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Subject:
Former Koppers Wood-Treating Site – Carbondale, Illinois
Revised Quality Assurance Project Plan

ENVIRONMENTAL

Date:
February 8, 2008

Contact:
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Email:
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Our ref:
B0039093

Dear Ms. Bury:

On behalf of Beazer East, Inc. (Beazer), enclosed for your review and approval is a *Quality Assurance Project Plan* (QAPP) for environmental sampling and analysis activities to be conducted as part of Remediation Support and Post-Remediation Operation, Maintenance, and Monitoring activities for the Former Koppers Wood-Treating Site in Carbondale, Illinois (the Site). Both a hard copy and CD of the QAPP are enclosed. This version of the QAPP supersedes the version previously submitted in May 2006, and has been revised to address the USEPA's February 1, 2007 comments (consistent with Beazer's May 9, 2007 responses to those comments). It has also been revised to reflect the current scope of the *Baseline Characterization Work Plan* sampling activities. The signature page will be completed and issued following USEPA approval of the QAPP.

Please feel free to contact me 860.645.1084 or Michael Slenska of Beazer 412.208.8867 with any questions or comments.

Sincerely,

ARCADIS

Jeffrey S. Holden
Senior Engineer

Copies:

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** CD only*

ARCADIS
Infrastructure, environment, facilities

Beazer East, Inc.

Pittsburgh, Pennsylvania

Quality Assurance Project Plan

Former Koppers Wood-Treating Site

Carbondale, Illinois

February 2008

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Quality Assurance Project Plan

Former Koppers Wood-Treating Site
Carbondale, Illinois

Prepared for:
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A Field Sampling Standard Operating Procedures

B Laboratory Standard Operating Procedures (on CD-ROM)

C Laboratory Quality Assurance Manuals (on CD-ROM)

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Quality Assurance Project Plan

Former Koppers
Wood-Treating Facility,
Carbondale, Illinois

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United States. Environmental Protection Agency

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Quality Assurance Project Plan

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Quality Assurance Project Plan

Former Koppers
Wood-Treating Facility,
Carbondale, Illinois

Acronyms and Abbreviations

AMEC	AMEC Earth & Environmental, Inc.
ASTM	American Society of Testing and Materials
BBL	Blasland, Bouck & Lee, Inc.
Beazer	Beazer East, Inc.
BTEX	benzene, toluene, ethylbenzene, and xylenes
CAMU	Corrective Action Management Unit
CAS	Columbia Analytical Services, Inc.
CLP	Contract Laboratory Program
COC	chain of custody
COPI	constituent of potential interest
DNAPL	dense, non-aqueous phase liquid
DQO	data quality objective
EDD	electronic data deliverable
FSP	Field Sampling Plan
FTS	Field & Technical Services LLC
GC/MS	gas chromatography/mass spectrometry
GIS	geographic information system
IDW	investigation-derived waste
IEPA	Illinois Environmental Protection Agency
IGMP	Interim Groundwater Monitoring Program
LCS	laboratory control sample
Lotic	Lotic, Inc.
MNR	monitored natural recovery
MS	matrix spike
MSD	matrix spike duplicate

MS/MSD	matrix spike/matrix spike duplicate
Nautilus	Nautilus Environmental
NEIC	National Enforcement Investigations Center
OMM	operation, maintenance, and monitoring
OSHA	Occupational Safety and Health Administration
PAH	polycyclic aromatic hydrocarbon
PCDDs/PCDFs	polychlorinated dibenzo-p-dioxins/polychlorinated dibenzofurans
PPE	personal protective equipment
QA	quality assurance
QAC	QA Coordinator
QAPP	Quality Assurance Project Plan
QA/QC	quality assurance/quality control
QAM	Quality Assurance Manual
QC	quality control
RCRA	Resource Conservation and Recovery Act
RPD	relative percent difference
SDG	sample delivery group
Site	Former Koppers Wood-Treating Site, Carbondale, Illinois
SOP	Standard Operating Procedure
Test America	Test America, Inc.
USACOE	U.S. Army Corps of Engineers
USEPA	United States Environmental Protection Agency
Vista	Vista Analytical Laboratory, Inc.
VOC	volatile organic compound

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Quality Assurance Project Plan

Former Koppers
Wood-Treating Facility,
Carbondale, Illinois

1. Introduction

This *Quality Assurance Project Plan* (QAPP) summarizes quality-assurance-related information applicable to future environmental sampling and analysis activities associated with the Former Koppers Wood-Treating Site in Carbondale, Illinois (the Site). Such sampling and analysis activities are to be conducted as part of Remediation Support and Post-Remediation Operation, Maintenance, and Monitoring (OMM) activities for the Site. Note that, because this QAPP applies to future sampling and analysis activities, work plans for those activities have not necessarily been developed. When such work plans are developed, they, in combination with this QAPP, will constitute the Sampling and Analysis Plan for the Site. If future Site activities result in the need for sampling and analysis activities beyond those currently envisioned and discussed in this QAPP, any quality-assurance-related information that is applicable to such activities, but is not addressed by this QAPP, will be summarized in the associated work plans and/or an addendum to this QAPP. This QAPP supersedes the QAPP that was developed for the Remedial Investigation (*QAPP for the Interim Groundwater Sampling Program, Feasibility Study and Remedial Action Carbondale Wood-Treating Site*, September 24, 1992).

On behalf of Beazer East, Inc. (Beazer), ARCADIS has prepared this QAPP to address anticipated future sampling and field investigations. While certain of those investigations will be conducted by ARCADIS, others will be conducted by other consultants on behalf of Beazer. Based on the foreseeable scope of future investigation and monitoring activities, other consultants expected to be involved in Site-related sampling and analysis include AMEC Earth & Environmental, Inc. (AMEC) and Field & Technical Services LLC (FTS). Accordingly, input into the development of this QAPP has been provided by those firms.

This QAPP was prepared in a manner consistent with the following reference and guidance documents:

- United States Environmental Protection Agency (USEPA) guidance document titled *EPA Requirements for Quality Assurance Project Plans for Environmental Operations*, EPA-QA/R-5 (USEPA, 2001), which replaces QAMS-005/80, *Interim Guidance and Specifications for Preparing Quality Assurance Project Plans* (USEPA, 1980)

- USEPA *Guidance for Quality Assurance Project Plans* (USEPA, 2002b)

- USEPA Region 5 guidance document titled *Instructions on the Preparation of a Superfund Division Quality Assurance Project Plan* (USEPA, 2000)

- USEPA National Enforcement Investigations Center (NEIC) guidance titled *NEIC Policies and Procedures Manual* (USEPA, 1991)

Following this introduction, the information contained in this QAPP has been organized into the sections outlined in the following table. References cited in this document are listed in Section 23.

Section	Content
Project Management	
2	Project Organization – Identifies the project team and their roles/responsibilities
3	Project Background – Discusses the Site location, description, history, activities, and current status
4	Project Description – Discusses the various sampling/analysis activities that are anticipated to be conducted at the Site
5	Quality Objectives and Criteria for Measurement Data – Discusses data quality objectives, data and reporting categories, and the scope of the anticipated sampling and analysis activities
6	Special Training Requirements/Certification – Discusses special training/certification requirements for field and laboratory personnel
7	Documentation and Records – Discusses field and laboratory documentation, reporting, and record keeping requirements
Measurement/Data Acquisition	
8	Sampling Process Design – Discusses the anticipated sampling scopes and refers to previous sections and specific work plans for additional details
9	Sampling Method Requirements – Discusses the anticipated sampling methods and refers to previous sections, attached Standard Operation Procedures (SOPs), and specific work plans for additional details
10	Sample Handling and Custody Requirements – Discusses sample containers and preservation requirements; field and laboratory custody procedures; sample packaging, handling and shipping; and management of investigation wastes
11	Analytical Method Requirements – Discusses analytical method requirements for field and laboratory parameters and refers to applicable tables
12	Quality Control Requirements – Discusses quality assurance (QA) indicators, field and laboratory quality control (QC) checks, and data precision/accuracy/completeness assessment procedures
13	Instrument/Equipment Testing, Inspection, and Maintenance Requirements – Discusses testing, inspection, and maintenance requirements for field and laboratory instruments/equipment
14	Instrument Calibration and Frequency – Discusses calibration requirements for field and laboratory instruments/equipment

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Quality Assurance Project Plan

Former Koppers Wood-Treating Facility, Carbondale, Illinois

Section	Content
15	Inspection/Acceptance Requirements for Supplies and Consumables – Discusses inspection/acceptance requirements for field and laboratory supplies
16	Data Acquisition Requirements for Non-Direct Measurements – Discusses the method for assessing the usability of historical data
17	Data Management – Discusses sample designation, documentation and security of field data, and sample/data management and tracking
Assessment/Oversight	
18	Assessment and Response Actions – Discusses field and laboratory audits and corrective actions
19	Reports to Management – Discusses field and laboratory reporting requirements
Data Validation and Usability	
20	Data Reduction and Review – Discusses reduction and review of field and laboratory data
21	Data Verification and Validation – Describes the data verification and validation process
22	Reconciliation with User Requirements – Discusses the process of determining data usability and corrective actions

2. Project Organization

2.1 Project Team

Investigations performed as part of the Remediation Support and Post-Remediation OMM activities at the Site will require integration of personnel from the organizations identified below, collectively referred to as the "project team." A list of key project organizations and personnel that comprise the project team is provided in the following table:

Company/Organization	Title	Name	Phone Number
United States Environmental Protection Agency (USEPA)	Project Manager	Carolyn Bury	(312) 886-3020
	Quality Assurance Coordinator (QAC)	Brian Freeman	(312) 353-2720
Illinois Environmental Protection Agency (IEPA)	Project Manager	James Moore	(312) 353-7922
Beazer East, Inc. (Beazer)	Project Manager	Michael Slenska	(412) 208-8867
Beazer Consultants			
ARCADIS	Program Manager	Jeffrey Holden	(860) 645-1084
	Project Manager	David Bessingpas	(218) 829-4607
	Quality Assurance Coordinator (QAC)	Dennis Capria	(315) 446-9120
Field & Technical Services LLC (FTS)	Project Manager	Karen Fromme	(412) 279-3363
	Task/Field Manager	Michael Relich	(724) 756-6400
AMEC Earth & Environmental, Inc. (AMEC)	Project Manager	Paul Anderson	(978) 692-9090
	Task/Field Manager	Alissa Weaver	
Analytical Laboratories			
Test America, Inc. (Test America; formerly Severn Trent Laboratories, Inc.).	Project Manager	Veronica Bortot	(412) 963-7058
	QAC	Nasreen DeRubeis	
Vista Analytical Laboratory, Inc. (Vista; formerly ALTA Laboratory, Inc.)	Project Manager	Martha Maier	(916) 933-1640
	QAC	Rose Harrelson	(916) 933-1640
Lotic, Inc. (Lotic)	Project Manager	Bruce Grantham	(207) 948-3062
	QAC	John Tipping	(207) 948-3062

Company/Organization	Title	Name	Phone Number
Nautilus Environmental (Nautilus)	Project Manager	John Rudolph	(858) 587-7010
	QAC	Angi Hotz	
Columbia Analytical Services, Inc. (CAS)	Project Manager	Jeff Christian	(360) 501-3316
	QAC	Lee Wolf	(360) 501-3317

A project organizational chart illustrating the inter-relation among the various organizations is provided in Appendix A.

2.2 Roles and Responsibilities

Information regarding the roles and responsibilities of the various organizations and personnel is presented below. Note that the descriptions are specific to the various key roles within each organization. These roles remain applicable regardless of any subsequent personnel changes within the specific organizations comprising the project team.

2.2.1 Beazer

Project Manager

- Overall responsibility for conducting and coordinating corrective action activities for the Site.
- Provide direction to consultants (ARCADIS, AMEC, and FTS).
- Review consultant work products, including memoranda, letters, reports, and all other documents transmitted to the USEPA and the IEPA.
- Coordinate with regulatory agencies.

2.2.2 Beazer Consultants

Program/Project Manager

- Manage and coordinate the project activities on behalf of Beazer.
- Review documents prepared prior to submittal to Beazer.
- Coordinate and direct support personnel as needed to implement project tasks, such as field sampling, reporting, etc.
- Verify that corrective actions are taken for deficiencies cited during any audits of Site activities.
- Coordinate with Beazer and other members of the project team.

Task Managers

- Manage relevant day-to-day activities.
- Develop, establish, and maintain files on relevant Site activities.
- Review data reductions from relevant Site activities.
- Perform final data review of field data reductions and reports on relevant Site activities.
- Verify that corrective actions are taken for deficiencies cited during audits of relevant Site activities.
- Perform overall quality assurance/quality control (QA/QC) of the relevant portions of the Site activities.
- Review relevant field records and logs.
- Coordinate with personnel conducting field work.
- Coordinate field and laboratory schedules pertaining to relevant Site activities.
- Coordinate with analytical laboratory(ies) to arrange for sample bottles, analysis, and reporting of data.

- Review field instrumentation, maintenance, and calibration data.
- Prepare reports pertaining to relevant Site activities.
- Maintain field and laboratory files of notebooks/logs, data reductions, and calculations.

Field Personnel

- Review work plans and SOPs.
- Calibrate, operate, and maintain field equipment.
- Perform field procedures and sample applicable media associated with the investigation(s) in accordance with work plans and SOPs.
- If required, perform field monitoring and/or analyses and collect QA samples.
- Maintain sample integrity. Use of proper Chain-of-Custody forms and proper inventory during packing and shipping.
- Prepare field records and logs.

