	70-130	30	70-130	30	ug/L	60-11-7
Pentachlorobenzene	TRG		0.31	10	70-130	30	70-130	30	ug/L	608-93-5
Pentachloroethane	TRG		0.28	25	70-130	30	70-130	30	ug/L	76-01-7
Pentachloronitrobenzene	TRG		0.23	50	70-130	30	70-130	30	ug/L	82-68-8
PGMEA	TRG		0.96	10	70-130	30	70-130	30	ug/L	108-65-6
Phenacetin	TRG		0.42	50	70-130	30	70-130	30	ug/L	62-44-2
Phenanthrene	TRG		0.482	10	61-111	30	64-116	30	ug/L	85-01-8
Phorate	TRG		0.36	10	70-130	30	70-130	30	ug/L	298-02-2
p-Phenylenediamine	TRG		19	100	70-130	30	70-130	30	ug/L	106-50-3
Pronamide	TRG		0.41	10	70-130	30	70-130	30	ug/L	23950-58-5

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Analyte Name	Parm Type	Type	MDL	MRL	Surr/MS Limits	DMS RPD	LCS Limits	DLCS RPD	Units	Analyte ID
Pyridine	TRG		7.50	25	10-142	30	10-142	30	ug/L	110-86-1
Quinoline	TRG		10	10	70-130	30	70-130	30	ug/L	91-22-5
Safrole	TRG		0.36	10	70-130	30	70-130	30	ug/L	94-59-7
Sulfotep	TRG		0.26	10	70-130	30	70-130	30	ug/L	3689-24-5
Thionazin	TRG		0.71	25	70-130	30	70-130	30	ug/L	297-97-2

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Vista
Analytical Laboratory

QUALITY MANUAL

REVISION 12

EFFECTIVE DATE: OCTOBER 15, 2009

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Vista Analytical Laboratory

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FOREWORD

The Quality Manual (QM) describes the Quality System implemented at Vista Analytical Laboratory in El Dorado Hills, California. The policies and procedures outlined in this QM are designed and developed to comply with the established NELAC Standards. It is the intent of Vista to meet or exceed the Quality Assurance/Quality Control (QA/QC) requirements set by ISO 17025, NELAC, the USEPA or other appropriate governmental or private entities to assure that all analytical data generated are scientifically valid, defensible, comparable, and of known acceptable precision and accuracy.

The QM shall be amended to reflect any changes to Vista's capability, location or Quality System. The Quality Assurance Manager is responsible for the maintenance and annual review of the QM.

1. INTRODUCTION

Vista Analytical Laboratory in El Dorado Hills, CA was established in 1990 and is a privately owned California corporation. Vista provides state-of-the-art mass spectrometry services to chemical manufacturers, environmental engineering firms, and the pulp and paper industry as well other industrial and governmental clients. Vista operates with the intent of providing data of the highest quality with responsive service in a short turnaround time.

Vista has an expanding national and international client base attributable to its reliable reputation in performing difficult trace level analyses. Vista's expertise lies in the analysis of semivolatile organic compounds such as Dioxin/Furans (PCDD/PCDF), Polynuclear Aromatic Hydrocarbons (PAHs), Polychlorinated Biphenyls (PCBs), Polychlorinated Naphthalenes (PCNs), Hexachlorobenzene (HCB), Hexachlorocyclopentadiene (HCP), and Polybrominated Diphenyl Ethers (PBDEs).

1.1. Policy

It is the policy of Vista to meet the specific quality requirements and to satisfy the needs of the client, the regulatory authorities or organizations providing recognition throughout data generation and process operations. A Quality System has been established to achieve this policy. The system encompasses all of the applicable elements of the established NELAC Standards. It is Vista's intent to provide full compliance with this Quality System throughout all phases of client services and to ensure that only an acceptable final product is presented to the client.

1.1.1. It is Management's responsibility to instill a commitment of the quality standards throughout the company, and to ensure each employee has a clear understanding of the Quality System.

- Quality is the responsibility of all Vista employees.
- All Vista employees must comply with all QA/QC procedures as it pertains to their function.
- All employees shall be accountable for the quality of their individual assignments and functional responsibilities.
- Employees shall be responsible for reporting any non-conformance to Management or the QA Manager.
- The laboratory shall have sufficient personnel with necessary education training, technical knowledge and experience for the assigned positions.

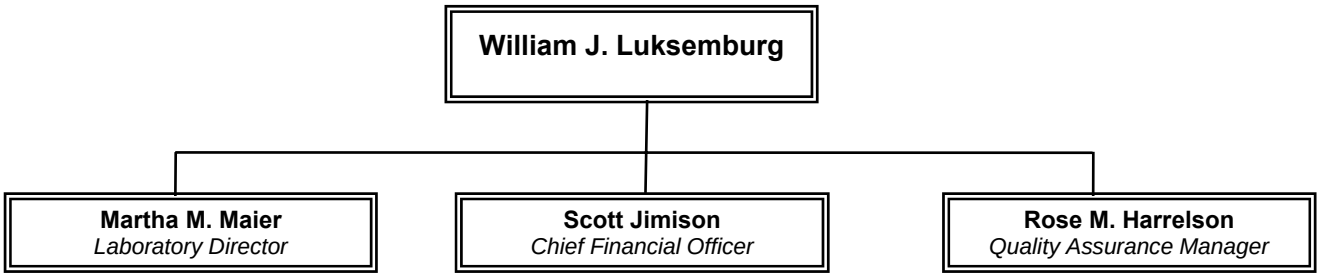
1.1.2. Management is responsible to ensure personnel are free from any commercial, financial, and other undue pressures, which might affect the quality of work.

1.1.3. All Vista employees shall be confident in their independence of judgment and maintain integrity at all times.

2. ORGANIZATION AND FACILITIES

The management staff of Vista consists of a Laboratory President, a Chief Financial Officer, the Laboratory Director, and QA Manager.

The organization and management structure of Vista Analytical Laboratory is shown in the following organizational chart.



2.1. Management Responsibilities

2.1.1. President

The President is responsible for the management of financial/technical operations, as well as implementation of corporate goals, objectives and policies and review of laboratory operations. This includes directing the routine analysis and method development work and overseeing marketing of laboratory services. In addition, the President is responsible for overseeing the Quality Assurance Department and ensuring that the Quality System is in compliance with applicable regulations.

2.1.2. Chief Financial Officer

The Chief Financial Officer is responsible for all financial and facility services. The management of the facility includes overseeing building maintenance. The Chief Financial Officer supervises all administrative personnel.

2.1.3. Laboratory Director

The Laboratory Director manages the production scheduling and client management for the laboratory, is responsible for final review and interpretation of analytical data and final reports, and also serves as technical director. The Laboratory Director is also responsible for certifying the qualifications and training of the analysts. Should the Technical Director be absent for more than 15 consecutive calendar days, the Director of the Instrument

Laboratory shall be designated acting Technical Director. In the absence of the Technical Director for more than 65 calendar days, all laboratory Accrediting Bodies shall be notified in writing by the Quality Assurance Manager.

2.1.4. Quality Assurance Manager

The Quality Assurance Manager is responsible for managing the QA activities of the entire laboratory. The Quality Assurance Manager reports directly to the President of the laboratory. The Quality Assurance Manager serves as the focal point for QA/QC and is responsible for the oversight and/or review of quality control data. When QA oversight is necessary, the QA Manager functions must be independent from the laboratory operations. The QA Manager works with management to ensure that the Vista QM and associated SOPs are followed as written. QA Manager maintains a position that is free from outside influence in order to evaluate the data and perform all other QA Manager responsibilities objectively.

2.2. Approved signatories

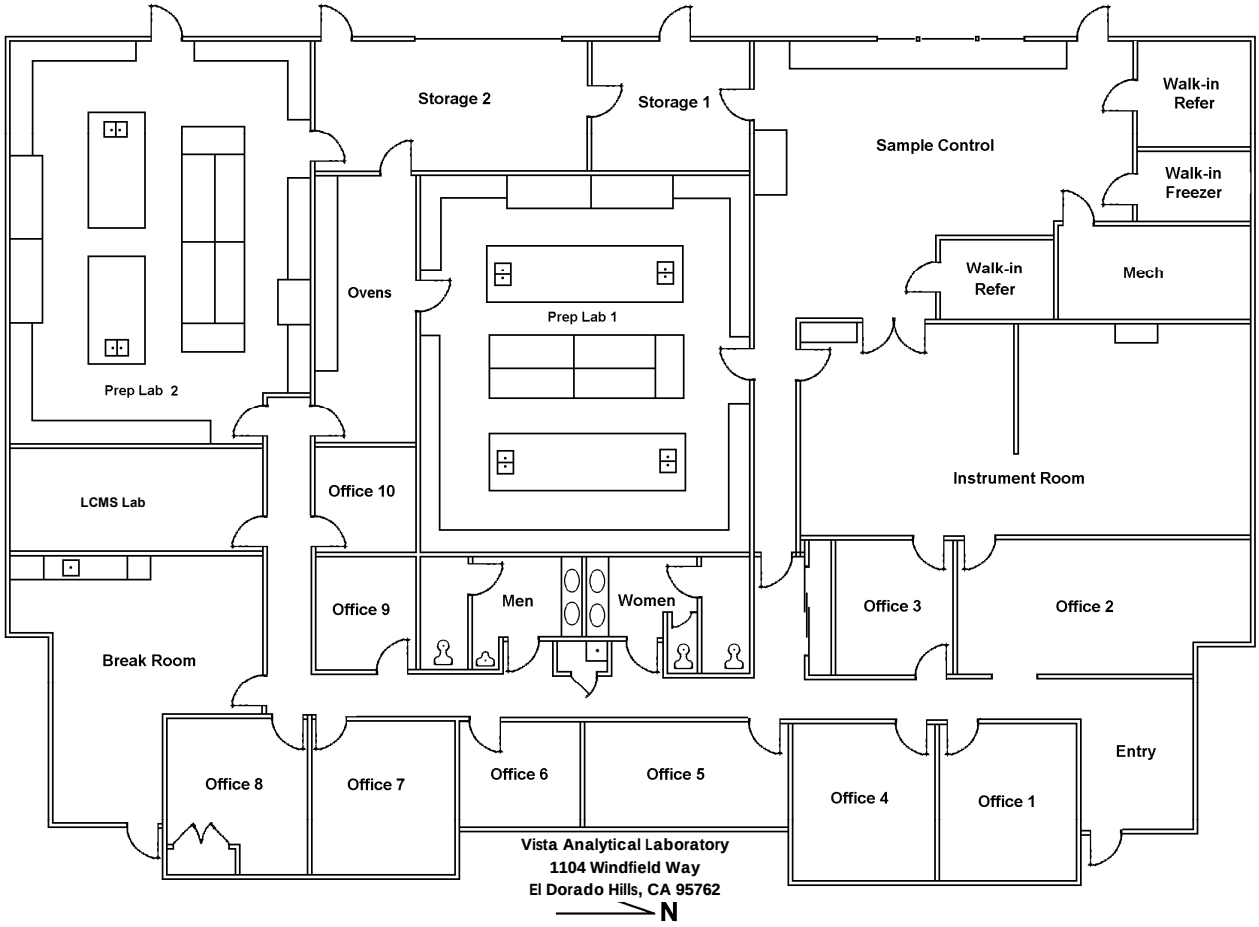
2.2.1. Approved signatories include the laboratory President, the Laboratory Director, the QA Manager and the Director of the Instrumentation Laboratory. These responsible parties are listed on the QM title page.