Quality Assurance Coordinator

- Ensure laboratory schedules and turnaround times are met.
- Review laboratory data packages.
- Oversee and interface with the analytical laboratory(ies).
- Coordinate field QA/QC procedures with Task Managers (including audits of field activities), concentrating on field analytical measurements and practices to meet data quality objectives (DQOs).
- Review field reports.
- Perform and review audit reports.
- Prepare interim QA/QC compliance reports, as appropriate.

- Prepare, as appropriate, a QA/QC report in accordance with USEPA guidelines, including an evaluation of field and laboratory data and data usability reports.

2.2.3 Analytical Laboratories

- Supply proper sampling containers, labels, chain of custody (COC) forms and seals and shipping containers.
- Inventory incoming samples against COC forms. Maintain laboratory custody of samples.
- Perform sample analyses and associated laboratory QA/QC procedures.
- Strictly adhere to all protocols in the QAPP.

Project Manager

- Serve as primary communication link between Beazer Consultants and laboratory technical staff.
- Monitor workloads and maintain availability of resources.
- Oversee preparation of analytical reports.
- Supervise in-house COC.

Quality Assurance Coordinator

- Supervise personnel reviewing and inspecting all project-related laboratory activities.
- Conduct audits of all laboratory activities.

2.2.4 USEPA

Project Manager

- Coordinate USEPA review and approval of Site-related documents (e.g., work plans, reports, etc.).
- Monitor progress of Site activities.
- Facilitate compliance with regulatory requirements and remedial action objectives for the Site.
- Manage USEPA internal and consultant resources, including their compliance with QAPP requirements, as appropriate.

Quality Assurance Coordinator

- Review and approve the QAPP.
- Review the QA/QC portion of any submitted report, as appropriate.
- Verify that all activities are performed in compliance with applicable federal and regional requirements.
- Perform field and laboratory audits, if necessary.

2.2.5 IEPA

Project Manager

- Provide IEPA review of Site-related documents (e.g., work plans, reports, etc.).
- Monitor progress of Site activities on behalf of the state agency.

3. Project Background

3.1 Site Location and Description

The Site is located along North Marion Street, immediately north of the City of Carbondale in Jackson County, Illinois (Figure 1). The Site is at the junction of Sections 9, 10, 15, and 16 of Township 9S, Range 1W. Land use surrounding the Site consists of a mixture of residential, agricultural, and commercial properties. The southern edge of the Site is bordered by the partially abandoned railroad track belonging to Illinois Central Gulf Railroad. A residential portion of the City of Carbondale, along with some vacant and wooded areas, lies south of the railroad property. The western edge of the Site is bounded by North Marion Street and another railroad track belonging to Illinois Central Gulf Railroad. North Marion Street cuts through the west end of the property and provides the primary means of access to the Site. The area further west of North Marion Street and the railroad track is a commercial area in the City of Carbondale. To the north and east, the Site is bounded by a combination of agricultural, vacant, and wooded areas.

3.2 Site History, Summary of Activities, and Current Status

The Site was operated as a wood-treating facility from approximately 1905 to 1991. When operations were discontinued in 1991, a number of facility components were decommissioned and interim response measures were taken to address certain impacted areas. The facility is currently inactive.

As a result of the former wood-treating operations, soil, groundwater, and sediment in portions of the Site have been impacted with various constituents from the wood-treating processes. A number of investigations have been conducted since the early 1980s to determine the nature and extent of impacted materials at the Site. The primary constituents of potential interest (COPIs) at the Site include certain metals, polycyclic aromatic hydrocarbons (PAHs), and pentachlorophenol. The primary media that have been affected by these constituents include soils, sediment, and shallow groundwater. In addition, creosote dense, non-aqueous phase liquid (DNAPL) is present in the subsurface in certain portions of the Site.

Between 2004 and 2006, Beazer has conducted or will conduct various remediation activities as part of the Resource Conservation and Recovery Act (RCRA) corrective action program for the Site, including the following activities:

- additional building/structure demolition in the former process area
- relocation of a portion of Glade Creek
- installation of a trench-based DNAPL barrier near the former Glade Creek channel
- construction of a containment cell within a Corrective Action Management Unit (CAMU) to consolidate/manage various materials generated during the remediation activities
- excavation of waste piles and surficial “coal tar” materials from various areas of the Site
- installation of a surface cover over the former Process Area
- installation of a DNAPL recovery well at RW-23
- excavation of visually impacted Glade Creek sediments

In addition to the remedial construction, various operation, monitoring, and maintenance (OMM) activities are also performed at the Site. This includes a groundwater monitoring program that includes well gauging and sampling and analysis for selected Site wells. It also includes periodic operation and maintenance of certain remedial components, such as:

- removal of accumulated product from the trench-based DNAPL barrier, the RW-23 DNAPL recovery system, and well R-13E
- removal of accumulated leachate within the CAMU containment cell
- onsite treatment of generated waters (e.g., cogenerated water from DNAPL collection, impacted runoff from construction activities, containment cell leachate, well purge water, etc.) and discharge to a publicly owned treatment works
- National Pollutant Discharge Elimination System monitoring of stormwater discharge points

Certain of these OMM activities (e.g., groundwater monitoring) include sampling and analysis procedures subject to the requirements of this QAPP. As further discussed in Sections 4 and 5, various other anticipated or potential future sampling and analysis activities may also be performed and will be subject to the requirements of this QAPP.

4. Project Description

This section discusses the various sampling/analysis activities that are anticipated to be conducted at the Site and for which this QAPP will be applicable.

4.1 Investigation Objectives and Anticipated Scope

The overall objectives of future sampling and analysis activities associated with the Remedial Support and Post-Remediation OMM activities are anticipated to include:

- further delineation of the nature and extent of potential Site-related impacts in isolated areas
- support remedial construction and documentation activities
- support the development and implementation of the Monitored Natural Recovery (MNR) component of the remedial approach, including supporting risk assessment activities
- provide groundwater monitoring, both under the current Interim Groundwater Monitoring Program (IGMP) and a revised program that will be developed for post-remediation groundwater monitoring
- support characterization of disposal requirements for materials subject to offsite transport and disposal

The ongoing/future sampling and analysis activities that are expected to be performed in support of these general objectives include:

- Implementation of the IGMP, which currently includes semiannual groundwater sampling (conducted in February and August of each year) in accordance with the scope of work documented in a letter from Blasland, Bouck & Lee, Inc. (BBL) to the USEPA dated July 9, 2001. Once remediation activities are complete, a revised groundwater/surface water monitoring program will be established as part of a Post-Remediation OMM Plan and will supersede the current IGMP[1].

---

[1] A draft *Groundwater Monitoring Plan*, which described the proposed scope of a long-term groundwater monitoring program, was submitted to the USEPA in November 2007.

- Assessment and delineation of potential impacts in the southern drainage ditches area. This includes sampling in accordance with the January 4, 2008 *Addendum to the June 15, 2006 Southern Drainage Ditches Report/Work Plan*[2], and. any follow-up investigations that may be required.

- Collection of sediment, floodplain soil, biota, and surface-water data from various water bodies to characterize post-remediation baseline conditions, support risk-based human health and/or ecological evaluations, and determine where MNR is required, in accordance with the December 2007 *Baseline Characterization Work Plan* and the June 22, 2007 *Large Pond Work Plan*, and any follow-up investigations that may be required.

- Collection of sediment, biota, and/or surface-water data from water bodies where MNR is determined to be required. The purpose of such monitoring will be to confirm that natural recovery is occurring in water bodies where MNR is deemed necessary. The scope of this sampling program is yet to be developed.

These various investigations will be subject to the requirements of this QAPP. As stated above, it is possible that the need for additional sampling and analysis activities may be identified as part of the ongoing remediation and/or future OMM-related activities. Unless otherwise stated in the specific work plans, such activities will also be subject to the requirements of this QAPP.

Table 1 presents a list of the analyte groups for which samples collected as part of the Remedial Support and Post-Remediation OMM activities may be analyzed. If additional analytical parameters are determined to be necessary as part of future investigations, information regarding those parameters will be identified as part of the respective work plan and/or through updates to this QAPP.

4.2 Approach

Details associated with planned or ongoing investigations have been provided in the respective work plans. Details regarding future investigations will similarly be identified as part of the associated work plans. The sampling approaches are/will be presented in the applicable work plans. Work plans will be submitted to the USEPA for approval prior to beginning sampling events.

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[2] Revised tables from the January 4, 2008 submittal were provided to the USEPA on February 1, 2008.

4.3 Project Schedule

The project schedule has been or will be presented in the respective work plans addressing each investigation activity.

5. Data Quality Objectives and Criteria for Measurement

5.1 DQO Process Overview

The DQO process, as described in the USEPA's *Guidance on Systematic Planning Using the Data Quality Objective Process* (EPA QA/G-4 Guidance Document; USEPA, February 2006), is intended to provide a "logical framework" for planning field investigations. This section addresses each of the seven sequential steps in the DQO process outlined in the EPA QA/G-4 Guidance Document.

Step 1: State the Problem

As indicated in Section 4.1, the objectives of the ongoing and anticipated future investigation activities are anticipated to include:

- further delineation of the nature and extent of potential Site-related impacts in isolated areas
- support remedial construction and documentation activities
- support the development and implementation of the MNR component of the remedial approach, including supporting risk assessment activities
- provide groundwater monitoring, both under the current IGMP and a revised program that will be developed for post-remediation groundwater monitoring
- support characterization of disposal requirements for materials subject to offsite transport and disposal

Step 2: Identify the Goals of the Study

The initial use of the data is descriptive (i.e., to assess distribution and concentration), and there is no decision point for this descriptive application other than, in some cases, comparison to established criteria (e.g., discharge requirements, delineation objectives, regulatory standards, MNR goals, etc.). Subsequent to review of the descriptive information, an exposure evaluation may be performed based on the analytical results. The decision in this case is to determine the potential presence and significance of complete exposure pathways based on the distribution and concentrations of COPIs present in a specific media or area of the Site (listed in Tables 2A, 2B, and 2C).

Step 3: Identify Information Inputs

Decision inputs incorporate both the concentration and distribution of COPIs in Site media. A fundamental basis for decision making is that a sufficient number of data points of acceptable quality must be available from the investigation to support the decision. Thus, the necessary inputs for the decision are: 1) the proportion of non-rejected (usable) data points; and 2) the quantity of data required to evaluate whether there is unacceptable risk to human health and the environment at the Site. Data usability will be determined in consideration of several factors, including completeness, general conformance with the requirements of this QAPP, consistency among data sets and with historical data, and the findings of data verification and validation processes, as discussed in Section 21. The second decision input will be determined through development and USEPA approval of applicable work plans.

Step 4: Define the Boundaries of the Study

A description of the Site location is provided in Section 3.1 and the Site boundaries are depicted on Figure 1. The anticipated area(s) within which a particular investigation will focus will be identified in the respective work plans, and may include all or a portion of the overall Site, as well as areas not within the Site boundary.

Step 5: Develop the Analytic Approach

The decision on whether data can be used for their intended purpose will be based on the data verification and validation results. Following verification and validation, the data will be flagged, as appropriate, and any use restrictions will be noted. The work plans have been/will be devised so that the loss of any single data point will not hinder the description of the distribution of COPIs or the use of the dataset as a whole. Given this, a reasonable decision rule would be that a dataset is usable if 90% of the data points are not rejected or otherwise deemed unusable. As an initial screening step, the usable data will be evaluated versus the potential applicable and/or appropriate screening levels summarized in Tables 2A, 2B, and 2C. The required reporting limits are also documented in these tables so that the lowest achievable detection limit will be reported by the laboratory and, where possible, will be at or below the guidance values and target cleanup levels. Applicable actions would be evaluated, if necessary, based on the results of the comparison to the screening values. Additional evaluations (e.g., comparison to background data, site-specific risk assessments, etc.) may also be performed in lieu of or in addition to the screening level comparison.

Step 6: Specify Performance or Acceptance Criteria

Specifications for this step call for: 1) giving forethought to corrective actions to improve data usability; and 2) understanding the representative nature of the sampling design. This QAPP has been designed to meet both specifications for this step. The sampling and analysis program has been developed based on a review of previous Site data and on knowledge of present Site conditions. Corrective actions are described within this document. The representative nature of the sampling designs has been/will be facilitated by discussions among professionals familiar with the Site and the appropriate government agencies through development and approval of applicable work plans.

This step in the DQO process establishes the acceptable probability of making an incorrect decision based on the input data. Sources of potential measurement error, types of errors, and potential corrective actions are also discussed.

Decision errors are based on the development of a null hypothesis (H_o) and alternative hypothesis (H_a) which establishes the baseline condition. For the Site, two null hypotheses have been established:

1. COPIs are present at the Site.

2. Source material is present at the Site.

A false acceptance decision error would occur if it were determined that COPIs or source material was present at the Site when, in fact, it was not present. Conversely, a false rejection decision error would occur if it were determined that COPIs or source material was not present at the Site when, in fact, it was present. With this scenario, a false rejection error is more severe because potentially hazardous material would be left as a result of the false rejection error. As such, a more stringent probability rate of 0.05 (5%) is established for a false rejection decision. A false acceptance rate would result in further action related to material that, in reality, did not require further action. The consequences of a false acceptance decision would result in increased investigation or evaluation costs that are possibly less than the potential liability associated with leaving source material or COPIs in place. As such the false acceptance decision rate for the Site is established at 0.2 (20%).

Sources of potential measurement error include:

- poor sample recovery
- surface or subsurface features (e.g., existing buildings or underground utilities) that may inhibit representative sampling
- incorrect sample collection, handling, transport, or analysis
- incorrect data reduction methods
- inadequate sampling design

Potential corrective actions for these measurement errors include:

- review of standard operating procedures (SOPs)
- alternative sample collection methods
- additional sampling points
- removal of surface or subsurface interferences
- review of data reduction methods by qualified professionals
- review of laboratory QA/QC procedures

Step 7: Develop the Plan for Obtaining Data

The overall QA objective is to develop and implement procedures for field sampling, COC, laboratory analysis, and reporting that will provide results to support the evaluation of the Site data. Specific procedures for sampling, COC, laboratory instrument calibration, laboratory analysis, data reporting, internal QC, audits, preventive maintenance of field equipment, and corrective action are described in other sections of this QAPP.

In certain instances, work plans may involve a phased approach to both sampling and analysis. This provides the opportunity to evaluate and focus each data collection step to optimize the overall data collection process.

A DQO summary for the sampling investigation efforts is presented in the following section. The summary consists of stated DQOs relative to data uses, data types, data quantity, sampling and analytical methods, and data measurement performance criteria.

5.2 Data and Reporting Categories

Three data categories have been defined to address various analytical data uses and the associated QA/QC effort and methods required to achieve the desired levels of quality. These categories are:

Screening Data: Screening data afford a quick assessment of Site characteristics or conditions. This DQO is applicable to data collection activities that involve rapid, non-rigorous methods of analysis and QA. This objective is generally applied to physical and/or chemical properties of samples, semi-quantitative determination of COPI levels, and preliminary health and safety assessment.

Screening Data with Definitive Confirmation: Screening data allow rapid identification and quantitation, although the quantitation can be relatively imprecise. This DQO is available for data collection activities that require qualitative and/or quantitative verification of a select portion of sample findings (10% or more). This objective can also be used to verify less rigorous laboratory-based methods.

Definitive Data: Definitive data are generated using analytical methods such as approved USEPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce raw data (e.g., chromatograms, spectra, digital values) in the form of paper printouts or computer-generated electronic files.

It is anticipated that both screening and definitive data categories will be used during the investigation. Field parameters (e.g., dissolved oxygen, turbidity, specific conductivity, temperature, pH that may be obtained during groundwater and/or surface water) will be determined using screening techniques. Sampling of macroinvertebrates will be evaluated by an expert aquatic entomologist and identified to the most practicable taxon (screening data with definitive confirmation). All remaining parameters are expected to be determined using definitive techniques.

For this project, three levels of data reporting have been defined. They are as follows:

Level 1 - Minimal Reporting: Minimal or "results only" reporting is used for analyses that, due either to their nature (i.e., field monitoring) or the intended data use (i.e., preliminary screening), do not generate or require extensive supporting documentation.

Level 2 - Modified Reporting: Modified reporting is used for analyses that are performed following standard USEPA-approved methods and QA/QC protocols. Based on the intended data use, modified reporting may require some supporting documentation, but not full Contract Laboratory Program- (CLP-) type reporting.

Level 3 - Full Reporting: Full CLP-type reporting is used for those analyses that, based on the intended data use, require full documentation.

The majority of analytical data are expected to be generated by laboratories approved under Beazer's laboratory vendor program. Based on the target constituents and media, this is anticipated to include Test America, Vista, Lotic, Nautilus, and CAS. The analytical results will be reported by the laboratory in the electronic data deliverable (EDD) format outline presented in Table 5. Complete data packages, including Form Is (results sheets) will be provided in hard copy and/or electronic (PDF) formats.

Table 1 identifies the laboratories anticipated to be used. In addition, Nautilus is anticipated to be used for the sediment toxicity testing and Lotic is anticipated to be used for taxonomic identification and enumeration of aquatic macroinvertebrates. In the event that specialty analyses are required or alternate laboratories are used for other purposes, the analytical laboratory(ies) will be specified in the associated work plan. In addition, other Beazer-approved laboratories may be utilized in the future. Unless otherwise specified, such laboratories would also be required to comply with the requirements of this QAPP.

5.3 Anticipated Sampling and Analysis Activities

This section provides additional details related to the anticipated sampling and analysis activities identified in Section 4.1 and for which this QAPP is applicable. As indicated in Section 1, if future Site activities result in the need for sampling and analysis activities beyond those currently envisioned and discussed in this QAPP, any quality-assurance-related information that is applicable to such activities, but is not addressed by this QAPP, will be summarized in the associated work plans and/or an addendum to this QAPP.

5.3.1 Groundwater Sampling/Analysis

As indicated in Section 4.1, groundwater sampling at the Site is currently conducted via the IGMP. Once remediation activities are complete, a revised groundwater monitoring program will be proposed as part of a Post-Remediation OMM Plan.

The current IGMP includes analysis of groundwater samples for:

- benzene, toluene, ethylbenzene, and xylenes (BTEX) by USEPA SW846 Method 8021
- polycyclic aromatic hydrocarbons (PAHs) and pentachlorophenol by USEPA Method 8270
- total phenolics by USEPA Method 9066
- total and dissolved arsenic, chromium, and copper by USEPA SW846 Method 6010

In addition to the above, field parameters testing may include specific conductance, temperature, and/or pH. Note that the groundwater sampling analyte list may be subject to modification during development of the Post-Remediation OMM Plan.

Data Use

The data generated as part of the groundwater sampling program is and/or will be used to comply with RCRA monitoring requirements, assess and monitor changes in groundwater conditions, and to compare groundwater analytical data at compliance points to applicable groundwater standards. Analytical data will be compared to applicable Tiered Approach to Corrective Action Objectives (TACO) Tier 1 Groundwater Remediation Objectives (35 IAC 742 Appendix B, Table E).

Data Quantity

The current IGMP includes collection and analysis of groundwater samples from 48 wells. The number of wells sampled may be subject to modification in consultation with the USEPA based on post-remediation monitoring objectives and requirements.

Sampling and Analytical Methods

Groundwater samples will be collected using low-flow sampling protocols. An SOP for low-flow groundwater sampling is provided in Attachment A. Field parameters will be measured with field-measurement devices. Laboratory analytical methods are specified in Table 1. Field parameters will be subject to Level 1 reporting and laboratory analytical data will be subject to Level 2 reporting. Consistent with the existing practice for the IGMP sampling, Level 2 reporting for future groundwater monitoring events will include case narratives, Form I data sheets, surrogate recovery data, internal standard recoveries, laboratory control sample (LCS) data, method blank data, and matrix spike/matrix spike duplicate (MS/MSD) data.

Measurement Performance Criteria

Precision and accuracy QC limits for chemical constituents used during data review to assess analytical performance are included in Table 3. Reporting limits are presented in Table 2A. Data representativeness is addressed by the sample quantities, repetitive nature of the sampling program, and locations identified in the work plans. Data comparability will be achieved through the use of standard USEPA-approved methods. Data completeness will be assessed at the conclusion of the analytical activities. Data will be verified and validated in accordance with the procedures outlined in Section 21.

5.3.2 Southern Drainage Ditches Sampling/Analysis

Sampling/analysis will be conducted to assess and delineate potential impacts in the southern drainage ditches area. This includes sampling in accordance with the January 4, 2008 Addendum to the June 15, 2006 Southern Drainage Ditches Report/Work Plan, and. any follow-up investigations that may be required. Samples are anticipated to be analyzed for:

- PAHs and pentachlorophenol by USEPA SW-846 Method 8270C
- polychlorinated dibenzo-p-dioxins/polychlorinated dibenzofurans (PCDDs/PCDFs) by USEPA SW-846 Method 8290
- arsenic by USEPA SW-846 Method 6010

Data Use

Sampling results will be used to assess the presence, extent, and magnitude of COPIs in the southern drainage ditches. If and as necessary, the data may also be used to support human health and/or ecological risk assessments and determine the need for and scope of remedial activities to address conditions in this area. As an initial screening, analytical data will be compared to applicable USEPA Ecological Screening Levels and TACO Tier 1 Soil Remediation Objectives.

Data Quantity

Sample quantities and parametric requirements are/will be summarized in the above-referenced and future work plans.

Sampling and Analytical Methods

Sampling methods were specified in the *Sampling and Analysis Plan (SAP) for Southern Drainage Ditches*, and generally include the use of hand augers and similar manually operated equipment to collect shallow soil samples for visual inspection and/or laboratory analysis. Analytical methods are specified in Table 1. Laboratory analytical data will be subject to Level 3 reporting.

Measurement Performance Criteria

Precision and accuracy QC limits for chemical constituents used during data review to assess analytical performance are included in Table 3. Reporting limits are presented in Table 2B. Data representativeness is addressed by the sample quantities and locations identified in the above-referenced and future work plans. Data comparability will be achieved through the use of standard USEPA-approved methods. Data completeness will be assessed at the conclusion of the analytical activities. Data will be verified and validated in accordance with the procedures outlined in Section 21.

5.3.3 Water Body Sampling/Analysis

Sediment, floodplain soil, surface-water, and/or biota samples are anticipated to be collected from various water bodies to characterize post-remediation baseline conditions, to evaluate risks to potential human health and ecological receptors, and to determine if and where MNR is required. This includes sampling in accordance with the December 2007 *Baseline Characterization Work Plan* and the June 22, 2007

Large Pond Work Plan, and any follow-up investigations that may be required. Following completion of the initial sampling, evaluation/reporting of the associated data, and discussions with the USEPA, additional water body monitoring may be conducted as part of a multi-sampling-event MNR program.

Based on the currently envisioned scope of work, the water body samples are anticipated to be analyzed for the following parameters:

1. Sediment

- PAHs[3] and pentachlorophenol by USEPA SW-846 Method 8270C
- selected metals by USEPA SW-846 Method 6010/7000
- total organic carbon by Walkley-Black
- PCDDs/PCDFs by USEPA SW-846 Method 8290
- toxicity (10-day tests with *Chrironomus tentans* and *Hyalella azteca*), and/or
- geotechnical including the following:
  - grain size (sieve and hydrometer) by American Society of Testing and Materials (ASTM) Method D1140/D422
  - Atterberg limits by ASTM Method D4318
  - bulk density by USACOE Method EM-1110-2-1906 Appendix 2, and/or
  - specific gravity by ASTM Method D854

2. Floodplain Soil

- PAHs and pentachlorophenol by USEPA SW-846 Method 8270C
- selected metals by USEPA SW-846 Method 6010/7000

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[3] Both sediment and sediment porewater samples will be analyzed for PAHs.

- total organic carbon by Walkley-Black, and/or
- PCDDs/PCDFs by USEPA SW-846 Method 8290

3. Surface Water

- PAHs and pentachlorophenol by USEPA SW-846 Method 8270C
- selected metals by USEPA SW-846 Method 6010/7000
- PCDDs/PCDFs by USEPA SW-846 8290, and/or
- total suspended solids by Method 160.2

4. Biota

- PAHs and pentachlorophenol by USEPA SW-846 Method 8270
- selected metals by USEPA SW-846 Method 6010/7000
- PCDDs/PCDFs by USEPA SW-846 Method 8290
- percent lipids by USEPA SW-846 Method 3541, and/or
- taxonomic identification and enumeration of aquatic macroinvertebrates

In addition to the above, field parameters testing for surface water may include may include temperature, pH, dissolved oxygen, specific conductivity, turbidity, and flow. Field parameters for biota may include a habitat assessment.

If additional constituents/parameters are added to the analyte list in the course of developing work plans, standard USEPA methods will be used, as applicable. To the extent possible, the types of analyses that may be included, including the associated analytical methods, are indicated in Table 1 such that the provisions of this QAPP would apply in the event of an expanded scope.

Data Use

The initial water body sampling data are anticipated to be used to characterize post-remediation baseline conditions, to evaluate risks to potential human health and ecological receptors, and to determine if and where MNR is required. If needed, sampling data collected as part of the MNR program will be used to verify that natural recovery is occurring.

Data Quantity

Sample quantities and parametric requirements are/will be summarized in the above-referenced and future work plans.

Sampling and Analytical Methods

Sampling methods are included in Attachment A and are/will be summarized in the above-referenced and future work plans. Laboratory analytical methods are specified above and in Table 1. Field parameters and biota taxonomic identification will be subject to Level 1 reporting and the remainder of the laboratory analytical data will be subject to Level 3 reporting.

Measurement Performance Criteria

Precision and accuracy QC limits for chemical constituents used during data review to assess analytical performance are included in Table 3. Reporting limits are presented in Tables 2A, 2B, and 2C. Data representativeness is addressed by the sample quantities and locations identified in the Work Plan. Data comparability will be achieved through the use of standard USEPA-approved methods. Data completeness will be assessed at the conclusion of the analytical activities. Data will be verified and validated in accordance with the procedures outlined in Section 21.

6. Special Training Requirements/Certification

In compliance with the Occupational Safety and Health Administration's (OSHA's) final rule, "Hazardous Waste Operations and Emergency Response," 29 Code of Federal Regulations §1910.120(e), all personnel performing sampling activities at the Site will have completed the requirements for OSHA 40-Hour Hazardous Waste Operations and Emergency Response training. Persons in field supervisory positions will have also completed the additional OSHA 8-Hour Supervisory Training. Additional training requirements for field and laboratory personnel are summarized in the Field Sampling SOPs (Attachment A), Laboratory SOPs (Attachment B), and Laboratory Quality Assurance Manuals (Attachment C).

7. Documentation and Records

7.1 General

Samples of the various media will be collected as described in the applicable work plans. General descriptions of documentation and reporting requirements are presented below; additional details may be provided in the applicable work plans.

7.2 Sample Designation System

Samples will be identified with a unique designation system that will facilitate sample tracking. The sample designation system to be employed during the sampling activities will be consistent, yet flexible enough to accommodate unforeseen sampling events and conditions. An alpha-numeric system will be used by field personnel to assign each sample with a unique sample identification number. To the extent possible, the sample ID will identify the type of sample (e.g., soil, sediment, groundwater, surface water, biota, etc.) and the specific sample location.