2.3. Facilities

2.3.1. Vista Analytical Laboratory operates from El Dorado Hills, CA. The facility consists of 9,000 square feet.

2.3.2. The facility has been constructed and maintained to ensure that results are not invalidated or do not adversely affect the required accuracy of measurement.

2.3.3. Layout – 1104 Windfield Way, El Dorado Hills, CA


Storage 2
Storage 1
Sample Control
Walk-in Refer
Walk-in Freezer
Ovens
Prep Lab 1
Prep Lab 2
Walk-in Refer
Mech
Instrument Room
LCMS Lab
Office 10
Office 9
Men
Women
Office 3
Office 2
Break Room
Office 8
Office 7
Office 6
Office 5
Office 4
Office 1
Entry
Vista Analytical Laboratory
1104 Windfield Way
El Dorado Hills, CA 95762
N

3. QUALITY SYSTEM

The Quality System applies to Vista Analytical Laboratory.

The company's Quality System is designed to comply with the applicable requirements of NELAC Standards and to satisfy the needs of the client or organization providing recognition. All policies, systems, and procedures are documented to assure quality of the data. Personnel shall familiarize themselves with quality documentation and implement the policies and procedures in their work.

Senior Management will review the effectiveness and suitability of the Quality System at least annually. The reviews shall address issues that impact quality. The results of the reviews shall be used to design and implement improvements to the system. The reviews include reports from management and supervisory personnel, recent internal audits, external audits, proficiency testing, client feedback, and corrective action reports. The QA Manager will maintain records of the review meeting, findings, and corrective actions.

3.1. Quality Documents

3.1.1. The Quality System is outlined and documented in the Quality Manual and supporting quality documents. The documented quality system assures that services provided to clients comply with specified quality criteria.

3.1.2. The Quality Manual contains Quality Policies covering the applicable requirements of the NELAC quality standard.

3.1.3. Program specific quality criteria are specified in the Quality Assurance Program Plan (QAPP).

3.1.4. Procedural activities that affect quality are described in more detail in the Standard Operating Procedures (SOPs).

3.2. Use of Quality Documents

3.2.1. Management will review and approve all quality documents prior to issuance. All quality documentation shall be communicated to, understood by, available to, and implemented by the appropriate personnel.

3.2.2. A QAPP or other project-specific requirements submitted by the client will be reviewed to determine whether they are within the scope of the Analytical Procedures. Any discrepancies will be discussed with the client and documented prior to commencement of the project.

3.2.3. The Quality Manual will be understood and implemented throughout the company. The QAPP and SOPs will be understood and implemented throughout applicable operations.

3.2.4. Quality documents shall be periodically reviewed to ensure continuing suitability and compliance with applicable requirements. The Quality System will be reviewed on an ongoing basis and revised as needed to ensure that it effectively encompasses the company's quality criteria. The QA Manager will maintain the Quality Manual. Revisions to the Quality Manual may be made by replacing individual policies or the entire manual.

3.2.5. Any departures from policies or planned activities that affect quality will be approved by management prior to occurrence.

3.2.6. The QAPP will be maintained by the designated responsible manager, or the QA Manager. Revision may be made to individual sections of the entire plan.

3.2.7. Standard Operating Procedures will be maintained as designated in the specific SOP with revisions being made on an as needed basis.

3.3. Document Control

Standard Operating Procedures (SOPs) or any documents that specify quality requirements or otherwise affect quality are Controlled Documents. All controlled documents will be prepared, issued and revised in accordance with the applicable SOPs. The SOPs are presented in Table 3.1.

3.3.1. Procedures are established to control and maintain the issue, distribution, and revisions of all controlled documentation.

3.3.2. Appropriate documents shall be made available at all locations where operations essential to the effective functioning of the laboratory are performed.

3.3.3. Complete and current copies of the controlled documents shall be made available upon issuance, and obsolete copies will be removed from all points of issue or use. The controlled document copies will be stamped, in red, as an "Official QA Copy".

3.3.4. All original controlled documents are archived by QA Manager.

3.3.5. A master list will be used to ensure that the correct revision of each SOP is available for use, and that obsolete revisions are removed from service. Each controlled document has an associated revision number and effective date to enable tracking of current revisions.

3.3.6. Document changes are reviewed and approved by the appropriate personnel.

3.3.7. Documents are periodically reviewed and, where necessary, revised to ensure continuing suitability and compliance with applicable requirements. The Quality Manual (QM) will be revised as needed and reviewed annually.

3.3.8. QA Manager will maintain records of revisions for Controlled Documents and the QAPP.

Table 3.1 List of Standard Operating Procedures	
SOP #	**Title**
1	Laboratory Security
2	Laboratory Audits
3	Standard Operating Procedures
5	Data Collection, Reporting, and Archival
6	Corrective Actions
7	Control Charts
8	Method Detection Limits
9	Manual integrations
10	Instrument Maintenance Logbooks and Schedule
11	Laboratory Support instrument Calibration
12	Sample Receiving and Sample Control Procedures
13	Consignment Tracking
14	Bottle Order Preparation
15	Reagents and Standards – Preparation, Handling, and Documentation
16	Sample Preparation and Analysis of PUF Samples for PCDD/PCDFs by EPA Method TO-9A
17	Preparation and Shipping of Air Sampling Media for in Field Use
18	Sample Preparation of MM5 Train for Analysis of PAHs by Method CARB 429
19	Sample Preparation of MM5 Train for Analysis of PCBs and PCDD/PCDFs by Methods CARB 428 and Method 23 or Method 0023A
20	Sample Preparation and Analysis of Sampling Trains and PUFs and PUF/XAD2 for Analysis of PCBs by Modified Method 1668
21	Sample Preparation and Analysis of Sampling Trains and PUFs and PUF/XAD for Analysis of PBDEs by Modified Method 1614 (Draft)
22	Preparation of Surface Wipes
23	Polychlorinated Dibenzo Dioxin/Furans by USEPA Method 8280A
24	Polychlorinated Dibenzo Dioxin/Furans by USEPA Method 8290
25	Tetrachlorodibenzodioxin in Aqueous Samples by Modified USEPA Method 613
26	Polychlorinated Dibenzo Dioxin/Furans by Method 1613B
28	Sample Analysis of HCB/B by Modified Method 1625B
29	Modified Method 8290 for the Analysis for PCDD/PCDFs, Coplanar, and mono-ortho PCBs in Human Serum or Blood
30	Polybrominated Dibenzo-Dioxin/Furans by Modified EPA Method 8290
31	Analysis of Polychlorinated Biphenyls (PCBs) by Method 1668
32	Analysis of Various Matrices for Polybrominated Diphenyl Ethers (PBDE) by EPA Method 1614
33	Analysis of Polychlorinated Naphthalenes (PCN) by Modified EPA Method 1668A

Table 3.1 List of Standard Operating Procedures	
SOP #	**Title**
34	Preparation And Analysis Of Human Serum/Blood Using Modified Method 8290 For PCDD/PCDFs And Modified Method 1668A For Coplanar/Mono-Ortho PCBs
35	Glassware Preparation
36	Sample Preparation of MM5 Train for Analysis of PCDDs/PCDFs/PCBs/PAHs by EPA Method 0023A/CARB 428/CARB 429
37	NCASI 551
38	Computer Systems

3.4. Quality Assurance Objectives and Quality Control Procedures

Quality assurance objectives employed at Vista provide routine mechanisms of ongoing control and evaluation of measurement data quality. The quality control (QC) procedures routinely followed evaluate method performance in terms of accuracy and criteria specified by the method or protocol.

3.4.1. Accuracy and precision

Accuracy and precision objectives for HRMS analyses are listed in Table 3.2. Vista's internal quality control procedures include the analysis of method blanks, duplicate samples, laboratory control samples, and matrix spikes.

3.4.2. Definitions

3.4.2.1. **Accuracy:** Accuracy is the nearness of a measurement to the true or theoretical value. Accuracy is assessed by determining recoveries from laboratory control samples, matrix spikes or by comparing values obtained from reference samples.

3.4.2.2. **Analytical Batch:** An analytical batch is a set of samples of the same matrix that are analyzed together using the same method, reagents, and standards. QC results associated with individual analytical batches such as ongoing precision and recovery samples, laboratory control samples, method blanks, matrix spike samples, and duplicate samples are evaluated together to assess data quality. Each batch will be assigned a unique batch number, which will be used to associate sample results with quality control data. All samples associated with a particular batch must be extracted on the same day.

3.4.2.3. **Clean-up Recovery Standard:** A clean-up recovery standard is a reference substance that is an isotopically labeled analyte that is added to the sample extract prior to any clean-up procedures. This standard is used to quantitatively assess losses occurring throughout the clean-up process.

3.4.2.4. **Control/Warning Limits:** Warning and control limits are limits used in laboratory control charts tracking average recovery and relative percent difference. For a Means Chart, typical warning and control levels are ± 2 and ± 3 standard deviations (s) from the central line (i.e., average mean recovery),

respectively. Similarly, the warning and control limits for a RPD Chart are usually set at + 2s and + 3s above the mean RPD, respectively.

3.4.2.5. **Detection Limit (DL):** The lowest concentration of an analyte within an environmental matrix that a method or equipment can detect.

3.4.2.6. **Duplicate Sample (DS):** Duplicate samples are two separate aliquots taken from the same source. Duplicate samples are analyzed independently to assess laboratory precision.

3.4.2.7. **Estimated Maximum Possible Concentration (EMPC):** The EMPC is calculated when the response has a S/N in excess of 2.5, but the ion abundance criteria are not met.

3.4.2.8. **Internal Standards (IS):** An internal standard is a reference substance that is an isotopically labeled analyte which is added to the sample prior to extraction and used in the quantitation and identification of native analytes.

3.4.2.9. **Laboratory Control Sample:** A laboratory control sample is prepared by adding a known quantity of native standards to an interferant free matrix.

3.4.2.10. **Method Blank (MB):** A method blank is a sand, XAD or deionized water preparation that is free of native analyte or interferants that has been prepared and analyzed using the same procedures followed for the rest of the analytical batch. The method blank is used to determine the level of background laboratory contamination, if present.

3.4.2.11. **Method Detection Limit:** The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero in the matrix tested. MDLs follow 40 CFR Part 136.

3.4.2.12. **Method Quantitation Limit (MQL):** The method quantitation limit is defined as the quantity of native analyte that corresponds to the lowest concentration of the calibration curve. The Method Quantitation Limit is also known as the Reporting Limit.

3.4.2.13. **Matrix Spike (MS/MSD):** A matrix spike sample is prepared by adding a known quantity of native standards to a sample matrix prior to extraction. Matrix spike concentration levels will vary according to the matrix encountered and study objectives.

3.4.2.14. **Native Standard:** A native standard is a reference substance that is a non-isotopically labeled analyte. Native standards are used in conjunction with internal standards to determine response factors and quantitatively assess accuracy.

3.4.2.15. **Ongoing Precision and Recovery (OPR):** A laboratory blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the specified limits.

3.4.2.16. **Precision:** Precision is the agreement between a set of replicate measurements. RPD is used as the principal measure of precision and is based on the analysis of duplicate quality control samples.

3.4.2.17. **Pre-Spike Standards:** A pre-spike standard is an isotopically labeled analyte that is spiked into an MM5 resin cartridge or PUF prior to sampling. The recoveries of pre-spike standards provide a measure of the air sampling efficiency for native analytes.

3.4.2.18. **Quality Control Sample:** Quality control samples are analyzed to access the various aspects of the analytical process in order to monitor quality within the laboratory. The most frequently used QC samples are method blanks, duplicates, matrix spikes, matrix spike duplicates and LCS pairs.

3.4.2.19. **Recovery Standard:** A recovery standard is a reference substance that is an isotopically labeled analyte which is added to the sample extract after clean-up and prior to injection. This standard is used to quantitatively assess the absolute recoveries of the internal and clean-up recovery standards.

3.4.2.20. **Resin QC:** A resin QC is an XAD-2 preparation that is analyzed to assess possible background contamination originating from the resin.

3.4.2.21. **Reporting Limit:** See Method Quantitation Limit.

3.4.2.22. **Signal to Noise Ratio:** Dimensionless measure of the relative strength of an analytic signal to the average strength of background instrument noise.

3.4.3. Calculations

3.4.3.1. Percent Recovery (%R): Percent recovery is a measure of accuracy and is calculated according to the following expression:

$$\%R = \frac{(Amount\ \ Found)}{(Amount\ \ Spiked)}\ X\ 100$$

3.4.3.2. Relative Percent Difference (RPD): Percent Recovery (%R) from duplicate LCS or matrix spike analyses are used to calculate RPD using the following expression:

$$RPD\ = \frac{|\%\ R_1 - \%\ R_2|}{\left(\frac{(\%\ R_1 + \%\ R_2)}{2}\right)} \times 100$$

3.4.3.3. Similarly, the RPD for duplicate sample analyses, is calculated using the sample concentration (C), as follows:

$$RPD_{DS} = \frac{|C_1 - C_2|}{\frac{(C_1 + C_2)}{2}} \times 100$$

3.4.3.4. Relative Standard Deviation (RSD): Also known as the coefficient of variation.

$$RSD = \frac{SD}{Mean} \times 100$$

3.4.4. Quality Control Procedures

3.4.4.1. Method Blanks:

A method blank is run with each analytical batch or 20 samples (whichever is less) per method and matrix type.

For any method involving the determination of native 2,3,7,8-substituted isomers except hepta- or octa-PCDD/PCDF, the levels measured in the method blank must be less than the MQL, or ten times lower than the concentration found in samples within the analytical batch, unless otherwise mandated by project or client requirements.

All samples within an analytical batch are re-extracted and analyzed if the method blank associated with that batch does not meet internal standard recovery criteria or contamination limits specified above. Otherwise, the data is qualified appropriately.

3.4.4.2. Ongoing Precision and Recovery/Laboratory Control Samples

A single OPR or a pair of LCS is analyzed with every batch of clients' samples.

All samples within an analytical batch are re-extracted and analyzed if the native or internal standard recoveries from the LCS do not fall within the acceptable control range for accuracy or if the RPD falls outside the specified precision limit established by the method. If the OPR/LCS is not within the acceptable control range and the analytes are not detected in the samples, then it is at the discretion of the Laboratory Director to re-extract the QC sample or qualify the data that is reported.

3.4.4.3. Matrix Spike and Duplicate Sample Analyses

An MS, MS/MSD, or duplicates are analyzed upon client request, method requirements, or at the discretion of the Laboratory Director.

If the RPD from duplicate samples exceeds 25% or the MS/MSD exceeds 20%, corrective action will be taken as directed in the method, unless there is demonstrated matrix effect.

3.4.5. Quality Control Charts

Quality control data are calculated as needed by the QA Manager and distributed to the Laboratory Director for review if necessary. A set of current QC control charts is maintained in QA Manager.

Original copies of the QC charts and any associated tabular data are stored in QA Manager. QC control charts are available upon written request of clients or regulatory agencies or may be reviewed during facility audits.

Table 3.2 Accuracy and Precision Objectives

DATA ACCEPTANCE/REJECTION CRITERIA **Precision/Accuracy and QC Requirements**					
METHOD	**Method Blank**	**Internal Standard Recovery Limits**	**OPR Recovery Limits**	**Duplicate Sample Analysis**	**MS/MSD**
EPA 8280/ 8280A	One/extraction batch ≤ML, report in ng/g or ng/L ≤5% regulatory limit or amount in sample	25-150%	70-130%	By client request RPD≤25%	By client request RPD≤20%
EPA 8290/0023A	One/extraction batch Run between calibration std and 1st sample	40-135%	70-130%	By client request RPD≤25%	By client request RPD≤20%
EPA 23	One/extraction batch Run between calibration std and 1st sample	Surrogate 70-130% IS Tetra-Hexa 40-130% Hepta-Octa 25-130%	70-130%	Not applicable	Not applicable
T0-9A	One/extraction batch Run between calibration std and 1st sample	Surrogate 70-130% IS Tetra-Hexa 50-120% Hepta-Octa 40-120%	70-130%	Not applicable	Not applicable
EPA 613	One/extraction batch	25-150%	70-130%	By client request RPD≤25%	10% of samples or 1/month RPD≤20%

Table 3.2 Accuracy and Precision Objectives

DATA ACCEPTANCE/REJECTION CRITERIA Precision/Accuracy and QC Requirements					
METHOD	**Method Blank**	**Internal Standard Recovery Limits**	**OPR Recovery Limits**	**Duplicate Sample Analysis**	**MS/MSD**
EPA 1613A EPA 1613B	One/extraction batch after OPR Must be ≤ 1/3 of minimum level (10 pg/L or regulatory compliance level whichever is greater).	Tables 7 and Table 7A	See Tables 6 and 6A	By client request RPD≤25%	By client request RPD≤20%
EPA 1668	One/extraction batch ≤ 10X amount in sample	Samples 25-150% OPR Recovery per SOP 31	OPR Recovery per SOP 31	By client request RPD≤25%	By client request RPD≤20%
NCASI 551	Method Blank IS & RS Recovery >40%	40-120% or S/N > 10:1 if %R is >20% "H" Qualifier	70-130%	By client request RPD≤25%	By client request RPD≤20%
CARB 428 PCB's	One/extraction batch ≤ 10X amount in sample	40-120% or S/N >10:1	60-140%	Not applicable	Not applicable
CARB 428 D/F	One/extraction batch Must be ≤ ML	Surrogates= 60-140% IS= 40-120% or S/N >10:1	60-140%	Not applicable	Not applicable
CARB 429	One/extraction batch ≤ 5% amount in sample	50-150% or S/N > 10:1 "H" Qualifier	Field Spikes 50-150%	By client request RPD≤25%	Not applicable
EPA 1614 (DRAFT)	Method Blank ≤ML; ≤1/3 regulatory limit or amount in sample	Tetra-Hepta: 30-140% Tetra-Hepta: 25-150% Samples Deca: 20-200%	Tetra-Hepta: 50-150% Deca: 40-200%	By client request RPD≤25%	By client request RPD≤20%

Table 3.2 Accuracy and Precision Objectives

DATA ACCEPTANCE/REJECTION CRITERIA Precision/Accuracy and QC Requirements					
METHOD	**Method Blank**	**Internal Standard Recovery Limits**	**OPR Recovery Limits**	**Duplicate Sample Analysis**	**MS/MSD**
Mod 1668A (PCN)	One/extraction batch	30-140% 25-150% Samples	50-150%	By client request RPD≤25%	By client request RPD≤20%
Method 1625	One/extraction batch	Method Table 8	Method Table 8	By client request RPD≤25%	By client request RPD≤20%

4. PURCHASING

4.1. Quality Materials and Services

Materials and services that affect the quality of the company's services will be designated as quality material and services. Purchases shall be made only from approved suppliers (based on historical experience or quality certifications).

4.2. Control of Quality Materials and Services

Quality Materials and Services and, where appropriate, potential suppliers' Quality Systems, shall be evaluated to ensure that specified quality requirements are met. Any purchased equipment and consumable materials, whenever possible, shall be inspected, calibrated, or otherwise verified as complying with any standard specifications relevant to the calibrations or tests concerned prior to use. Records of actions taken to check compliance shall be maintained.

4.3. Procurement Documents

Procurement documents will clearly specify all information and requirements necessary to ensure that the correct materials and services are purchased and received. Any discrepancies between request and contracts shall be resolved before any work commences. Request and contracts shall be reviewed to determine the effect of financial, legal and time schedule aspects. Any amendments to the request or contract after work has commenced shall require another review process.

5. SAMPLE CONTROL

Samples and other material received from clients shall be handled and maintained in accordance with laboratory SOPs.

5.1. Receipt of Materials

5.1.1. Samples and materials received from clients, and any other materials received from an outside source in the regular course of business, will be inspected upon receipt to insure that they meet specified quality requirements. All conditions, including any abnormalities or departures from standard conditions, shall be recorded according to SOPs.

5.1.2. Immediately after inspection samples will be logged into the laboratory computer system. A unique laboratory identification number is assigned to each sample at the time of login. This unique laboratory identification allows the sample to be controlled and tracked during storage, handling, and disposal.

5.1.3. Other materials will be properly identified upon verification that they meet specified quality requirements.

5.2. Storage, Handling, and Disposal

5.2.1. Samples and materials received from clients will be stored and handled in a manner that ensures the integrity and quality characteristics are maintained.