Where necessary, the code system will be supplemented to accommodate additional sample identification information. For example, the code for soil and sediment samples will include a qualifier to identify the associated sample depth interval (e.g., 0 to 0.5 foot). The date and time each sample is collected will also be recorded to generate a unique designation for each sample.

Additional sample volumes collected for matrix spike (MS) and matrix spike duplicate (MSD) analysis will be noted on the COC forms, and the associated additional sample containers will be labeled with the appropriate suffix (MS or MSD). Rinse blanks will use the prefix “RB.” Field duplicates will use the prefix “DUP.” Duplicate sample locations will not be identified, and the laboratory will analyze them as “blind” quality control samples.

7.3 Field Documentation

Field personnel will provide comprehensive documentation covering various aspects of field sampling, field analysis, and sample COC. This documentation consists of a record that allows reconstruction of field events to aid in the data review and interpretation process. Documents, records, and information relating to the performance of the field work will be retained in the project file.

The various forms of documentation to be maintained throughout the action include:

- Daily Production Documentation – A field notebook(s), consisting of a waterproof, bound notebook(s), will contain a record of all activities performed at the Site.

- Sampling Information – Detailed notes will be made as to the exact sampling location, physical observations, and weather conditions (as appropriate).

- Sample COC – COC forms will provide the record of responsibility for sample collection, transport, and submittal to the laboratory. COC forms will be filled out for each sampling event, or at the end of each day of sampling by the field personnel responsible for sample custody. In the event that samples are relinquished by the designated sampling person to other sampling or field personnel, the COC form will be signed and dated by the appropriate personnel to document the sample transfer. The original COC form will accompany the samples to the laboratory, and copies will be forwarded to the project files. A sample COC form is included in Appendix B of this QAPP.

  Persons will have custody of samples when the samples are in their physical possession, in their view after being in their possession, or in their physical possession and secured so they cannot be tampered with. In addition, when samples are secured in a restricted area accessible only to authorized personnel, they will be deemed to be in the custody of such authorized personnel.

- Field Equipment, Calibration, and Maintenance Logs – To document the calibration and maintenance of field instrumentation, calibration and maintenance logs will be maintained for each piece of field equipment that is not factory calibrated.

7.4 Laboratory Documentation Files

7.4.1 Laboratory Project Files

Laboratories will establish files for pertinent data, including correspondence, faxed information, phone logs, and COC forms. Each laboratory will retain its project files and data packages for a period not less than five years. Beazer and/or its designated representative will retain copies of the analytical data report for a minimum of 10 years after commencement of construction of any remedial action.

7.4.2 Laboratory Logbooks

Workbooks, bench sheets, instrument logbooks, and instrument printouts will be used to trace the history of samples through the analytical process and to document important aspects of the work, including the associated quality controls. As such, logbooks, bench sheets, instrument logs, and instrument printouts will be part of the permanent record of the laboratory.

Each page or entry will be dated and initialed by the analyst at the time of entry. Errors in entry will be crossed out in indelible ink with a single stroke, corrected without the use of white-out or by obliterating or writing directly over the erroneous entry, and initialed and dated by the individual making the correction. Pages of logbooks that are not used will be completed by lining out unused portions.

Information regarding the sample, analytical procedures performed, and the results of the testing will be recorded on laboratory forms or personal notebook pages by the analyst. These notes will be dated and will also identify the analyst, the instrument used, and the instrument conditions.

Laboratory notebooks will be periodically reviewed by the laboratory group leaders for accuracy, completeness, and compliance with this QAPP. All entries and calculations will be verified by the laboratory group leader. If all entries on the pages are correct, the laboratory group leader will initial and date the pages. Corrective action will be taken for incorrect entries before the laboratory group leader signs the laboratory notebook.

7.4.3 Computer Tape and Hard Copy Storage

All electronic files and deliverables will be retained by the laboratory for not less than five years; hard-copy data packages (or electronic copies) will also be retained for not less than five years.

7.5 Data Reporting Requirements

Data will be reported both in the field and by the analytical laboratory, as described below.

7.5.1 Field Data Reporting

Information collected in the field through visual observation, manual measurement, and/or field instrumentation will be recorded in field notebooks or data sheets and/or on forms. Such data will be reviewed by the appropriate Task Manager for adherence to the associated work plan and for consistency. Concerns identified as a result of this review will be discussed with the field personnel, corrected if possible, and (as necessary) incorporated into the data evaluation process.

If applicable, field data forms and calculations will be processed and included in appendices to the appropriate reports (when generated). The original field logs, documents, and data reductions will be kept in the project files.

7.5.2 Laboratory Data Reporting

Data reports for all parameters will include, at a minimum, the following items:

Narrative: Summary of activities that took place during the course of sample analysis, including the following information:

- laboratory name and address
- date of sample receipt
- cross reference of laboratory identification number to contractor sample identification
- analytical methods used
- deviations from specified protocol
- corrective actions taken

Included with the narrative will be any sample handling documents, including field and internal COC forms, air bills, and shipping tags.

Analytical Results: These will be reported according to analysis type and will include the following information, as applicable:

- sample ID
- laboratory ID
- date of collection
- date of receipt
- date of extraction
- date of analysis
- detection limits

Sample results on the report forms will be corrected for dilutions. Soil, sediment, and biota data will be reported on a dry-weight basis. Unless otherwise specified, all results will be reported uncorrected for blank contamination.

The data for CLP-equivalent reporting will be expanded to include supporting documentation necessary to provide a CLP-equivalent package. This additional documentation will include, but not be limited to, raw data required to recalculate any result, including instrument printouts and quantitation reports. The report also will include standards used in calibration and calculation of analytical results; sample extraction, digestion, and other preparation logs; standard preparation logs; instrument run logs; and moisture content calculations.

The analytical results will be reported by the laboratory in the EDD format outlined in Table 5. Complete data packages, including Form Is (results sheets) will be provided in hard copy and/or electronic (PDF) formats.

7.6 Project File

Each organization involved in this project will maintain project-related documentation in dedicated project files. Filing systems may vary among the various parties, but, at a minimum, will include copies of correspondence, data, reports, field notes, and other pertinent information, as appropriate. Files will be maintained such that project-related information can be located and retrieved when needed.

8. Sampling Process Design

The general purpose and scope of the various sampling/analysis activities that are anticipated to be conducted at the Site and for which this QAPP will be applicable are discussed in Sections 4 and 5 of this QAPP. Additional details regarding the scope, purpose, and methods of the sampling activities are/will be presented in the work plans associated with specific investigations and monitoring programs.

9. Sampling Method Requirements

The general purpose and scope of the various sampling/analysis activities that are anticipated to be conducted at the Site and for which this QAPP will be applicable are discussed in Sections 4 and 5 of this QAPP. Additional details regarding the scope, purpose, and methods of the sampling activities are/will be presented in the work plans associated with specific investigations and monitoring programs. Field sampling SOPs are also included in Attachment A to this QAPP.

10. Sampling Handling and Custody Requirements

10.1 Sample Containers and Preservation

Appropriate sample containers, preservation methods, and laboratory holding times for are shown in Table 4.

The analytical laboratory will supply appropriate sample containers and preservatives, as necessary. The bottles will be purchased pre-cleaned according to USEPA Office of Solid Waste and Emergency Response Directive 9240.05A requirements. The field personnel will be responsible for properly labeling containers and preserving samples (as appropriate). Sample labeling procedures are discussed in Section 10.2.2.

10.2 Field Custody Procedures

The objective of field sample custody is to protect samples from tampering from the time of sample collection through the time of transport to the analytical laboratory. Persons will have custody of samples when the samples are in their physical possession, in their view after being in their possession, or in their physical possession and secured so they cannot be tampered with. In addition, when samples are secured in a restricted area accessible only to authorized personnel, they will be deemed to be in the custody of such authorized personnel.

Field custody documentation consists of both field logbooks and field COC forms.

10.2.1 Field Logbooks

Field logbooks will provide the means of recording the data-collecting activities that are performed. As such, entries will be described in as much detail as possible so that persons going to the Site could reconstruct a particular situation without reliance on memory.

Field logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel but will be stored in a secure location when not in use. Each logbook will be identified by the project-specific document number. The title page of each logbook will contain the following:

- person to whom the logbook is assigned
- logbook number

- project name
- project start date(or logbook start date in the case of multiple volumes)
- project (or logbook) end date

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather conditions, names of all sampling team members present, level of personal protection being used, and signature of the person making the entry will be provided. The names of visitors to the Site and field sampling or investigation team personnel, as well as the purpose of their visit, will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. Entries will be made in ink, with no erasures. If an incorrect entry is made, the information will be crossed out with a single strike mark. Whenever a sample is collected or a measurement is made, a detailed description of the sample location will be recorded. The number of photographs taken, if any, will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the sampling procedures documented in Section 9. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume, and number of containers. Sample identification numbers will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description.

10.2.2 Sample Labeling

Preprinted sample labels will be affixed to sample bottles prior to delivery at the sampling Site. The following information is required on each sample label:

- project name
- date collected
- time collected
- location
- sampler

- analysis to be performed
- preservative
- sample ID/number

10.2.3 Field Chain-of-Custody Forms

Completed COC forms will be required for all samples to be analyzed. COC forms will be initiated by the sampling crew in the field. The COC forms will contain the unique sample identification number, sample date and time, sample description, sample type, preservation (if any), and analyses required. The original COC form will accompany the samples to the laboratory. Copies of the COC will be made prior to shipment (or multiple copy forms will be used) for field documentation. The COC forms will remain with the samples at all times. The samples and signed COC forms will remain in the possession of the sampling crew until the samples are delivered to the express carrier (e.g., FedEx), hand delivered to a mobile or permanent laboratory, or placed in secure storage.

Sample labels will be completed for each sample using waterproof ink. The labels will include the information listed in Section 10.2.2, above. The completed sample labels will be affixed to each sample bottle and covered with clear tape.

Whenever samples are split with a government agency or other party, a separate COC will be prepared for those samples and marked to identify the party with whom the samples are being split. The person relinquishing the samples to the facility or agency should request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "Received by" space.

10.3 Management of Investigation-Derived Materials and Wastes

Investigation-derived wastes (IDW) include soils, groundwater, materials, and personal protective equipment (PPE) generated during field investigation activities. Where necessary to maintain the safe, efficient, and environmentally protective performance of work, the management of investigation-derived materials and wastes will be performed consistent with the *Guide to Management of Investigation-Derived Wastes,* 9345.3-03FS (USEPA, 1992). Unless otherwise specified in a specific work plan, IDW will be managed as follows:

- Disposable equipment (including PPE) and debris will be containerized, appropriately labeled, and disposed of according to applicable requirements depending on the nature of the materials.

- Purged groundwater and water generated during equipment cleaning will be containerized, transported to, and treated via the onsite water treatment system in accordance with the system's operational permits.

- Excess soil cuttings associated with the drilling of soil borings will be collected, containerized, appropriately labeled, and disposed of in accordance with applicable regulations based on the characteristics of the material.

10.4 Packing, Handling, and Shipping Requirements

Sample packaging and shipment procedures are designed so that the samples and COC will arrive at the laboratory intact and in a timely manner.

Samples will be packaged for shipment as outlined below:

- Securely affix the sample label to the container with clear packing tape.

- Check the caps on the sample container to confirm that it is properly sealed.

- Wrap the sample container cap with clear packing tape to prevent the label from becoming loose.

- Complete the COC form with the required sampling information and confirm that the recorded information matches the sample labels. NOTE: If the designated sampler relinquishes the samples to other sampling or field personnel for packing or other purposes, the sampler will complete the COC prior to this transfer. The appropriate personnel will sign and date the COC form to document the sample custody transfer.

- Using duct tape, secure the outside drain plug at the bottom of the cooler.

- Wrap sample containers in bubble wrap or other cushioning material.

- Place 1 to 2 inches of cushioning material at the bottom of the cooler.

- Place the sealed sample containers into the cooler.

- Place ice in plastic bags and seal. Place loosely in the cooler.
- Fill the remaining space in the cooler with cushioning material.
- Place COC forms in a plastic bag and seal. Tape the forms to the inside of the cooler lid.
- Close the lid of the cooler, lock, and secure with duct tape.
- Wrap strapping tape around both ends of the cooler at least twice.
- Mark the cooler on the outside with the shipping address and return address, affix "Fragile" labels, and draw (or affix) arrows indicating "this side up." Cover the labels with clear plastic tape.
- Place a signed custody seal over the sample cooler lid.

Samples will be packaged by the field personnel and transported as low-concentration environmental samples. EnCore® samples must be shipped on the day they are collected. The other samples will be hand delivered or delivered by an express carrier within 48 hours of the time of collection. Shipments will be accompanied by the COC form identifying the contents. The original form will accompany the shipment; copies will be retained by the sampler for the sampling office records. If the samples are sent by common carrier, a bill of lading will be used. Receipts or bills of lading will be retained as part of the permanent project documentation. Commercial carriers are not required to sign off on the COC form as long as the forms are sealed inside the sample cooler, and the custody seals remain intact.

Sample custody seals and packing materials for filled sample containers will be provided by the analytical laboratory. The filled, labeled, and sealed containers will be placed in a cooler on ice and carefully packed to eliminate the possibility of container breakage.

10.5 Laboratory Custody Procedures

10.5.1 General

Upon sample receipt, laboratory personnel will be responsible for sample custody. The original field COC form will accompany all samples requiring laboratory analysis. The laboratory will use COC guidelines described in the USEPA guidance documents. Samples will be kept secured in the laboratory until all stages of analysis are complete.