5.2.1.1. All samples are stored away from all standards; reagents, food, or any other potentially contaminating sources in such a manner as to prevent cross contamination.

5.2.2. Samples, sample extracts, and any other sample preparation fractions are stored according to the conditions specified by preservation protocols or according to the appropriate test method.

5.2.3. Samples are stored for a minimum of 90 days. If the client provides any relevant instructions regarding sample storage, then the samples are stored according to the client's request.

5.2.4. Samples will be disposed of in a manner that:

- Protects the environment
- Complies with applicable regulatory requirements

- Complies with any project specific requirements

5.2.5. Excess materials will either be returned to the client, or disposed of in accordance with the applicable SOPs.

5.2.6. Access to laboratories and sample storage facilities will be restricted to authorized personnel to further ensure that sample integrity is maintained.

5.2.7. Ambient conditions will be monitored in storage facilities and laboratories where control of those conditions is necessary to maintain the integrity of the sample.

5.3. Notification of Problems

Clients or suppliers will be notified if the integrity of their samples or materials is jeopardized either upon receipt or while in the possession of the company.

5.4. Records

Records of all procedures to which a sample is subjected to while in the laboratory shall be maintained. Chain of custody records shall establish an intact, continuous record of the physical possession, storage, and disposal of all samples.

Table 5 Sample Containers, Preservatives and Maximum Holding Times

Method	Sample Type	Maximum Holding Times	Container Type	Preservation
EPA Method 8280	Aqueous Solid	Extraction: 30 days (1) Analysis: 45 days (2)	Amber Glass Glass Container	4°C 4 °C
EPA Method 8290	Aqueous Solid Fish/Tissue	Extraction: 30 days (1) Analysis: 45 days (2) Tissues: Extraction: 30 days (1) Analysis: 45 days (1)	Amber Glass Glass Container Glass Container	4 °C dark 4 °C -20 °C
EPA Method 1668	Aqueous Solid Fish/Tissue	Extraction: 1 year (1) Analysis: 1 year (2)	AGB AGJ AGJ	0 – 4 °C (3,6) dark < 4 °C dark (7) < -10 °C dark (8) < 4 °C dark (7) < -10 °C dark (8)
EPA Methods 1613A & 1613B	Aqueous Solid Fish/Tissue	Extraction: 1 year (1) Analysis: 1 year (2)	AGB AGB AGJ	0 – 4 °C (3) dark < 4 °C dark (7) < -10 °C dark (8) < 4 °C dark (7) < -10 °C dark (8)
EPA Method 613	Aqueous	Extraction: 7 days (1) Analysis: 40 days (2)	AGB	4 °C (3) dark
EPA Method 513	Aqueous	Extraction: 90 days (1) Analysis: 40 days (2)	AGB	Ambient dark
EPA Method 23	MM5 Train	Extraction: 30 days (1) Analysis: 45 days (2) Trap Prep: 30 days	Train and/or AGB	Adsorbents on ice (7)
EPA Method T0-9A (4)	PUF	Extraction: 7 days (1) Analysis: 40 days (2) PUF Prep: 30 days		< 4 °C

Table 5 Sample Containers, Preservatives and Maximum Holding Times

Method	Sample Type	Maximum Holding Times	Container Type	Preservation
CARB Method 428 (4)	MM5 Train	Extraction: 30 days (1) Analysis: 45 days (2) Trap Prep: 30 days	Train and/or AGB	0 – 4 °C dark (5)
CARB Method 429	MM5 Train	Extraction: 21 days (1) Analysis: 40 days (2) Resin QC Date: 21 days	Train and/or AGB	4 °C dark
NCASI 551 (4)	All Samples			4 °C
EPA Method 1614 (Draft)	Aqueous (3) Solid Fish/Tissue	Extraction: 1 year (1) Analysis: 1 year (2)	AGB AGJ AGJ	0 – 4 °C (3) dark < 6 °C dark < -10 °C dark < 6 °C dark < -10 °C dark
PCN	Aqueous Solid Fish/Tissue	Extraction: 1 year (1) Analysis: 1 year (2)	AGB AGJ AGJ	0 – 4 °C (3) dark < -10 °C dark < -10 °C dark < -10 °C dark
EPA Method 1625	All samples	Extraction: 7 days (1) Analysis: 40 days (2)	Amber Glass Containers	0 – 4 °C (3) dark

(1) From collection
(2) From extraction
(3) If residual chlorine is present sodium thiosulfate is added as per the method
(4) Holding times set by Vista Analytical Laboratory
(5) Recommended by Vista Analytical Laboratory
(6) Adjust sample to pH 2-3 with sulfuric acid
(7) From collection until laboratory receipt
(8) Storage at laboratory

6. TRACEABILITY OF MATERIALS

Procedures for identifying, controlling, and tracking items purchased from vendors, items developed in-house, samples received from clients, and client reports are detailed in SOPs.

Purchased materials and supplies will be checked to confirm that they meet quality specifications.

6.1. Verification of Items Developed In-house

6.1.1. Items developed in-house such as computer programs, equipment, and procedures, will be tested to verify that they meet the intended objectives. Test records will be maintained so that client reports can be traced to specific items.

6.2. Control of Laboratory Samples

6.2.1. Each sample will be assigned a unique laboratory ID number that will be used to track the sample as it is processed through the laboratory. This unique ID number is also used to associate the analytical results with the sample.

6.2.2. Samples will be batched for analysis. Each batch will be assigned a unique batch number that will be used to associate sample results with quality control data.

6.3. Standards and Reagents Traceability

6.3.1. Documented procedures shall exist for the purchase, reception, and storage of consumable materials used for the technical operations within the laboratory. Certificate of Analysis records for all standards shall be retained by QA Manager. Reagent and standard preparation documentation shall indicate traceability to purchased stock or neat compounds, reference to method of preparation, date of preparation, expiration date, and preparer's initials.

6.4. Quality Control Records

6.4.1. Records will be maintained to trace calibration standards and instrument calibration data to NIST or USEPA standards as appropriate. If NIST or USEPA standards are not available other standards will be used which are acceptable to specific project requirements.

6.4.2. Each instrument will be assigned a unique ID number. Records will be maintained to document the performance and maintenance of each instrument.

6.4.3. Records will be maintained to identify the individuals responsible for preparing calibration standards, analyzing samples, and reviewing analytical data.

6.4.4. Quality control records will be maintained to demonstrate that individual test procedures have been verified. Individual analytical results will be traceable to these quality control records.

6.5. Certificates of Analysis

6.5.1. All client reports and certificate of Analysis will be uniquely identified. Where appropriate, contract or purchase order numbers will be referenced on client reports. When requested, test procedures will be referenced on Certificates of Analysis.

6.6. Instruments and Equipment

6.6.1. All measuring operations and testing equipment effecting accuracy or validity of tests shall be calibrated and verified before being put into service and on a continuing basis.

7. PROCESS CONTROL

Analytical procedures and other processes that directly affect the quality of services will be conducted under controlled conditions using SOPs that are written at a level of detail appropriate to the complexity of the process.

Personnel will be properly trained before being given responsibility for an analytical procedure or other process that directly affects the quality of a service.

7.1. Instruments and Facilities

7.1.1. Analytical instruments will be maintained in a condition, which will ensure that they are able to meet specified operating conditions.

7.1.2. Laboratory facilities will be designed to meet specific operating conditions, and maintained in a condition, which will ensure that the operating conditions are consistently met.

7.1.3. Results of quality control checks will be recorded.

7.2. Performance Audits

7.2.1. The laboratory shall ensure the quality of results provided to clients by implementing checks to monitor the quality of the laboratories analytical activities.

7.2.1.1. Internal QC procedures.

7.2.1.2. Participation in proficiency testing or other interlaboratory comparisons.

7.2.1.3. Use of certified reference materials.

8. LABORATORY INSTRUMENTATION

All laboratory instrumentation and testing equipment used by the company will be maintained and calibrated in accordance with SOPs to verify proper operation. Table 8 details a list of current laboratory instrumentation for analysis.

Instrumentation will be placed into service dependent upon the capability of achieving the accuracy required and shall comply with relevant specifications to the instrument.

Authorized personnel shall operate laboratory instrumentation and testing equipment.

Instrumentation and equipment will be used in a manner that ensures that measurement uncertainty is known and consistent with specified quality requirements.

Methods and intervals of calibration specified for each instrument will be based on the individual operating characteristics of the instrument and the quality requirements of the analytical procedure.

8.1. Calibration Standards and Instruments

8.1.1. Calibration and verification procedures will use standards and instruments, whenever applicable, that are traceable to recognized national or international standards. Where traceability to national standards does not exist, the basis for the calibration will be documented.

8.1.2. Prior to use, laboratory instrumentation and testing equipment shall be calibrated and checked to establish that it meets the laboratory's specification requirements and complies with the relevant standard specifications.

8.1.3. Where applicable, reference standards and instrumentation will be checked periodically between calibration and verification procedures.

8.2. Calibration Records

8.2.1. Except for procedures requiring reanalysis, calibration prior to each analysis and previous calibration data will be reviewed when an instrument is out of calibration to determine whether or not the analytical results are acceptable.

8.2.2. Instruments that are unable to maintain calibration or not operating properly will be taken out of service. Instruments will not be placed back into service until they have been repaired and verified to be operating properly.

8.2.3. The records for each test or calibration shall contain sufficient information to indicate whether specified quality or process parameters are achieved. Each instrument will be assigned a unique ID number. Records will be maintained to document the performance and maintenance of each instrument.

Table 8 Instrument List

Name	ID	Acquired
Waters Autospec Ultima High Resolution Mass Spectrometer	VG-5	1998
Waters Autospec Ultima High Resolution Mass Spectrometer	VG-7	2001
Waters Autospec Ultima High Resolution Mass Spectrometer	VG-8	2001
Waters Autospec Ultima High Resolution Mass Spectrometer	VG-9	2004
Waters Autospec Ultima High Resolution Mass Spectrometer	VG-10	2008

9. QUALITY RECORDS

Procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records shall be in accordance with SOPs. Quality records shall include internal audits and management reviews as well as records of corrective actions and preventative actions. Technical records include original observations, calculations and derived data, calibration records and a copy of final report.

9.1. Documentation of Quality Records

9.1.1. Quality records will be generated in accordance with the specification of applicable procedures, programs, and contracts. These records will be maintained to demonstrate that specified quality requirements are met, and that the Quality System is functioning successfully.

9.1.2. Quality records of subcontractor services which affect the quality of the company's services will be required to meet the conditions of this section.

9.1.3. Documents will be clean and legible, and will reference back to the specific activities or procedures to which they apply.

9.2. Quality and Technical Records

9.2.1. Quality and technical records shall be conducted in accordance with SOPs.

9.2.2. History of all samples must be traceable and readily understood through the documentation.

9.2.3. Instruments may not be used in analytical procedures unless maintenance and calibration records indicate that specified quality requirements are achieved. The results of instrument maintenance and calibration inspections will be clearly identified either on the instrument or in maintenance and calibration documents

9.2.4. Work must pass specified quality requirements before it will be released to the succeeding step in the process or, finally, to the clients. The results of quality control checks on work processes will be documented in a manner that clearly indicates the status of the work to the responsible personnel.

9.2.5. Individuals authorized to conduct instrument maintenance and calibration procedures and quality control checks will be identified in the documentation.

9.3. Records Management and Storage

9.3.1. The laboratory shall retain on record all original observations, calculations and derived data, calibration records and a copy of report for a minimum of five years. This applies to both manual and electronic data.

- Individual records will be reviewed and noted if storage requirements longer than five years are required based on client, project or state specific regulations.

9.3.2. Records must provide sufficient information for an adequate audit trail that produces the same results for the sample analytical data. The sample from receipt to analysis must be readily understood through documentation.

9.3.3. All records shall be safely stored, held secure and in confidence to the clients. NELAP related records shall be available to the accrediting authority

9.3.4. All records shall be archived and protected from fire, theft, loss, and environmental deterioration. Any access to archived information shall be documented in the Archive Access Log

9.3.5. Quality documents will be stored in a manner that protects them from loss, damage, unauthorized alterations, and held in confidence to the client.

9.3.6. Documents will be indexed and filed in a manner that allows them to be readily retrieved. Clients will be provided access to records that document the quality of work done for them.

9.3.7. If the laboratory were to transfer ownership, the procedures on handling documents would remain the same. The transfer would ensure that the procedures in place prior to transfer show little significant change for client ease into transition.

9.3.8. If the laboratory were to go out of business, the laboratory would contact the client with the option of how they would like to proceed with their data. All data would be handled according to client or Vista approval for proper destruction or safekeeping.

10. CORRECTIVE ACTION

Nonconforming conditions are when any aspect of the quality system or technical operations does not conform to procedures or to client requirements. Nonconforming conditions have an adverse effect to the quality specifications and are handled in accordance with SOPs. If a nonconformance occurs, where necessary, the client shall be notified.

The applicable SOPs provide instructions for determining the root cause of nonconforming conditions, designing and implementing corrective action, and evaluating the effectiveness of the corrective action.

10.1. Causes of Nonconformance

Procedures will be implemented to determine the root cause of nonconformance conditions, and the corrective action will be designed to eliminate the root cause and prevent reoccurrence.

10.2. Corrective Action

10.2.1. Corrective actions are taken immediately, together with any decision about the acceptability of the nonconforming work. When nonconforming work is identified, the Laboratory Director and Quality Assurance Manger work together to investigate the source of the nonconformance. Either manager may halt work and withholding test reports, as necessary. Work shall not resume until the Laboratory Director has authorized the resumption of work.

10.2.2. Procedures that result in or allow nonconformance conditions will be revised. If necessary, new procedures will be written.

10.2.3. The revised or new procedures will be implemented and evaluated to ensure that the corrective action steps taken effectively eliminate the nonconformance conditions.

10.3. Documentation

10.3.1. Results of root cause analyses and corrective action steps implemented to eliminate nonconformance conditions will be documented and reported to appropriate levels of management in accordance with laboratory SOPs. Records of corrective actions are maintained by QA Manager.

11. REPORTS

Handling, storage, packaging, and, when applicable, delivery of client reports will be conducted in accordance with SOPs to ensure that specified quality requirements and confidentiality of the reports are maintained. The reports shall include all the information requested by the client or required by the method used. Reports may also include electronic data. Electronic data will follow the same criteria as reports. Any information not reported to the client shall be readily available in the laboratory.

11.1. Handling and Storage of Reports

11.1.1. Reports and files will be handled in a manner that ensures that client confidentiality is maintained, and that the reports are protected from loss, damage, or unauthorized alterations.

11.1.2. All reports and files will be coded for ease of identification and retrieval.

11.1.3. File cabinets and storage rooms will be designed to protect filed copies of reports from loss, damage, or unauthorized alterations.

11.1.4. Computer files will be backed up to electronic storage media and stored in a manner that protects them from loss, damage, or unauthorized personnel.

11.1.5. The condition of reports and files in storage will be periodically evaluated to ensure that there is no deterioration, and that the reports remain readily accessible to authorized personnel.

11.1.6. NELAP related records shall be made available to the accrediting authority, and shall be maintained for a minimum of five years.

- Individual records will be reviewed and noted if storage requirements longer than five years are required based on client, project or state specific regulations.

11.2. Packaging and Delivery of Reports

11.2.1. Client reports will be inspected prior to delivery to ensure that they meet specified quality requirements. Then the reports will be packaged for delivery to the client in a manner that ensures protection while in transit.

11.2.2. When required by specific contractual stipulations, the company will assume responsibility for protection of client reports while en route to the client.

11.3. Laboratory Report Format and Content

All laboratory reports shall include, at least, the following information:

11.3.1. A title, indicating the nature of the document (i.e. Test Report, Laboratory Results);

11.3.2. Name and address of the laboratory, location analysis was conducted if different from the address of the laboratory, and a phone number with name of a contact person;

11.3.3. Unique identification of the report and of each page, and the total number of pages. It must be clear that discrete pages are associated with a specific report, and that the report contains a specified number of pages;

11.3.4. NELAC accredited logo and a statement certifying that the report meets all requirements of NELAC and cannot be reproduced;

11.3.5. Name and address of client, where appropriate and project name if applicable;

11.3.6. Description and unambiguous identification of the tested sample including the client identification code;

11.3.7. Identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature;

11.3.8. Date of receipt of sample, date and time of sample collection, date(s) of performance test, and time of sample preparation and/or analysis if the required holding time for either activity is less than or equal to 72 hours;

11.3.9. Identification of the test method used, or unambiguous description of any non-standard method used;

11.3.10. If the laboratory collected the sample, reference to sampling procedure;

11.3.11. Any deviations from, additions to or exclusions from the test method, and any non-standard conditions that may have affected the quality of results, and including the use and definitions of data qualifiers

11.3.12. Measurements, examinations and derived results, supported by tables, graphs, sketches and photographs as appropriate, and any

failures identified; identify whether data are calculated on a dry weight or wet weight basis, identify the reporting units

11.3.13. A signature and title, or an equivalent electronic identification of the person(s) accepting responsibility for the content of the report, and date of issue;

11.3.14. Clear identification of all test data provided by outside sources, such as subcontracted laboratories, clients, etc.

- The original report from subcontracted laboratories should be included in the client laboratory report.

11.3.15. Reports shall, when required, include a statement of compliance/non-compliance with requirements and/or specifications, including identification or test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature.

11.3.16. Additional information, which may be required by specific methods, clients or groups of clients.

11.3.17. After issuance of the report, the report remains unchanged.

11.3.18. Any report that requires amending must clearly state that the report has been revised. The amended report must also meet the requirements set forth within Chapter 5 of the NELAC standards.

DATA QUALIFIERS & ABBREVIATIONS

B	This compound was also detected in the method blank.
C	Result was obtained from a confirmation analysis using either a DB-225 or SP-2331 GC column.
D	Dilution
E	The associated compound concentration exceeded the calibration range of the instrument.
H	The signal-to-noise ratio is greater than 10:1.
I	Chemical Interference
J	The amount detected is below the Lower Calibration Limit of the instrument.
P	The amount reported is the maximum possible concentration due to possible chlorinated diphenylether interference.
*	See Cover Letter
Conc.	Concentration
DL	Sample-specific estimated detection limit
MDL	The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero in the matrix tested.
EMPC	Estimated Maximum Possible Concentration
NA	Not applicable
RL	Reporting Limit – concentrations that correspond to low calibration point
ND	Not Detected
TEQ	Toxic Equivalency

Unless otherwise noted, solid sample results are reported in dry weight. Tissue samples are reported in wet weight.

12. PERFORMANCE AND SYSTEM AUDITS

Performance, System, and External audits are conducted to verify conformance with Vista's quality assurance program, to determine the effectiveness of the QA program, and to continually improve Vista's data quality.

12.1. System Audits

12.1.1. Internal audits (facility audits) of activities affecting the quality of the company's services will be conducted by the QA Manager on a regular schedule in accordance with laboratory SOPs. Internal audits are performed periodically and at least annually. The QA Manager is trained and qualified as an auditor who, wherever possible, is independent of the activities being audited. Internal audits verify that operations continue to comply with the requirements of the quality system and NELAC standards.

12.1.2. It is the responsibility of the QA Manager to plan and organize audits based on a predetermined schedule or as requested by management.

12.1.3. SOPs and checklists will be used to focus the internal audit on specific activities of the area to be audited.

12.1.4. Personnel will not be allowed to audit activities for which they are responsible or in which they are directly involved, unless it is demonstrated that an effective, nonbiased, audit can be performed.

12.1.5. Results of internal audits will be documented by the audit team and submitted to the manager(s) in charge of the audited area and the management of the QA Manager.

12.1.6. Appropriate corrective action steps will be promptly taken to address any deficiencies or areas for improvement identified by the internal audit. Laboratory management shall ensure that these actions are within the agreed time frame.

12.1.7. Laboratory management shall immediately notify, in writing, any client whose work may have been affected by any found deficiencies.

12.1.8. All records of internal facility inspections and responses will be maintained by the QA Manager.

12.2. Management Reviews

12.2.1. Management shall review the quality system annually to evaluate its continuing suitability and effectiveness and make any necessary changes or improvements.

12.2.2. The review may include account reports from managerial and supervisory personnel, the outcome of recent internal audits, assessments by external bodies, the results of interlaboratory comparisons or proficiency tests, any changes in the volume and type of work undertaken, feedback from clients, corrective actions, in-depth monitoring of data integrity, and other relevant factors.

12.3. Performance Testing Samples

12.3.1. Performance testing samples are conducted as single blind assay samples. A performance testing sample (PT), purchased from an independent contractor, is analyzed twice a year. The acceptable result for the PT sample is unknown until after the experimental result is reported to the contractor. Other externally originated PTs are analyzed when supplied by the client as either a single blind or as a double blind sample and are scheduled through the laboratory as routine samples. All performance testing samples are handled in the same manner as real environmental samples including staff, method, procedures, equipment, facilities, and frequency.

12.3.2. The samples shall be analyzed and the results returned to the PT Provider no later than 45 calendar days from the opening of the study.