All laboratory personnel having samples in their custody will be responsible for documenting and maintaining sample integrity.

10.5.2 Sample Receipt and Storage

Immediately upon sample receipt, the laboratory sample custodian will verify the integrity of the cooler seal, open the cooler, and compare the contents against the field COC. If a sample container is missing, a sample container is received broken, the sample is in an inappropriate container, or the sample has not been preserved by appropriate means the designated representative of the organization that performed the sampling will be notified immediately. The laboratory sample custodian will be responsible for logging the samples in, assigning a unique laboratory identification number to each sample, labeling the sample bottle with the laboratory identification number, and moving the sample to an appropriate storage location to await analysis. The project name, field sample code, date sampled, date received, analysis required, storage location and date, and action for final disposition will be recorded in the laboratory tracking system. Relevant custody documentation will be placed in the project file.

10.5.3 Sample Analysis

Analysis of an acceptable sample will be initiated by worksheets that contain all pertinent information for analysis. The analyst will sign and date the laboratory COC form when removing the samples from storage.

Samples will be organized into sample delivery groups (SDGs) by the laboratory. An SDG may contain up to 20 field samples (field duplicates, trip blanks, and rinse blanks are considered field samples for the purposes of SDG assignment). All field samples assigned to a single SDG will be received by the laboratory over a maximum of 7 calendar days and must be processed through the laboratory (preparation, analysis, and reporting) as a group. Every SDG must include a minimum of one Site-specific MS/MSD pair, which shall be received by the laboratory at the start of the SDG assignment.

11. Analytical Method Requirements

11.1 Field Parameters and Methods

The type of field parameters to be measured, and the method(s) for doing so, may vary depending on the nature of a particular field investigation. Field parameters and measurement methods will be specified in work plans associated with the various investigations.

11.2 Laboratory Parameters and Methods

The methods listed below include the range of analyses expected to be performed. The associated laboratory SOPs can be found in Attachment B.

Laboratory analytical requirements presented in the sub-sections below include a general summary of requirements, specifics related to each sample medium to be analyzed, and details of the methods to be used for this project. USEPA SW-846 methods with QA/QC and reporting deliverable requirements will be used for all analytes, as applicable. Note that the analytical methods referenced throughout this QAPP may be subject to change if an updated USEPA-approved method is available and more applicable.

The following tables summarize the general analytical requirements:

Table	Title
Table 1	Sample Quantities and Quality Control Frequencies
Table 2A	Parameters, Methods, and Target Reporting Limits (Groundwater and Surface Water)
Table 2B	Parameters, Methods, and Target Reporting Limits (Soil and Sediment)
Table 2C	Parameters, Methods, and Target Reporting Limits (Biota)
Table 3	Analytical Quality Control Limits
Table 4	Sample Containers, Preservation Methods, and Holding Times
Table 5	Electronic Data Deliverable (EDD) Format

Note that the media-specific analytical parameters listed in the above-referenced tables and discussed below represent those that, based on current Site knowledge, may possibly be analyzed for as part of future investigations. Analytical parameters associated with a particular investigation or activity will be indicated in the associated work plan.

The following specialty analyses/tests are not included in the above-referenced tables:

- soil/sediment geotechnical tests, including grain size (ASTM D1140/D422), Atterberg limits (ASTM D 4318), bulk density (U.S. Army Corps of Engineers [USACOE] EM-1110-2-1906 Appendix 2), and specific gravity (ASTM D854)
- sediment toxicity tests (10-day tests with *Chironomus tentans* and *Hyalella azteca*)
- taxonomic identification and enumeration of aquatic macroinvertebrates.

11.2.1 Sample Matrices

11.2.1.1 Groundwater

Analyses will be performed following the methods listed in Table 1. Analytical results for all analyses will be reported in units identified in Table 2A.

11.2.1.2 Surface Water

Analyses will be performed following the methods listed in Table 1. Analytical results for all analyses will be reported in the units identified in Table 2A.

11.2.1.3 Soil

Analyses will be performed following the methods listed in Table 1. Results will be reported as dry weight, in the units presented in Table 2B. Moisture content will be reported separately.

11.2.1.4 Sediment

Analyses will be performed following the methods listed in Table 1. Results will be reported as dry weight, in the units presented in Table 2B. Moisture content will be reported separately.

11.2.1.5 Biota

Analyses will be performed following the methods listed in Table 1. Results will be reported as dry weight, in the units presented in Table 2C. Moisture content will be reported separately.

11.2.2 Analytical Requirements

The primary sources to describe the analytical methods to be used during the investigation are provided in USEPA SW-846 – Test Methods for Evaluating Solid Waste, Third Edition QA/QC, and reporting deliverables requirements.

12. Quality Control Requirements

12.1 Quality Assurance Indictors

The overall QA objective for this QAPP is to develop and implement procedures for sampling, COC, laboratory analysis, instrument calibration, data reduction and reporting, internal QC, audits, preventive maintenance, and corrective action, such that valid data will be generated. These procedures are presented or referenced in the following sections. Specific QC checks are discussed in Section 12.2.

QA indicators are generally defined in terms of five parameters:

1. Representativeness
2. Comparability
3. Completeness
4. Precision
5. Accuracy

Each parameter is defined below. Specific objectives for the Site actions are set forth in other sections of this QAPP, as referenced below.

12.1.1 Representativeness

Representativeness is the degree to which sampling data accurately and precisely represent Site conditions, and is dependent on sampling and analytical variability and the variability of environmental media at the Site. The sampling activities have been/will be designed to assess the presence of the chemical constituents at the time of sampling. The applicable work plans will present the rationale for sample quantities and location and also discuss field sampling methods. This QAPP discusses laboratory analytical methodologies. The use of the prescribed field and laboratory analytical methods with associated holding times and preservation requirements are intended to provide representative data.

12.1.2 Comparability

Comparability is the degree of confidence with which one data set can be compared to another. To the extent possible, comparability between phases of the sampling (if additional phases are required) will be maintained through consistent use of the sampling and analytical methodologies set forth in the applicable work plans and this QAPP, established QA/QC procedures, and the utilization of appropriately trained personnel. This will not preclude the use of improved standards or methodologies to incorporate any technological advances in the areas of field sampling and/or laboratory analysis.

12.1.3 Completeness

Completeness is defined as a measure of the amount of valid data obtained from an event and/or investigation compared to the total amount that was obtained. This will be determined upon final assessment of the analytical results, as discussed in Section 12.6.

12.1.4 Precision

Precision is a measure of the reproducibility of sample results. The goal is to maintain a level of analytical precision consistent with the objectives of the action. To maximize precision, sampling and analytical procedures will be followed. All work for the Site actions will adhere to established protocols presented in the applicable work plans and this QAPP. Checks for analytical precision will include the analysis of MS/MSDs, laboratory duplicates, and field duplicates. Checks for field measurement precision will include duplicate field measurements. Further discussion of precision QC checks is provided in Section 12.4.

12.1.5 Accuracy

Accuracy is a measure of how close a measured result is to the true value. Both field and analytical accuracy will be monitored through initial and continuing calibration of instruments. In addition, reference standards, MSs, blank spikes, and surrogate standards will be used to assess the accuracy of the analytical data.

12.2 Field Quality Control Checks

12.2.1 Field Measurements

To verify the quality of data obtained with field instrumentation, data will be compared to previous measurement data where available. If necessary, duplicate measurements will be obtained and reported.

12.2.2 Sample Containers

Certified-clean sample containers (I-Chem 300 Series or equivalent) will be supplied by the laboratory. Certificates of analysis will be filed in the project file.

12.2.3 Field Duplicates

Field duplicates will be collected from the different Site media to verify the reproducibility of the sampling methods. Field duplicates will be prepared by placing well-homogenized aliquots (except samples for volatile organic compound [VOC] analysis) from the same sampling location into individual sample containers, which will then be submitted blind to the laboratory. Field duplicate water samples and soil samples for VOC analysis will constitute co-located samples rather than homogenized aliquots. In general, field duplicates will be analyzed at a 10% frequency (every 10 samples) for the chemical constituents.

12.2.4 Rinse Blanks

Rinse blanks are used to monitor the cleanliness of the sampling equipment and the effectiveness of the cleaning procedures. Rinse blanks will be prepared and submitted for analysis once per day per matrix, or once per 20 samples, whichever is more frequent. Rinse blanks will be prepared by filling sample containers with analyte-free water (supplied by the laboratory) that has been routed through a cleaned sampling device. When dedicated sampling devices or sample containers are used to collect the samples, rinse blanks will not be necessary.

12.3 Analytical Laboratory Quality Control Checks

12.3.1 General

Internal laboratory QC checks will be used to monitor data integrity. These checks will include method blanks, MS/MSDs, spike blanks, internal standards, surrogate samples, calibration standards, and reference standards. Project quality control limits for duplicates and MSs are identified in Table 3. Laboratory control charts will be used to determine long-term instrument trends.

12.3.2 Method Blanks

Sources of contamination in the analytical process, whether specific analyses or interferences, must be identified, isolated, and corrected. The method blank is useful in identifying possible sources of contamination within the analytical process. For this reason, it is necessary that the method blank be initiated at the beginning of the analytical process and encompass all aspects of the analytical work. As such, the method blank would assist in accounting for any potential contamination attributable to glassware, reagents, instrumentation, or other sources that could affect sample analysis. One method blank will be analyzed with each analytical series associated with no more than 20 samples.

12.3.3 MS/MSDs

MS/MSDs will be used to measure the accuracy of analyte recovery from the sample matrices and will be Site-specific. MSD pairs will be analyzed at a 5% frequency (every 20 samples or once each week, whichever comes is greater).

When MS recoveries are outside QC limits, associated control sample and surrogate spike recoveries will be evaluated, as applicable, to attempt to verify the reason for the deviation and to determine the effect on the reported sample results.

12.3.4 Surrogate Spikes

Surrogates are compounds that are unlikely to occur under natural conditions but that have properties similar to the analytes of interest. This type of control is primarily used for organic samples analyzed by gas chromatography/mass spectrometry (GC/MS) and GC methods and is added to the samples prior to purging or extraction. The surrogate spike is utilized to provide broader insight into the proficiency and efficiency of an analytical method on a sample-specific basis. This control reflects analytical conditions that may not be attributable to sample matrix.

If surrogate spike recoveries exceed specified QC limits, the analytical results must be evaluated thoroughly in conjunction with other control measures. In the absence of other control measures, the integrity of the data may not be verifiable, and reanalysis of the samples with additional control may be necessary.

Surrogate spike compounds will be selected utilizing the guidance provided in the analytical methods.

12.3.5 Serial Dilutions

For inorganics, serial dilutions are the dilutions of a sample by a known factor. When corrected by the dilution factor, the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

12.3.6 Contract Required Detection Limit (CRDL) Check Standards

For inorganics, the CRDL check standard is a single parameter or multi-parameter standard solution prepared at the contract required quantitation limit (CRQL) and used to verify the instrument calibration at low levels.

12.3.7 Interference Check Standards (ICS)

For inorganics, the ICS is a solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.

12.3.8 Laboratory Duplicates

For inorganics, laboratory duplicates will be analyzed to assess laboratory precision. Laboratory duplicates are defined as a separate aliquot of an individual sample that is analyzed as a separate sample. Table 1 presents an estimated number of laboratory duplicates for each applicable parameter.

12.3.9 Calibration Check Standards

Calibration check standards analyzed within a particular analytical series provide insight regarding instrument stability. A calibration check standard will be analyzed at the beginning and end of an analytical series, or periodically throughout a series containing a large number of samples.

In general, calibration check standards will be analyzed after every 12 hours or more frequently, as specified in the applicable analytical method. If results of the calibration check standard exceed specified tolerances, samples analyzed since the last acceptable calibration check standard will be reanalyzed.

Laboratory instrument calibration standards will be selected utilizing the guidance provided in the analytical methods, as summarized in Section 14.

12.3.10 Reference Standards/Control Samples

Reference standards are standards of known concentration and are independent in origin from the calibration standards. The intent of reference standard analysis is to provide insight into the analytical proficiency within an analytical series. This includes preparation of calibration standards, validity of calibration, sample preparation, instrument set-up, and the premises inherent in quantitation. Reference standards will be analyzed at the frequencies specified within the analytical methods.

12.4 Data Precision Assessment Procedures

Field precision is difficult to measure because of temporal variations in field parameters. However, precision will be controlled through the use of experienced field personnel, properly calibrated meters, and duplicate field measurements. Field duplicates will be used to assess precision for the entire measurement system, including sampling, handling, shipping, storage, preparation, and analysis.

Laboratory data precision will be monitored through the use of MS/MSD and laboratory duplicate sample analyses.

The precision of data will be measured by calculation of the relative percent difference (RPD) by the following equation:

$$RPD = \frac{(A-B)}{(A+B)/2} \times 100$$

Where:

A = Analytical result from one of two duplicate measurements

B = Analytical result from the second measurement

Precision objectives for duplicate analyses are identified in Table 3.

12.5 Data Accuracy Assessment Procedures

The accuracy of field measurements will be controlled by experienced field personnel, properly calibrated field meters, and adherence to established protocols. The accuracy of field meters will be assessed by review of calibration and maintenance logs.

Laboratory accuracy will be assessed via the use of MSs, surrogate spikes, and reference standards. Where available and appropriate, QA performance standards will be analyzed periodically to assess laboratory accuracy. Accuracy will be calculated in terms of percent recovery as follows:

$$\% \text{ Recovery} = \frac{A-X}{B} \times 100$$

Where:

A = Value measured in spiked sample or standard

X = Value measured in original sample

B = True value of amount added to sample or true value of standard

This formula is derived under the assumption of constant accuracy between the original and spiked measurements. Accuracy objectives for MS recoveries are identified in Table 3.

12.6 Data Completeness Assessment Procedures

Completeness of a field or laboratory data set will be calculated by comparing the number of valid sample results generated to the total number of results generated.

$$\text{Completeness} = \frac{\text{Number valid results}}{\text{Total number of results generated}} \times 100$$

As a general guideline, overall project completeness is expected to be at least 90%. The assessment of completeness will require professional judgment to determine data usability for intended purposes.

13. Instrument/Equipment Testing, Inspection, and Maintenance Requirements

13.1 General

This section summarizes the testing and maintenance schedules have been developed for both field and laboratory instruments.

13.2 Field Instruments and Equipment

Prior to field sampling, each piece of field equipment will be inspected to confirm that it is operational. If the equipment is not operational, it will be serviced prior to its use. Meters that require charging or batteries will be fully charged or have fresh batteries. If instrument servicing is required, it is the responsibility of the appropriate Task Manager or field personnel to follow the maintenance schedule and arrange for timely service. Field instruments will be maintained according to the manufacturers' instructions.

Operation, maintenance, calibration, and any problems and repairs associated with field instruments and equipment will be recorded on log sheets or in a field notebook. The Task Managers will review calibration and maintenance information.

All measuring and test equipment to be used in support of the sampling activities that directly affect the quality of the analytical data will be subject to preventative maintenance measures that minimize equipment downtime. Equipment will be examined to certify that it is in operating condition. This includes checking the manufacturer's operating manual to confirm that all maintenance requirements are being observed. Field notes from previous sampling events will be reviewed to verify that any prior equipment problems are not overlooked and that any necessary repairs to equipment have been carried out.

Field equipment returned from a Site will be inspected to confirm that it is in working order. The inspection will be recorded in the logbook or field notebooks, as appropriate. It will also be the obligation of the last user to record any equipment problems in the logbook. Non-operational field equipment will either be repaired or replaced. Appropriate spare parts will be made available for field meters. Consultant-/subcontractor-owned or leased equipment maintenance will be in accordance with the manufacturer's instructions.

13.3 Laboratory Instruments and Equipment

13.3.1 General

Laboratory instrument and equipment documentation procedures include details of any observed problems, corrective measure(s), routine maintenance, and instrument repair (including information regarding the repair and the name of the individual who performed the repair).

Preventive maintenance of laboratory equipment generally will follow the guidelines recommended by the manufacturer. A malfunctioning instrument will be repaired immediately by in-house staff or through a service call from the manufacturer.

13.3.2 Instrument Maintenance

Maintenance schedules for laboratory equipment adhere to each manufacturer's recommendations. Records reflect the complete history of each instrument and specify the time frame for future maintenance. Major repairs or maintenance procedures are performed through service contracts with the manufacturer or qualified contractors. Paperwork associated with service calls and preventative maintenance calls will be kept on file by the laboratory.

Laboratory Systems Managers are responsible for the routine maintenance of instruments used in the particular laboratory. Any routine preventative maintenance carried out is logged into the appropriate logbooks. The frequency of routine maintenance is dictated by the nature of samples being analyzed, the requirements of the method used, and/or the judgment of the Laboratory Systems Manager.

All major instruments are backed up by comparable (if not equivalent) instrument systems in the event of unscheduled downtime. An inventory of spare parts is also available to minimize equipment/instrument downtime.

14. Instrument Calibration and Frequency

14.1 Field Instruments and Equipment

The calibration of field instruments is governed by specific SOPs documented in the Field Sampling Plan (FSP) for the applicable field analysis method, and such procedures take precedence over the following discussion.

Field personnel are responsible for confirming that a master calibration/maintenance log is maintained following the procedures specified for each measuring device. Where applicable, each log will include, at a minimum, the following information:

- name of device and/or instrument calibrated
- device/instrument serial/identification numbers
- calibration method
- tolerance
- calibration standard used
- frequency of calibration
- date(s) of calibration(s)
- name of person(s) performing calibration(s)

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated at the intervals specified by the manufacturer or more frequently, and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications. In the event that an internally calibrated field instrument fails to meet calibration/checkout procedures, it will be returned to the manufacturer for service. Equipment found to be out of tolerance during the period of use will be removed from the field, and measuring and testing activities performed using the equipment will be addressed via the corrective action system described in Section 18.4 of this QAPP.

14.2 Laboratory Instrument and Equipment

When analyses are conducted according to USEPA SW-846 methods, the calibration procedures and frequencies specified in the applicable method will be followed, as noted in the attached SOPs (Attachment B). For analyses governed by SOPs, see the appropriate SOP for the required calibration procedures and frequencies. Records of calibrations will be filed and maintained by the laboratory. These records will be subject to QA audit. For all instruments, the laboratory will maintain trained repair staff with in-house spare parts or will maintain service contracts with vendors.

All standards used in the calibration of equipment are traceable, directly or indirectly, to National Institute of Standards and Technology. All standards received shall be logged into standard receipt logs maintained by the individual analytical groups. Each group will maintain a standards log that tracks the preparation of standards used for calibration and QC purposes.

15. Inspection/Acceptance Requirements for Supplies and Consumables

All supplies to be used in the field and laboratory will be available when needed. They will be free of target chemicals and interferences. All reagents will be tested prior to use with Site samples. All standards will be verified against a second source standard. The laboratory will follow a "first in/first out" procedure for the storage and use of all consumables to minimize the risk of contamination and degradation. The various supplies and consumables required on-site are or will be noted in the associated work plan(s).

16. Data Acquisition Requirements for Non-Direct Measurements

Prior to use, historical data will be reviewed according to the procedures identified in subsequent sections of this QAPP to determine the appropriate uses of such data. The extent to which these data can be validated will be determined by the analytical level and QC data available. The evaluation of historical data requires the following:

- identification of analytical levels
- evaluation of QC data, when available
- development of conclusions regarding the acceptability of the data for intended uses

Acceptability of historical data for intended uses will be determined by application of these procedures and by professional judgment.

17. Data Management

The purpose of data management is to facilitate accuracy and ready accessibility of all of the necessary data to meet the analytical and reporting objectives of the project. The field investigations will encompass a large number of samples and analytes from a large geographic area. Due to the large amount of resulting data, a structured, comprehensive, and efficient program for the management of data is necessary.

The data management program established for the project includes field documentation and sample QA/QC procedures, methods for tracking and managing the data, and a system for filing all Site-related information. More specifically, data management procedures will be employed to efficiently process the information collected such that the data are readily accessible and accurate. These procedures are described in detail in the following section.

The data management plan has four elements: 1) sample designation system; 2) field activities; 3) sample tracking and management; and 4) data management system.

17.1 Sample Designation System

A concise and easily understandable sample designation system is an important part of the project sampling activities. It provides a unique sample number that will facilitate both sample tracking and easy re-sampling of select locations to evaluate data gaps, if necessary. The sample designation system to be employed during the sampling activities will be consistent, yet flexible enough to accommodate unforeseen sampling events or conditions. A combination of letters and numbers will be used to yield a unique sample number for each field sampled collected, as outlined in Section 7.2.1.

17.2 Field Activities

Field activities designed to gather the information necessary to make decisions require consistent documentation and accurate record keeping. During sampling activities, standardized procedures will be used for documenting field activities, data security, and QA. These procedures are described in further detail in the following subsections.

17.2.1 Field Documentation

Complete and accurate record keeping is a critical component of the field investigation activities. When interpreting analytical results and identifying data trends, investigators realize that field notes are an important part of the review and data verification/ validation process. Multiple types of information records will be maintained to

document the field investigations, including field logs and COC forms. Each type of record has its own specific reporting requirements. These types of field documentation are described in Sections 10.2.1 and 10.2.3.

17.2.2 Data Security

Measures will be taken during the field investigation to prevent samples and records from being lost, damaged, or altered. When not in use, all field notebooks will be stored at the field office or locked in the field vehicle. Access to these files will be limited to the field personnel who utilize them.

17.3 Sample Management and Tracking

A record of all field documentation will be maintained to provide verification of the validity of the data used in the Site analysis. To effectively execute such documentation, specific sample tracking and data management procedures will be used throughout the sampling program.

Sample tracking will begin with the completion of COC forms, as summarized in Section 10.2.3. The completed COC forms associated with samples collected will be faxed to the QAC. Copies of all completed COC forms will be maintained the designated project files. The laboratory will verify receipt of the samples via a phone call or electronically (via email) on the following day.

When analytical data are received from the laboratory, the QAC will review the incoming analytical data packages against the information on the COCs to confirm that the correct analyses were performed for each sample and that results for all samples submitted for analysis were received. Any discrepancies noted will be promptly addressed by the QAC.

17.4 Data Management System

In addition to the sample tracking system, a data management system will be implemented. The central focus of the data management system will be the development of a personal-computer-based project database. The project database, to be maintained by the Database Administrator, will combine pertinent geographical, field, and analytical data. Information that will be used to populate the database will be derived from three primary sources: surveying of sampling locations, field observations, and analytical results. Each of these sources is discussed in the following sections.

17.4.1 Computer Hardware

The database will be constructed on Pentium®-based personal computer work stations connected through a network server. The network will provide access to various hardware peripherals (e.g., laser printers, backup storage devices, image scanners, modems). Computer hardware will be upgraded to industrial and corporate standards, as necessary, in the future.

17.4.2 Computer Software

The database will be written in Microsoft® Access, or other suitable program. Custom applets, such as diskette importing programs, will be written in Microsoft® VBA, Microsoft® Visual Basic, or other suitable program. Geographic Information System (GIS) applications will be developed in ESRI ArcGIS, or other suitable program. Tables and other database reports will be generated with Microsoft® Excel, Microsoft® Word, Seagate Crystal Reports, or other suitable programs. These software products will be upgraded to current industrial standards, as necessary.

17.4.3 Survey Information

Sample locations may be surveyed to provide accurate documentation of sampling locations for mapping and GIS purposes; to facilitate the re-sampling of select sampling locations during future monitoring programs, if needed; and for any additional activities. The surveying activities that will occur in the field will consist of the collection of information that will be used to compute a northing and easting in state plane coordinates (or a Site-specific coordinate system) for each sampling location and to compute elevations relative to the National Geodetic Vertical Datum of 1988 (or a Site-specific vertical datum) for select sampling locations, as appropriate. All field books associated with the surveying activities will be stored as a record of the project activities.

17.4.4 Field Observations

An important part of the information that will ultimately reside in the data management system for use during the project will originate in the observations that are recorded in the field.

During each sampling event, appropriate field documentation will be prepared by the field personnel who performed the sampling activities. The purpose of the documentation is to create a summary and a record of the sampling event. Items to be included are the locations sampled, the sampling methodologies used, blind duplicate and MS/MSD sample identification numbers, equipment decontamination procedures, personnel involved in the activity, and any noteworthy events that occurred.

17.4.5 Analytical Results

Analytical results will be provided by the laboratory in both digital and a hard copy format. The data packages will be examined to confirm that the correct analyses were performed for each sample submitted and that all of the analyses requested on the COC form were performed. If discrepancies are noted, the QAC will be notified and will promptly follow up with the laboratory to resolve any issues.

Each data package will be verified and validated in accordance with the procedures presented in Section 21.1. Any data that do not meet the specified standards will be flagged pending resolution of the issue. The flag will not be removed from the data until the issue associated with the sample results is resolved. Although flags may remain for certain data, the use of those data may not necessarily be restricted.

Following completion of the data verification and validation, the digital files will be used to populate the appropriate database tables. An example of the format of EDD format is included in Table 5. This format specifies one data record for each constituent for each sample analyzed. Specific fields include:

- sample identification number
- date sampled
- date analyzed
- parameter name
- analytical result
- units
- detection limit
- qualifier(s)

The individual EDDs, supplied by the laboratory in either an ASCII comma-separated value format or in a Microsoft® Excel worksheet, will be loaded into the appropriate database table via a custom-designed user interface Visual Basic program. Any analytical data that cannot be provided by the laboratory in electronic format will be entered manually. After entry into the database, the EDD data will be compared to the field information previously entered into the database to confirm that all requested analytical data have been received.

17.4.6 Data Analysis and Reporting

The database management system will have several functions to facilitate the review and analysis of the data. Routines have been developed to permit the user to scan analytical data from a given Site for a given media. Several output functions are also available that can be modified, as necessary, for use in the data management system.

A valuable function of the data management system will be the generation of tables of analytical results from the project databases. The capability of the data management system to directly produce tables reduces the redundant manual entry of analytical results during report preparation and precludes transcription errors that may occur otherwise. This data management system function creates a digital file of analytical results and qualifiers for a given media. The file can then processed into a table of rows and columns that can be transferred to word processing software (e.g., Microsoft® Word) for final formatting and addition of titles and notes. Tables of analytical data will be produced as part of data interpretation tasks and the reporting of data to the USEPA.

The data management system also has the capability of producing a digital file of select parameters that exist in one or more of the databases. This type of custom function is accomplished on an interactive basis and is best used for transferring select information into a number of analysis tools, such as statistical or graphing programs.

18. Assessment and Response Actions

18.1 General

Performance and systems audits will be completed in the field and laboratory during the sampling activities, as described below.

18.2 Field Audits

The appropriate Task Manager will monitor field performance. Field performance audit summaries will contain an evaluation of field activities to verify that the activities are performed according to established protocols. The QAC will review field reports and communicate concerns to the Project Manager and/or Task Managers, as appropriate. In addition, the QAC will review the rinse and trip blank data to identify potential deficiencies in field sampling and cleaning procedures. In addition, systems audits comparing scheduled QA/QC activities from this QAPP with actual QA/QC activities completed will be performed. The appropriate Task Manager and QAC will periodically confirm that work is being performed consistent with this QAPP and the Work Plan.