12.3.3. When analyzing a PT sample, the same calibration, laboratory quality control and acceptance criteria, sequence of analytical steps, number of replicates and other procedures are employed as used when analyzing routine samples.

12.3.4. No PT sample, or a portion of a PT sample, shall be sent to another laboratory for any analysis

12.3.5. No PT sample or portion of a PT sample shall be received from another laboratory for any analysis

12.3.6. Vista Analytical Laboratory management or staff shall not communicate with any individual at another laboratory concerning

the PT sample or attempt to obtain the assigned value of any PT sample from their PT Provider.

12.3.7. Vista shall maintain copies of all written, printed, and electronic records, resulting from the analysis of any PT sample for five years or for as long as is required by the applicable regulatory program, whichever is greater. All of these laboratory records shall be made available to the assessors during on-site audits of the laboratory.

12.3.8. In the event that the laboratory receives test results that are "unacceptable", the likely cause is investigated, identified, and resolved. A Corrective Action PT sample, for which the laboratory shall report only the analytes for which corrective action was required, will then be analyzed. Documentation of the corrective action as well as the corrective action PT scores will then be submitted to the applicable accrediting authorities.

12.4. External Audits

12.4.1. External audits are performed on an on-going basis by clients, regulating agencies (State and Federal), or other third party auditors. These audits are pre-scheduled with the client and Quality Assurance Manager to ensure that the appropriate laboratory personnel are available to address all audit inquiries. All deviations or deficiencies noted during the audit are to be addressed in the time frame provided by the auditor.

12.5. Data Audits

12.5.1. Data audits at Vista utilize a three tier data review system involving laboratory directors, client managers and the QA Manager.

12.5.2. Tier 1. In the initial phase, the analyst, defined as the instrument operator, completes final data calculations, enters the data and submits the results to a laboratory director for review. In the case of anomalies, the laboratory director may require the analyst to prepare a corrective action report (CAR) discussing the potential causes for the problems encountered as well as the recommended corrective action. The analyst reviews the data, signs and dates the raw data and any CARs (if applicable). The laboratory director after review of the data will approve all final datasheets.

12.5.3. Tier 2. The second tier review requires the project manager, defined as the laboratory director signing the cover letter of the final report, to review and approve the data package. The project manager examines the data for completeness and assesses whether the package as a whole meets the data quality objectives set by the client. The project manager is required to discuss or explain any data anomalies in the text of the cover letter.

12.5.4. Tier 3. The third tier review is performed by the Quality Assurance Manager. The QA Manager will audit approximately 5% of the data packages and review all aspects of the data package covered during the second and third tier reviews. The QA Manager review may result in a request to the laboratory director for additional information regarding the data set and if necessary, re-analysis of selected samples.

13. TRAINING

Training assessments and all related training documentation shall be conducted in accordance with SOPs.

13.1. Initial On-Site Training

13.1.1. The training requirement of each employee will be assessed periodically to ensure the competency of their job responsibilities that career development objectives are being met, and that general-purpose educational opportunities are being utilized. The training program shall be relevant to the present and anticipated tasks of the laboratory.

13.1.2. Previous training, education, and experience will be considered when evaluating the training needs of each employee.

13.1.3. Manuals, texts, SOPs, journals, analytical methods and in-house Analytical Procedures are available for all new trainees, with on the job training performed by senior staff.

13.2. Training Programs

13.2.1. Job related training will be provided through regularly scheduled in-house seminars and courses, university courses, conferences and seminars, and one-on-one on the job tutorials.

13.2.2. Specified performance criteria must be successfully met while under supervision before personnel will be made responsible for activities that affect the quality objectives of the company.

13.3. Training Documentation

13.3.1. Training records will be maintained in each individual's training file. These records will be readily available to supervisors to ensure that employees have demonstrated capability prior to performing activities for which they are responsible. Employees are responsible for keeping their training file up-to-date. The training files shall maintain records of competence, education and professional qualifications, training, skills and experience of all technical personnel, including contracted personnel.

13.3.2. Evidence on file demonstrating each employee has read and understood the current version of in-house quality documents (QM, QAPP, SOPs).

13.3.3. Documentation of training courses.

13.3.4. Documentation of continued proficiency at least once per year.

14. CLIENT SERVICES

Routine client service as well as responses to client inquires, audit reports, recommendations, and complaints will be handled in accordance with SOPs.

14.1. Routine Services

14.1.1. Each client will be assigned a Project Manager who will be responsible for ensuring that the needs of the client are clearly understood and communicated to the appropriate areas of the company.

14.1.2. The Project Manager reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work. Once the Project Manager accepts the new work, an acknowledgement letter is sent to the client for confirmation.

14.1.3. Clients will be given the opportunity to verify that the company's services conform to specified requirements. Regardless of whether or not client verifications are conducted, the Quality System will be responsible for ensuring that all services conform to specified requirements.

14.1.4. As the client's representative, the Project Manager will be responsible for ensuring that the client's needs are met. The Project Manager will maintain good communication, advice and guidance in technical matters, and opinions and interpretations based on results.

14.1.5. All client data are managed and maintained with the utmost care and diligence to ensure that the protection of clients' confidential information and proprietary rights are a primary concern.

14.2. Contract Review

14.2.1. For all analytical service to be provided contract review is accomplished through the generation of a written quote or contract. Sales and client services personnel are responsible for implementing and documenting contract review. Client requirements are defined and documented in the written quote or contract.

14.3. Responses to Client Audits, Inquiries, and Complaints

14.3.1. The QA Manager will be responsible for coordinating responses to client audits.

14.3.2. Complaints received from clients or other parties regarding data or laboratory activities will be directed to the appropriate project manager and reported to the laboratory president or laboratory director.

14.3.3. If a corrective action(s), which may require completion of a CAR (corrective action report), is taken, this will be documented and archived with the appropriate project data.

14.3.4. All complaints will be documented and records of actions in response to any complaints will be maintained.

14.3.5. If a complaint raises doubt regarding the laboratory's policies or compliance with NELAP or other standards, those areas shall be promptly reviewed or audited by the laboratory QA Manager.

15. STATISTICAL TECHNIQUES

Statistical techniques used to monitor the performance of activities that directly affect quality objectives will be conducted in accordance with SOPs.

15.1. Statistical Process Control Procedures

15.1.1. Statistical Process Control will be used to monitor analytical procedure performance indicators such as accuracy and precision, and process performance indicators such as turnaround time and Nonconformance reports.

15.1.2. Results of SPC analyses will be used to improve processes that affect quality objectives.

16. SUBCONTRACTING

16.1. Vista Analytical may subcontract services, or may refer a client directly to another lab, for a particular analysis. Subcontracted laboratories are held responsible for the implementation of their own QM and meeting their data quality objectives.

16.2. Clients shall be notified prior to subcontracting any portion of their testing to another laboratory.

16.3. Services requiring NELAC accreditation will only be subcontracted to a laboratory with NELAC accreditation.

16.4. For DoD clients, subcontractor laboratories must have documented compliance with DoD QSM requirements, must be approved by the specific DoD laboratory approval process, must demonstrate the ability to generate acceptable results through the analysis of proficiency testing samples, and must receive project-specific approval from the DoD client before any samples are analyzed.

16.5. For services associated with projects outside of California, individual state accreditations may need to be met.

16.6. Vista Analytical shall retain records demonstrating that the above requirements have been met. Original reports received from a subcontracted laboratory will be included with the clients test report.

17. DATA INTEGRITY AND ETHICS

Vista Analytical Laboratory expects employee compliance with all laboratory SOPs and applicable regulatory guidelines and standards. Vista encourages participation in cooperative and educational efforts designed to promote and inform laboratory personnel of the necessity of active compliance.

17.1. Vista does not condone and will not tolerate the fraudulent manipulation or falsification of data, intentional non-compliance, gross negligence, or any other unethical conduct. Employees who are aware of, or reasonably suspicious of, any case fraudulent or unethical conduct shall notify the laboratory President, Director, or QA Manager. Allegations of unethical conduct may be reported anonymously and will be fully investigated under the direction of the Quality Assurance Manager.

17.2. Any employee who knowingly manipulates and/or falsifies data or documents or engages in any unethical conduct is subject to immediate release from employment and other serious consequences.

17.3. Vista Analytical Laboratory provides mandatory initial and annual or as needed, Laboratory Ethics and Data Integrity refresher training to all employees. Topics covered are approved by management, documented in writing, and provided to all trainees.

17.3.1. Training topics include:

- Quality System requirements
- Personnel training requirements
- Vista Analytical Laboratory Ethics policy
- Examples of actions that are strictly prohibited
- Other breaches of data integrity
- Pertinent SOPs and other quality documents
- Potential consequences of misconduct
- Confidential mechanism for reporting allegations
- Investigation procedures and documentation

17.3.2. All employees sign an ethics statement and documentation of training attendance that demonstrates they have participated and understand their obligations related to data integrity. This sheet is maintained in individual training records.

17.4. Upon hire, new employees are required to read and sign a confidentiality statement. This signed statement is maintained in personnel files.

APPENDIX

Key Resumes

Certifications

William J. Luksemburg
President

EDUCATION

B.S. Chemistry, California State University, Fresno, CA (1974)

EXPERIENCE

Present **President, Vista Analytical Laboratory**
Responsible for the management of business planning including venture funding, sales and marketing and the review of laboratory operations of Vista Analytical Laboratory, formerly Alta Analytical Laboratory.

1990 - 2000 **Director of HRMS Services, Alta Analytical Laboratory**
Mr. Luksemburg, a co-founder, directed the routine analysis and method development work in the High Resolution Mass Spectrometry department. He was responsible for marketing HRMS dioxin services to environmental engineering firms, the pulp and paper industry, government agencies and other industrial clients. Mr. Luksemburg was also responsible for the development of new markets using HRMS instrumentation. In addition Mr. Luksemburg directed routine and special projects, reviewed and interpreted data, and interfaced with clients.

1986 - 1990 **Principal Scientist/HRMS Manager, Enseco-Cal Lab**
As Principal Scientist in the Special Services department at Enseco-Cal Lab Mr. Luksemburg coordinated the operation and maintenance of five high resolution magnetic sector instruments. He was responsible for developing a business that now is one of the major suppliers of HRMS PCDD/PCDF analysis to the pulp and paper industry in the U.S. Mr. Luksemburg also coordinated the training and development of the staff in the operation and maintenance of HRMS instruments.

1979 - 1986 **Senior Chemist, Radian Corporation**
In Radian's Sacramento laboratory, Mr. Luksemburg was GC/MS supervisor for ABN and VOA analysis. He coordinated the activities of five chemists in the operation and maintenance of four quadrupole mass spectrometers.