18.3 Laboratory Audits

Internal laboratory audits are conducted by the Laboratory QAC. As part of the audit, the overall performance of the laboratory staff is evaluated and compared to the performance criteria outlined in the laboratory QA manual and SOPs. The results of the audits are summarized and issued to each department supervisor, the Laboratory Manager, and the Laboratory Director. A systems audit of each laboratory is also performed by the QAC to determine whether the procedures implemented by each laboratory are in compliance with the QA manual and SOPs.

As a participant in state and federal certification programs, the laboratory is audited by representatives of the regulatory agency issuing certification in addition to the laboratory's internal audits. Audits are usually conducted on an annual basis and focus on laboratory conformance to the specific program protocols for which the laboratory is seeking certification. The auditor reviews sample handling and tracking documentation, analytical methodologies, analytical supportive documentation, and final reports. The audit findings are formally documented and submitted to the laboratory for corrective action, if necessary.

Beazer and/or Beazer's consultants reserve the right to conduct an onsite audit of the laboratory prior to the start of analyses for the project. Additional audits may be performed during the course of the project, as deemed necessary.

18.4 Corrective Action

Corrective actions are required when field or analytical data are not within the objectives specified in this QAPP or the applicable work plans. Corrective actions include procedures to promptly investigate, document, evaluate, and correct data collection and/or analytical procedures. Field and laboratory corrective action procedures for the actions are described below.

18.4.1 Field Procedures

If, during field work, a condition is noted by the field crew that would have an adverse effect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause, and corrective action implemented by the Field Manager or a designee will be documented on a Corrective Action Form and reported to the appropriate Task Manager, QAC, and Project Manager.

Examples of situations that would require corrective actions are provided below:

- protocols as defined by the QAPP and Work Plan have not been followed
- equipment is not in proper working order or is not properly calibrated
- QC requirements have not been met
- issues resulting from performance or systems audits have not been resolved

Project personnel will continuously monitor ongoing work performance in the normal course of daily responsibilities.

18.4.2 Laboratory Procedures

In the laboratory, when a condition is noted to have an adverse effect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause, and corrective action taken will be documented and reported to the appropriate Project Manager and QAC.

Corrective action may be initiated, at a minimum, under the following conditions:

- protocols as defined by this QAPP have not been followed
- predetermined data acceptance standards are not obtained

- equipment is not in proper working order or calibrated
- sample and test results are not completely traceable
- QC requirements have not been met
- issues resulting from performance or systems audits have not been resolved

Laboratory personnel will continuously monitor ongoing work performance in the normal course of daily responsibilities. Corrective action is initiated at the point where the problem has been identified. At whatever level this occurs (analyst, supervisor, data review, or quality control), it is brought to the attention of the Laboratory QAC and, ultimately, the Laboratory Director. Final approval of any action deemed necessary is subject to the approval of the Laboratory Director.

Any corrective action deemed necessary based on system or performance audits, the analytical results of split samples, or the results of data review will be implemented. The corrective action may include sample re-extraction, re-preparation, re-analysis, cleanup, dilution, matrix modification, or other activities.

19. Reports to Management

The QAC will audit the implementation of the QAPP. Each project component will result in some type of QA report or, by its absence, will indicate that no significant QA or QC deviations occurred. Items that may result in a QA report include:

- changes or updates to the QAPP
- deviations from QAPP or Work Plan specification
- results of system and performance audits
- significant QA/QC problems, recommended solutions, and the results of corrective actions
- limitations on the use of measurement data

19.1 Field Reports

Reporting of the quality of field sample collection and field measurements will be the responsibility of the Field Supervisor or designee. Information from the field logbooks will be compiled, and a summary report on field activity QA will be prepared for the project file.

19.2 Laboratory Reports

The laboratory will maintain QA records related to analyses, QC, and corrective action. This information will be made available to the Project Manager upon request. Routine reporting will include documenting all internal QC checks performed for this project.

20. Data Reduction and Review

20.1 General

After field and laboratory data are obtained, the data will be subject to the following:

1. reduction, or manipulation mathematically or otherwise into meaningful and useful forms

2. review

3. organization, interpretation, and reporting

4. data verification and/or validation

Each of these topics is further discussed in the following subsections.

20.2 Field Data Reduction and Review

20.2.1 Field Data Reduction

Information collected in the field through visual observation, manual measurement, and/or field instrumentation will be recorded in field notebooks or data sheets, and/or on forms. Such data will be reviewed by the appropriate Task Manager for adherence to the Work Plan and this QAPP and for consistency. Concerns identified as a result of this review will be discussed with the field personnel; corrected if possible; and, as necessary, incorporated into the data evaluation process.

20.2.2 Field Data Review

Field data calculations, transfers, and interpretations will be conducted by the field personnel and reviewed weekly (or more frequently, if warranted by the nature of the specific investigation activity) for accuracy by the appropriate Task Manager and the QAC. Logs and documents will be checked for:

1. general completeness

2. readability

3. usage of appropriate procedures

4. appropriate instrument calibration and maintenance

5. reasonableness in comparison to present and past data collected

6. correct sampling locations

7. correct calculations and interpretations

20.3 Laboratory Data Reduction and Review

20.3.1 Laboratory Data Reduction

The calculations used for data reduction will be specified in each of the analytical methods referenced previously. Whenever possible, analytical data will be transferred directly from the instrument to a computerized data system. Raw data will be entered into permanently bound laboratory notebooks. The data entered must be sufficient to document all factors used to arrive at the reported value. The specific processes for data collection by computer and by hand are included in the laboratories' method-specific SOPs and Quality Assurance Manuals (QAMs) provided in Attachment C.

Concentration calculations for chromatographic analyses will be based on response factors. Quantitation will be performed using either internal or external standards.

Inorganic analyses will be based on regression analysis. Regression analysis is used to fit a curve through the calibration standard data. The sample concentrations will be calculated using the resulting regression equations.

Nonaqueous values will be reported on a dry-weight basis. Unless otherwise specified, all values will be reported uncorrected for blank contamination.

20.3.2 Laboratory Data Review

Data will be subject to multi-level review by the laboratory. The group leader will review all data reports prior to release for final data report generation. The QAC will review the final data reports, and the Laboratory Director will review a cross section of the final data reports prior to shipment to the appropriate Beazer consultant.

If discrepancies or deficiencies are present in the analytical results, corrective action will be taken, as discussed in Section 18. Deficiencies discovered as a result of internal data review, as well as the corrective actions to be used to rectify the situation, will be documented on a Corrective Action Form. This form will be submitted to the Project Manager.

20.4 Data Verification and Validation

All data generated for health and safety and engineering design/control purposes will be subjected to the data verification and validation procedures outlined in Section 21. Data generated for disposal purposes will not be reviewed.

21. Data Verification and Validation

Data verification is the process of evaluating the completeness, correctness, and conformance/compliance of a specific data set against the method, procedural, or contractual requirements. Data validation is an analyte- and sample-specific process that extends the evaluation of data beyond method, procedural, or contractual compliance (i.e., data verification) to determine the analytical quality of a specific data set.

Data validation entails a review of the quality control data and the raw data to verify that the laboratory was operating within required limits, the analytical results were correctly transcribed from the instrument read outs, and which, if any, environmental samples were related to any out-of-control quality control samples. The objective of data validation is to identify any questionable or invalid laboratory measurements.

Data validation will be conducted using the most recent versions of the USEPAs Function Guidelines (USEPA, 1999; 2002) and USEPA Region V SOPs (USEPA Region V, 1993; 1997) for data validation available at the time of project initiation, where appropriate. These procedures and criteria may be modified as necessary to address project-specific and method-specific criteria, control limits, and procedures. Data validation will consist of data screening, checking, reviewing, editing, and interpretation to document analytical data quality and to determine whether the quality is sufficient to meet the DQOs.

The data validator will verify that reduction of laboratory measurements and laboratory reporting of analytical parameters is in accordance with the procedures specified for each analytical method and/or as specified in this QAPP. Any deviations from the analytical method or any special reporting requirements apart from that specified in this QAPP will be detailed on COC forms.

Upon receipt of laboratory data, the following procedures will be executed by the data validator:

- Evaluate completeness of data package.
- Verify that field COC forms were completed and that samples were handled properly.

- Verify that holding times were met for each parameter. Holding time exceedences, should they occur, will be documented. Data for all samples exceeding holding time requirements will be flagged as either estimated or rejected. The decision as to which qualifier is more appropriate will be made on a case-by-case basis.

- Verify that parameters were analyzed according to the methods specified.

- Review QA/QC data (i.e., make sure duplicates, blanks, and spikes were analyzed on the required number of samples, as specified in the method; verify that duplicate and MS recoveries are acceptable).

- Investigate anomalies identified during review. When anomalies are identified, they will be discussed with the Project Manager and/or Laboratory Manager, as appropriate.

- If data appears suspect, investigate the specific data of concern. Calculations will be traced back to raw data; if calculations do not agree, the cause will be determined and corrected.

Deficiencies discovered as a result of the data verification/validation, as well as the corrective actions implemented in response, will be documented and submitted in the form of a written report addressing the following topics as applicable to each method:

- assessment of the data package

- description of any protocol deviations

- failures to reconcile reported and/or raw data

- assessment of any compromised data

- overall appraisal of the analytical data

- table of site name, sample quantities, matrix, and fractions analyzed

It should be noted that qualified results do not necessarily invalidate data. The goal to produce the best possible data does not necessarily mean producing data without quality control qualifiers. Qualified data can provide useful information.

Resolution of any issues regarding laboratory performance or deliverables will be handled between the laboratory and the data validator. Suggestions for reanalysis may be made by the ARCADIS QAC at this point. Upon review of data, any formal requests to the laboratory for re-analyses or unresolved laboratory issues affecting the quality of the data will be documented in a separate section of the data validation report.

Data validation reports will be kept in the project files at the designated consultant's office.

22. Reconciliation with User Requirements

The data results will be examined to determine the performance that was achieved for each data usability criteria. The performance will then be compared with the project objectives and DQOs. Deviations from objectives will be noted. Additional action may be warranted when performance does not meet performance objectives for critical data. Options for corrective action relating to incomplete information, questionable results or inconsistent data, may include any or all of the following:

- retrieval of missing information
- request for additional explanation or clarification
- reanalysis of sample from extract (when appropriate)
- recalculation or reinterpretation of results by the laboratory

These actions may improve the data quality, reduce uncertainty, and/or eliminate the need to qualify or reject data. If these actions do not improve the data quality to an acceptable level, the following additional actions may be taken:

- extrapolation of missing data from existing data points
- use of historical data
- evaluation of the critical/non-critical nature of the sample

If the data gap cannot be resolved by these actions, an evaluation of the data bias and potential for false negatives and positives can be performed. If the resultant uncertainty level is unacceptable, additional sample collection and analysis will be performed.

23. References

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USEPA. 1999. *Contract Laboratory Program National Functional Guidelines for Organic Data Review*. EPA-540/R-99-008 (October).

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USEPA. 2001. *EPA Requirements for Quality Assurance Project Plans for Environmental Operations*. EPA-QA/R-5. Office of Environmental Information. (March).

USEPA. 2002a. *Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*. EPA-540/R-01-008 (July).

USEPA. 2002b. *Guidance for Quality Assurance Project Plans*. EPA-QA/G-5. Office of Environmental Information. (December).

USEPA. 2006. *Guidance on Systematic Planning Using the Data Quality Objective Process*. EPA QA/G-4 (February).