1974 - 1979 **Chemist, Carnation Company**
As a staff chemist, Mr. Luksemburg was involved in the analysis of products and ingredients used in Carnation's animal feed division.

QUALIFICATIONS

Mr. Luksemburg has over 30 years experience in production analytical laboratories including 25 years experience in the field of environmental mass spectrometry. Much of this experience has involved PCDD/PCDF analysis of environmental samples, concentrated on High Resolution Mass Spectrometry analysis of PCDDs/PCDFs in a variety of matrices. Mr. Luksemburg is recognized throughout the pulp and paper industry for his research and production work on dioxins and furans. He recently was recognized on the international level when his chapter on dioxin analysis of pulp and paper (Rappe, 1991), was published by the World Health Organization. He is one of the few individuals in the world to successfully adapt the high-resolution magnetic sector instruments to "production" analysis of environmental samples at the picogram and femtogram levels.

RECENT PUBLICATIONS AND PRESENTATIONS

"Determination of Method Detection Limits in Pulp and Paper Mill Effluents," in Rotorua, New Zealand, at the *ISWPC Post Symposium Workshop*, May 1991.

"Comparison of NCASI Method 551, EPA Method 1613A, and the Proposed FDA Method for the Analysis of 2,3,7,8-TCDD and 2,3,7,8-TCDF in Food Packaging Material," in Boston, MA, at the *1993 TAPPI Environmental Conference*, March 1993.

"Extraction of Large Volumes of Aqueous Samples Using Solid Phase Extraction Disks," in Portland, OR at the *1994 TAPPI Environmental Conference*, April 1994.

"PCDDs and PCDFs in Urban Stormwater Discharged to San Francisco Bay, California," in Amsterdam at the *1996 Dioxin 16th Symposium on Chlorinated Dioxins and Related Compounds*, August 1996.

NCASI Technical Bulletin No. 551, "NCASI Procedures for the Preparation and Isomer Specific Analysis of Pulp and Paper Industry Samples for 2,3,7,8,-TCDD and 2,3,7,8-TCDF," LaFleur, L., Ramage, K., Bousquet, T., Brunck, R., Luksemburg, W., Miille, M., Peterson, R., and Valmores, S., (1989).

"Optimization of Extraction Procedures for the Analysis of TCDD/TCDF in Pulp, Paper Base Stocks, and Pulp Industry Solid Wastes," Lafleur, L., Ramage, K., Gillespie, W., Luksemburg, L., Miille, M., and Valmores, S., Chemosphere, Vol. 19, pp 643-648, 1989.

"Analytical Procedures for the Analysis of TCDD and TCDF in Food Sources," LaFleur, L., Bousquet, T., Ramage, K., Davis, T., Luksemburg, W., and Peterson, R., Presented by L. Lafleur at Dioxin '89, Toronto, Canada. Waiting publication in Chemosphere.

"Determination of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans in Pulp and Paper Industry Wastewaters, Solid Wastes, Ashes and Bleached Pulps," Luksemburg,

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Levels of Polybrominated Diphenyl Ethers(PBDEs) in the Hackensack River and Newark Bay, New Jersey USA, Wenning, R., Von Burg, A., and Luksemburg, W., Presented at BFR 2004, June, 2004, Toronto, Canada.

PROFESSIONAL AFFILIATIONS

American Society for Mass Spectrometry
American Chemical Society
Technical Association of the Pulp and Paper Industry
Society of Environmental Toxicology and Chemistry
American Association for the Advancement of Science

Martha M. Maier
Laboratory Director

EDUCATION

B.S.	Chemistry, University of Wisconsin, Madison, WI (1983)
B.S.	Philosophy, University of Wisconsin, Madison, WI (1983)

EXPERIENCE

Present **Laboratory Director, Vista Analytical Laboratory, Inc.**
The Laboratory Director for Vista Analytical Laboratory, formerly Alta Analytical, oversees the routine operations of the laboratory. Performs the interpretation and final review of analytical data, and issues final reports. Acts as a liaison between the laboratory and the Quality Assurance department. Project manager for routine and special projects.

1999-2001 **Director, Ultra-Trace Analyses Group, Paradigm Analytical Laboratories, Inc**
Responsible for extractions, analyses, final review and processing of all data generated by the group. Served as project manager. Oversaw the development of analytical procedures for the analysis for PCBs by HRMS (Method 1668A), as well as the implementation of NELAP certification.

1998-1999 **Bioanalytical Project Manager, Alta Analytical Laboratory**
Liaison between pharmaceutical clients and the Liquid Chromatography Mass Spectrometry (LCMS) Services group, ensuring efficient study management and timely reporting of laboratory results. Directed all phases of study conduct, including: review of study protocols and sponsor Standard Operating Procedures; initiation, maintenance and review of study and raw data files; scheduling of sample analyses; and preparation of final reports.

1992-1998 **Associate Scientist, Alta Analytical Laboratory**
Involved in sales and project management. Directed sample analysis, reviewed data and prepared reports. Presented papers and gave educational seminars and presentations on dioxin/furan analysis. Arranged exhibit schedule and represented the laboratory at technical meetings and industry conferences. From 1992-1997, acted as laboratory representative for the Eastern U.S., both in sales and project management capacities.

1990-1992 **Technical Sales, Enseco-Cal Lab**
Coordinated the dioxin/furan marketing program. Prepared bids, organized exhibits, and oversaw the production of marketing materials. Acted as a liaison between the salespeople and the dioxin/furan laboratory.

1988-1990 **HR GC/MS Operator, Enseco-Cal Lab**
Dioxin/furan analysis of pulp, food, and low-level environmental samples using high resolution GC/MS. Promoted to scientist position in December 1989. Involved in data review and project management.

1987-1988 **GC/MS Operator, Enseco-Cal Lab**
Dioxin/furan analysis using low-resolution GC/MS systems. Promoted to lead person in May 1988.

1986-1987 **GC/MS BNA Operations Supervisor, Radian Corporation**
Responsible for the scheduling and completion of all semi volatile analyses. Trained other operators in BNA analysis and routine instrument maintenance.

1984-1986 **GC/MS Operator, Radian Corporation**
Analyzed environmental samples for volatile and semi volatile organic pollutants using EPA Methods 624, 625, SW-8240, SW-8270, and by EPA Contract Lab Protocol. Performed routine maintenance on all systems. Responsible for interfacing the GC/MS lab with the laboratory database management system.

1984-1984 **Analytical Chemist, Wisconsin Department of Agriculture**
Assayed pesticide formulations using HPLC, GC, and TLC.
Researched, developed and modified methods.

QUALIFICATIONS

Ms. Maier has over 22 years of experience in the environmental laboratory, including 19 years of specialization in dioxin/furan analysis.

AFFILIATIONS

Air & Waste Management Association
American Chemical Society
Technical Association of the Pulp & Paper Industry

James M. Hedin
Director of Instrumentation Laboratory

EDUCATION

B.S. B.S. Chemistry, University of Minnesota, Duluth, MN (1986)

EXPERIENCE

Present **Director of Instrumentation Laboratory, Vista Analytical Laboratory**
Mr. Hedin performs routine analysis and method development work in the High Resolution Mass Spectrometry department at Vista Analytical Laboratory, formerly Alta Analytical Laboratory. He is responsible for routine maintenance of HR/MS instruments, training of new staff, review and interpretation of data, and client service.

1990 – 1999 **Associate Scientist, Alta Analytical Laboratory**
Mr. Hedin performs routine analysis and method development work in the High Resolution Mass Spectrometry department. He is responsible for routine maintenance of HR/MS instruments. Mr. Hedin also aids in the training of new staff, reviews and interprets data, and interfaces with clients.

1988 – 1990 **GC/MS Chemist, Enseco-Cal Lab**
As GC/MS Chemist at Enseco-Cal Laboratory, Mr. Hedin was responsible for the operation and maintenance of quadrapole GC/MS instruments. His duties entailed sample analysis by EPA methods for volatiles and semi-volatiles and also aided in the training of the staff in the department.

1987 – 1988 **Extraction Chemist, Enseco-Cal Lab**
Mr. Hedin's duties entailed sample extraction for Dioxin/Furan Analysis by High Resolution Mass Spectrometry, training of new staff, and the development of new extraction techniques.

QUALIFICATIONS

Mr. Hedin has over 20 years experience in production analytical laboratories and environmental mass spectrometry. Most of this experience has involved PCDD/PCDF analysis of environmental samples and High Resolution Mass Spectrometry analysis of PCDD's/PCDFs in a variety of matrices.

PROFESSIONAL AFFILIATIONS

American Society for Mass Spectrometry

Rose M. Harrelson
Quality Assurance Manager

EDUCATION

B.S. Physiology, University of California, Davis (1989)

EXPERIENCE

Present **Quality Assurance Manager, Vista Analytical Laboratory**
Ensure compliance to the laboratory Quality System according to the National Environmental Laboratory Accreditation Program (NELAP) standards and Alta's Quality Manual (QM); review and manage performance of MDLs, IPRs, PE samples; review data packages for compliance and completeness; maintain state certifications; maintain and update SOPs; maintain and update control charts; provide employee orientation and training; maintain and update QM and SOQ.

2001 – 2005 **Quality Assurance Specialist, Air Toxics Ltd.**
Technical and QA review of analytical data, technical liaison between clients and laboratory operations; create, implement, and maintain QA controls and documentation; review and revise SOPs; collection and assessment of QC data; internal and external lab audit reports and responses; implement preventive and corrective actions; manage laboratory certifications; develop, implement, and manage the internal training program; serve as project manager for proficiency testing samples.

1992 – 1999 **Quality Assurance Specialist, Quanterra Environmental Services**
Facilitated the implementation of Quality Assurance policies at the facility; performed as the QA Unit for the pesticide registration GLP program; reviewed work proposals and project plans for quality assurance aspects; coordinated audit activities at the facility; conducted QA training courses; responded to auditors regarding audits and performance evaluation samples; recommended corrective action as appropriate; maintained state certifications and agency approvals; maintained records pertaining to control charts, method validation and method detection limits, performance evaluation results, audit results, QC database, and customer service; assisted in the standardization and development of laboratory SOPs.

QUALIFICATIONS

Ms. Harrelson has over 18 years of experience in the environmental laboratory, including 15 years of specialization in laboratory Quality Assurance.

CERTIFICATIONS

Accrediting Authority	Certificate Number
State of Alaska, DEC	CA413-2008
State of Arizona	AZ0639
State of Arkansas, DEQ	08-043-0
State of Arkansas, DOH	Reciprocity through CA
State of California – NELAP Primary AA	02102CA
State of Colorado	N/A
State of Connecticut	PH-0182
State of Florida, DEP	E87777
State of Indiana Department of Health	C-CA-02
Commonwealth of Kentucky	90063
State of Louisiana, Health and Hospitals	LA08000
State of Louisiana, DEQ	01977
State of Maine	2008024
State of Michigan	9932
State of Mississippi	Reciprocity through CA
Naval Facilities Engineering Service Center	NFESC413
State of Nevada	CA004132007A
State of New Jersey	CA003
State of New Mexico	Reciprocity through CA
State of New York, DOH	11411
State of North Carolina	06700
State of North Dakota, DOH	R-078
State of Oklahoma	D9919
State of Oregon	CA200001-006
State of Pennsylvania	68-00490
State of South Carolina	87002001
State of Tennessee	TN02996
State of Texas	T104704189-08-TX
U.S. Army Corps of Engineers	N/A
State of Utah	CA16400
Commonwealth of Virginia	00013
State of Washington	C1285
State of Wisconsin	998036160
State of Wyoming	8TMS-Q

Current certificates and lists of licensed parameters are located in the Quality Assurance office and are available upon request.

NELAP - RECOGNIZED

CALIFORNIA STATE

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM BRANCH

CERTIFICATE OF NELAP ACCREDITATION

Is hereby granted to

VISTA ANALYTICAL LABORATORY

1104 WINDFIELD WAY

EL DORADO HILLS, CA 95762

Scope of the Certificate is limited to the
"NELAP Fields of Accreditation"
which accompany this Certificate.

Continued accredited status depends on successful
ongoing participation in the program.

This Certificate is granted in accordance with provisions of
Section 100825, et seq. of the Health and Safety Code.

Certificate No.: **02102CA**

Expiration Date: **01/31/2011**

Effective Date: **02/01/2010**

Richmond, California
subject to forfeiture or revocation

George C. Kulasingam, Ph.D., Chief
Environmental Laboratory Accreditation Program Branch

CALIFORNIA DEPARTMENT OF PUBLIC HEALTH

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM - NELAP RECOGNIZED

NELAP Fields of Accreditation

VISTA ANALYTICAL LABORATORY

1104 WINDFIELD WAY
EL DORADO HILLS, CA 95762
Phone: (916) 933-1520

Certificate No.: 02102CA
Renew Date: 1/31/2011

105 - Semi-volatile Organic Chemistry of Drinking Water			
105.230	000	EPA 1613	Dioxins
105.230	001	EPA 1613	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)
111 - Semi-volatile Organic Chemistry of Wastewater			
111.090	001	EPA 613	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)
111.111	000	EPA 1613B	Dioxins
111.111	001	EPA 1613B	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)
111.111	002	EPA 1613B	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)
111.111	003	EPA 1613B	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
111.111	004	EPA 1613B	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
111.111	005	EPA 1613B	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)
111.111	006	EPA 1613B	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)
111.111	007	EPA 1613B	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)
111.111	008	EPA 1613B	2,3,7,8-Tetrachlorodibenzofuran (TCDF)
111.111	009	EPA 1613B	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)
111.111	010	EPA 1613B	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)
111.111	011	EPA 1613B	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)
111.111	012	EPA 1613B	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)
111.111	013	EPA 1613B	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)
111.111	014	EPA 1613B	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)
111.111	015	EPA 1613B	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)
111.111	016	EPA 1613B	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)
111.111	017	EPA 1613B	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)
111.111	018	EPA 1613B	Total TCDD
111.111	019	EPA 1613B	Total PeCDD
111.111	020	EPA 1613B	Total HxCDD
111.111	021	EPA 1613B	Total HpCDD
111.111	022	EPA 1613B	Total TCDF
111.111	023	EPA 1613B	Total PeCDF
111.111	024	EPA 1613B	Total HxCDF
111.111	025	EPA 1613B	Total HpCDF
117 - Semi-volatile Organic Chemistry of Hazardous Waste			
117.120	000	EPA 8280A	Dioxins and Dibenzofurans
117.120	001	EPA 8280A	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)

As of 1/12/2010 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

ERRATA SHEET - SAMPLING AND ANALYSIS PLAN: SUPPLEMENTAL INVESTIGATION OF THE LYMAN MILL REACH SEDIMENT AND FLOOD PLAIN SOILS

Centredale Manor Restoration Project Superfund Site in
North Providence, Rhode Island

EPA Comment #1 – The sampling locations are OK.

Integral Response: No change required.

EPA Comment #2 – The maximum sampling depth listed is 36 inches, whereas we agreed to 48 inches should vadose extend below 36 inches. Please include contingency language in FSP (if not already there) that sampling will extend deeper as necessary to encompass the vadose zone.

Integral Response: Maximum sampling depth has been revised to 48 in (122 cm).

Revisions have been made to the following:

- Appendix A Field Sampling Plan: Page A-3, Section 2.2.1; Page A-6, Section 2.2.4; Page A-15, Section 3.5; and Tables A-3 and A-4
- SAP: Page 2-1, Sections 2.1 and 2.2

EPA Comment #3 – The FSP references two surface zones: 0-12 inches and 0-30 inches. Do you really propose 0-30 inch depth interval on some samples and what would these be used for? Per Region 1 risk assessment guidance, surficial samples should be collected over a depth of 0-12 inches.

Integral Response: The FSP refers to 0-12 inch and 0-30 cm sampling depths, which are equivalent. We could not locate where the FSP refers to a 0-30 *inch* depth, so no changes were made to the document. Nevertheless, Integral's intent is to sample the 0-30 cm depth as representative of the surface soil.

EPA Comment #4 – list of analytes – can you include bis(2-chloroisopropyl) ether?

Integral Response: Bis(2-chloroisopropyl)ether is included as an analyte, listed as 2,2'oxybis(1-Chloropropane); CAS #108-60-1. See Table 2-3, SAP

EPA Comment #5 – Need lower laboratory detection limits for several analytes:

- Dieldrin: 0.14 mg/kg MDL (1 mg/kg MRL) vs. 0.04 mg/kg ARAR (RIDEM residential direct exposure criteria)
- Technical Chlordane: 1.9 mg/kg MDL (10 mg/kg MRL) vs. 0.5 mg/kg ARAR (RIDEM residential direct exposure criteria)
- Antimony: 0.5 mg/kg MDL (2 mg/kg MRL) vs. 0.62 mg/kg cleanup goal
- Selenium: 0.7 mg/kg MDL (4 mg/kg MRL) vs. 0.7 mg/kg cleanup goal

- 4,4'-DDE: 0.11 mg/kg MDL (1 mg/kg MRL) vs. 0.02 mg/kg cleanup goal
- Aroclor 1254: 2.1 mg/kg MDL (10 mg/kg MRL) vs. 0.82 mg/kg cleanup goal

Integral Response: The detection limits currently listed in the Sampling and Analysis Plan for dieldrin, technical chlordane, 4,4'-DDE, and Aroclor 1254 are lower than those requested by EPA in the comment. Consequently, no change is necessary for these organic compounds.

USEPA Method 6020 (ICP-MS) will be used to analyze antimony and selenium. With this method the following detection limits for antimony and selenium will be achieved:

- Selenium MDL = 0.2 mg/kg; MRL = 0.7 mg/kg
- Antimony MDL = 0.02 mg/kg; MRL = 0.05 mg/kg

Revisions to the selenium and antimony MDLs and MRLs have been made to the following:

- SAP: Page 2-3, Section 2-4; Tables 2-2, 2-3, 2-6; and
- Appendix B of the QAPP

EPA Comment #6 – EPA recommends a field duplicate by means of collecting second core at same location rather than taking a second aliquot from the homogenization bowl.

Integral Response: The purpose of collecting a duplicate sample is to measure variability. There are several sources of variability that arise from field sampling programs. The primary sources are: (1) variability of analyte concentration in the sampled media by location and/or depth; and (2) a lack of precision resulting from inadequate field sampling method, such as during soil homogenization.

The first source of variability is a function of sample location, sample depth and analyte concentration. The sampling plan proposed by Integral will measure this variability by sampling soil at numerous locations. For example, we propose to collect surface soil samples at 42 different locations. The analytical data resulting from the 42 soil sampling locations will help quantify the variability of the data resulting from differences in concentration at these locations.

The purpose of the field duplicate proposed in the Sampling and Analysis Plan is not to measure the variability of the data resulting from location, which would be inherently measured if EPA's recommendation were followed, but rather to measure the variability resulting from sample handling and analysis procedures. In essence, the field duplicate is a measure of the precision primarily of the sample homogenization procedure. Perhaps the duplicate sample could have been referred to as a replicate rather than a duplicate, but the end result will be the same – a measure of the variability resulting from sample handling.

Based on the foregoing, we have not made any changes to the Sampling and Analysis Plan regarding EPA comment #6.

117.120	002	EPA 8280A	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)
117.120	003	EPA 8280A	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
117.120	004	EPA 8280A	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
117.120	005	EPA 8280A	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)
117.120	006	EPA 8280A	2,3,7,8-Tetrachlorodibenzofuran (TCDF)
117.120	007	EPA 8280A	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)
117.120	008	EPA 8280A	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)
117.120	009	EPA 8280A	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)
117.120	010	EPA 8280A	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)
117.120	011	EPA 8280A	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)
117.120	012	EPA 8280A	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)
117.120	013	EPA 8280A	Total TCDD
117.120	014	EPA 8280A	Total PeCDD
117.120	015	EPA 8280A	Total HxCDD
117.120	016	EPA 8280A	Total TCDF
117.120	017	EPA 8280A	Total PeCDF
117.120	018	EPA 8280A	Total HxCDF
117.120	019	EPA 8280A	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)
117.120	020	EPA 8280A	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)
117.120	021	EPA 8280A	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)
117.120	022	EPA 8280A	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)
117.120	023	EPA 8280A	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)
117.120	024	EPA 8280A	Total HpCDD
117.120	025	EPA 8280A	Total HpCDF
117.130	000	EPA 8290	Dioxins and Dibenzofurans
117.130	001	EPA 8290	2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)
117.130	002	EPA 8290	1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)
117.130	003	EPA 8290	1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
117.130	004	EPA 8290	1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)
117.130	005	EPA 8290	1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)
117.130	006	EPA 8290	2,3,7,8-Tetrachlorodibenzofuran (TCDF)
117.130	007	EPA 8290	1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)
117.130	008	EPA 8290	2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)
117.130	009	EPA 8290	1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)
117.130	010	EPA 8290	1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)
117.130	011	EPA 8290	1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)
117.130	012	EPA 8290	2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)
117.130	013	EPA 8290	1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)
117.130	014	EPA 8290	1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)
117.130	015	EPA 8290	1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)
117.130	016	EPA 8290	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin (OCDD)
117.130	017	EPA 8290	1,2,3,4,6,7,8,9-Octachlorodibenzofuran (OCDF)