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Tables

TABLE 1
SAMPLE QUANTITIES AND QUALITY CONTROL FREQUENCIES

BEAZER EAST, INC.
FORMER KOPPERS WOOD-TREATING SITE
CARBONDALE, ILLINOIS
QUALITY ASSURANCE PROJECT PLAN

Parameter	Laboratory	Estimated Environmental Sample Quality	Field QC Analyses						Laboratory QC Sample					
			Trip Blank		Rinse Blank		Field Duplicate		Matrix Spike		Matrix Spike Duplicate		Lab Duplicate	
			Freq.[2]	No.	Freq.[2]	No.	Freq.[2]	No.	Freq.[2]	No.	Freq.[2]	No.	Freq.[2]	No.
Soil/Sediment														
Semivolatile Organic Compounds (SW-846 8270)[1]	1	TBD	NA	NA	1/day	TBD	1/10	TBD	1/20	TBD	1/20	TBD	NA	NA
Dioxins/Furans (SW-846 8290)	2	TBD	NA	NA	1/day	TBD	1/10	TBD	1/20	TBD	1/20	TBD	NA	NA
Metals (SW-846 6010/7000)	1	TBD	NA	NA	1/day	TBD	1/10	TBD	1/20	TBD	NA	NA	1/20	TBD
Total Organic Carbon (TOC; Walkley-Black)	1	TBD	NA	NA	1/day	TBD	1/10	TBD	1/20	TBD	NA	NA	1/20	TBD
PAHs in Sediment Porewater (SW-846 8270)[1]	1	TBD	NA	NA	1/day	TBD	1/10	TBD	1/20	TBD	1/20	TBD	NA	NA
Groundwater														
Volatile Organic Compounds (SW-846 8021B)	1	TBD	1/cooler	TBD	1/day	TBD	1/10	TBD	1/20	TBD	1/20	TBD	NA	NA
Semivolatile Organic Compounds (SW-846 8270)[1]	1	TBD	NA	NA	1/day	TBD	1/10	TBD	1/20	TBD	1/20	TBD	NA	NA
Dioxins/Furans (SW-846 8290)	2	TBD	NA	NA	1/day	TBD	1/10	TBD	1/20	TBD	1/20	TBD	NA	NA
Metals (SW-846 6010/7000)	1	TBD	NA	NA	1/day	TBD	1/10	TBD	1/20	TBD	NA	NA	1/20	TBD
Total Phenolics (SW-846 9066)	1	TBD	NA	NA	1/day	TBD	1/10	TBD	1/20	TBD	NA	NA	1/20	TBD
Surface Water														
Semivolatile Organic Compounds (SW-846 8270)[1]	1	TBD	NA	NA	1/day	TBD	1/10	TBD	1/20	TBD	1/20	TBD	NA	NA
Dioxins/Furans (SW-846 8290)	2	TBD	NA	NA	1/day	TBD	1/10	TBD	1/20	TBD	1/20	TBD	NA	NA
Metals (SW-846 6010/7000)	1	TBD	NA	NA	1/day	TBD	1/10	TBD	1/20	TBD	NA	NA	1/20	TBD
Suspended Solids (TSS; EPA 160.2)	1	TBD	NA	NA	1/day	TBD	1/10	TBD	NA	--	NA	NA	1/20	TBD
Biota														
Semivolatile Organic Compounds (SW-846 8270)	3	TBD	NA	NA	1/day	TBD	1/20	TBD	1/20	TBD	1/20	TBD	NA	--
Dioxins/Furans SW-846(8290)	2	TBD	NA	NA	1/day	TBD	1/20	TBD	1/20	TBD	1/20	TBD	NA	--
Metals (SW-846 6010/6020)	3	TBD	NA	NA	1/day	TBD	1/20	TBD	1/20	TBD	NA	NA	1/20	10
Percent Lipids (SW-846 3541)	3	TBD	NA	NA	1/day	TBD	1/20	TBD	1/20	TBD	NA	NA	1/20	10

Notes:
TBD = To be determined
1/day = One rinse blank per day or one per 20 samples, whichever is more frequent. Rinse blanks not required when dedicated sampling equipment is used.
Freq = Frequency
NA = Not Applicable
No. = Number
QC = Quality Control
SDG = Sample delivery group
1. SVOCs may be analyzed by standard Method 8270C, 8270 SIM, or 8270 low level (LL).
2. The minimum frequency for QC samples will be one per sample delivery group (SDG), with a SDG consisting of no more than 20 samples.
3. Laboratories are as follows:
   1 - Test America
   2 - Vista
   3 - CAS
4. Analytical methods specified above may be subject to change if an updated USEPA-approved method is available and more applicable.

TABLE 2A
PARAMETERS, METHODS, AND TARGET REPORTING LIMITS
(Groundwater and Surface Water)

BEAZER EAST, INC.
FORMER KOPPERS WOOD-TREATING SITE
CARBONDALE, ILLINOIS
QUALITY ASSURANCE PROJECT PLAN

Analyte	Groundwater (ug/L)			Surface Water (ug/L)		
	Action Limit [1,2]	Laboratory MDL	Laboratory RL	Action Limit [1,3]	Laboratory MDL	Laboratory RL
Volatile Organic Compounds (8021)						
Benzene	5	0.013	1.0	310	0.013	1.0
Ethylbenzene	700	0.13	1.0	14	0.13	1.0
Toluene	1000	0.2	1.0	600	0.2	1.0
Xylenes (total)	10000	0.3	1.0	360	0.3	1.0
Total Recoverable Phenolics (9066)						
Total Recoverable Phenolics	NS	5.7	10	NS	5.7	10
Semivolatile Organic Compounds (8270)[5]						
Naphthalene	140	0.0382	0.2	68	0.0382	0.2
Acenaphthylene	NS	0.035	0.2	15	0.035	0.2
Acenaphthene	420	0.0382	0.2	62	0.0382	0.2
Fluorene	280	0.0363	0.2	4500	0.0363	0.2
Pentachlorophenol	1	0.0806	1.0	2.5	0.0806	1.0
Phenanthrene	NS	0.04099	0.2	3.7	0.04099	0.2
Anthracene	2100	0.0355	0.2	35000	0.0355	0.2
Fluoranthene	280	0.02687	0.2	120	0.02687	0.2
Pyrene	210	0.0238	0.2	3500	0.0238	0.2
Benzo(a)anthracene	0.13	0.0217	0.2	0.16	0.0217	0.2
Chrysene	1.5	0.03368	0.2	16	0.03368	0.2
Benzo(b)fluoranthene	0.18	0.04168	0.2	0.16	0.04168	0.2
Benzo(k)fluoranthene	0.17	0.06011	0.2	1.6	0.06011	0.2
Benzo(a)pyrene	0.2	0.07585	0.2	0.016	0.07585	0.2
Indeno(1,2,3-cd)pyrene	0.43	0.1043	0.2	0.16	0.1043	0.2
Dibenz(a,h)anthracene	0.3	0.1451	0.2	0.016	0.1451	0.2
Benzo(g,h,i)perylene	NS	0.1222	0.2	NS	0.1222	0.2
Total PAHs	NS	--	--	NS	--	--
Dioxins/Furans (8290)						
1,2,3,4,6,7,8-HpCDD	NS	0.00082634	0.025	NS	0.00082634	0.025
HpCDDs (total)	NS	--	--	NS	--	--
1,2,3,4,7,8,9-HpCDF	NS	0.0015246	0.025	NS	0.0015246	0.025
1,2,3,4,6,7,8-HpCDF	NS	0.00120879	0.025	NS	0.00120879	0.025
HpCDFs (total)	NS	--	--	NS	--	--
1,2,3,4,7,8-HxCDD	NS	0.00072669	0.025	NS	0.00072669	0.025
1,2,3,6,7,8-HxCDD	NS	0.00072895	0.025	NS	0.00072895	0.025
1,2,3,7,8,9-HxCDD	NS	0.00222235	0.025	NS	0.00222235	0.025
HxCDDs (total)	NS	--	--	NS	--	--
1,2,3,4,7,8-HxCDF	NS	0.00093226	0.025	NS	0.00093226	0.025
1,2,3,6,7,8-HxCDF	NS	0.0009371	0.025	NS	0.0009371	0.025
1,2,3,7,8,9-HxCDF	NS	0.00164887	0.025	NS	0.00164887	0.025
2,3,4,6,7,8-HxCDF	NS	0.00093156	0.025	NS	0.00093156	0.025
HxCDFs (total)	NS	--	--	NS	--	--
1,2,3,7,8-PeCDD	NS	0.0012165	0.025	NS	0.0012165	0.025
PeCDDs (total)	NS	--	--	NS	--	--
1,2,3,7,8-PeCDF	NS	0.00126496	0.025	NS	0.00126496	0.025
2,3,4,7,8-PeCDF	NS	0.00070733	0.025	NS	0.00070733	0.025
PeCDFs (total)	NS	--	--	NS	--	--
2,3,7,8-TCDD	NS	0.00069944	0.005	4.6E-09	0.00069944	0.005
TCDDs (total)	NS	--	--	NS	--	--
2,3,7,8-TCDF	NS	0.0004857	0.005	NS	0.0004857	0.005
TCDFs (total)	NS	--	--	NS	--	--
OCDD	NS	0.00242585	0.050	NS	0.00242585	0.050
OCDF	NS	0.0017753	0.050	NS	0.0017753	0.050

TABLE 2A
PARAMETERS, METHODS, AND TARGET REPORTING LIMITS
(Groundwater and Surface Water)

BEAZER EAST, INC.
FORMER KOPPERS WOOD-TREATING SITE
CARBONDALE, ILLINOIS
QUALITY ASSURANCE PROJECT PLAN

Analyte	Groundwater (ug/L)			Surface Water (ug/L)		
	Action Limit [1,2]	Laboratory MDL	Laboratory RL	Action Limit [1,3]	Laboratory MDL	Laboratory RL
Inorganics (6010)						
Arsenic - Soluble	50	3.293	10	190	3.293	10
Arsenic - Total	NS	3.293	10	NS	3.293	10
Chromium - Soluble	100	0.9316	5	See note 4	0.9316	5
Chromium - Total	NS	0.9316	5	NS	0.9316	5
Copper - Soluble	650	1.153	25	See note 4	1.153	25
Copper -Total	NS	1.153	25	NS	1.153	25

Notes:
MDL = maximum detection limit
RL = reporting limit
NS = no standard
1. Action limits listed above are for comparison to MDLs and RLs only, and do not indicate their applicability to any specific sample location/event. Other action limits may be specified in work plans.
2. Lowest value of TACO Tier 1 Groundwater Remediation Objectives for Class I or II Groundwater.
3. Lowest value of Illinois Water Quality Criteria for human health or aquatic life.
4. Hardness data required to calculate aquatic life criteria for chromium and copper.
5. SVOCs may be analyzed by standard Method 8270C, 8270 SIM, or 8270 LL (MDLs and RLs specified above are for 8270 SIM)
6. MDLs and RLs are subject to variability.

TABLE 2B
PARAMETERS, METHODS, AND TARGET REPORTING LIMITS
(Soil and Sediment)

BEAZER EAST, INC.
FORMER KOPPERS WOOD-TREATING SITE
CARBONDALE, ILLINOIS
QUALITY ASSURANCE PROJECT PLAN

Analyte	Soil [1] (ug/kg)			Sediment [1] (ug/kg)		
	Action Limit [2,3]	Laboratory MDL	Laboratory RL	Action Limit [2,3]	Laboratory MDL	Laboratory RL
Semivolatile Organic Compounds (8270)[4]						
Naphthalene	99	2.4	6.67	176	2.4	6.67
Acenaphthylene	24000	2.1	6.67	5.87	2.1	6.67
Acenaphthene	682000	2.1	6.67	6.71	2.1	6.67
Fluorene	122000	2.2	6.67	77.4	2.2	6.67
Pentachlorophenol	1.8	0.7	33.3	23000	0.7	33.3
Phenanthrene	45700	1.8	6.67	204	1.8	6.67
Anthracene	1480000	1.7	6.67	57.2	1.7	6.67
Fluoranthene	122000	2.2	6.67	423	2.2	6.67
Pyrene	78500	1.3	6.67	195	1.3	6.67
Benzo(a)anthracene	900	1.6	6.67	108	1.6	6.67
Chrysene	473	1.4	6.67	166	1.4	6.67
Benzo(b)fluoranthene	900	1.2	6.67	10400	1.2	6.67
Benzo(k)fluoranthene	9000	2.4	6.67	400	2.4	6.67
Benzo(a)pyrene	90	2.1	6.67	150	2.1	6.67
Indeno(1,2,3-cd)pyrene	900	1.3	6.67	200	1.3	6.67
Dibenz(a,h)anthracene	90	1.4	6.67	33	1.4	6.67
Benzo(g,h,i)perylene	19000	1.2	6.67	170	1.2	6.67
Dioxins/Furans (8290)						
1,2,3,4,6,7,8-HpCDD	NS	0.000058	0.0025	NS	0.000058	0.0025
HpCDDs (total)	NS	--	--	NS	--	--
1,2,3,4,7,8,9-HpCDF	NS	0.000038	0.0025	NS	0.000038	0.0025
1,2,3,4,6,7,8-HpCDF	NS	0.000043	0.0025	NS	0.000043	0.0025
HpCDFs (total)	NS	--	--	NS	--	--
1,2,3,4,7,8-HxCDD	NS	0.000031	0.0025	NS	0.000031	0.0025
1,2,3,6,7,8-HxCDD	NS	0.000027	0.0025	NS	0.000027	0.0025
1,2,3,7,8,9-HxCDD	NS	0.00004	0.0025	NS	0.00004	0.0025
HxCDDs (total)	NS	--	--	NS	--	--
1,2,3,4,7,8-HxCDF	NS	0.000045	0.0025	NS	0.000045	0.0025
1,2,3,6,7,8-HxCDF	NS	0.000021	0.0025	NS	0.000021	0.0025
1,2,3,7,8,9-HxCDF	NS	0.000033	0.0025	NS	0.000033	0.0025
2,3,4,6,7,8-HxCDF	NS	0.000021	0.0025	NS	0.000021	0.0025
HxCDFs (total)	NS	--	--	NS	--	--
1,2,3,7,8-PeCDD	NS	0.000015	0.0025	NS	0.000015	0.0025
PeCDDs (total)	NS	--	--	NS	--	--
1,2,3,7,8-PeCDF	NS	0.000016	0.0025	NS	0.000016	0.0025
2,3,4,7,8-PeCDF	NS	0.000014	0.0025	NS	0.000014	0.0025
PeCDFs (total)	NS	--	--	NS	--	--
2,3,7,8-TCDD	0.000199	0.000023	0.0005	0.00012	0.000023	0.0005
TCDDs (total)	NS	--	--	NS	--	--
2,3,7,8-TCDF	NS	0.000012	0.0005	NS	0.000012	0.0005
TCDFs (total)	NS	--	--	NS	--	--
OCDD	NS	0.00009	0.005	NS	0.00009	0.005
OCDF	NS	0.000042	0.005	NS	0.000042	0.005

TABLE 2B
PARAMETERS, METHODS, AND TARGET REPORTING LIMITS
(Soil and Sediment)

BEAZER EAST, INC.
FORMER KOPPERS WOOD-TREATING SITE
CARBONDALE, ILLINOIS
QUALITY ASSURANCE PROJECT PLAN

Analyte	Soil [1] (ug/kg)			Sediment [1] (ug/kg)		
	Action Limit [2,3]	Laboratory MDL	Laboratory RL	Action Limit [2,3]	Laboratory MDL	Laboratory RL
Inorganics (6010)						
Arsenic	5700	329	1000	9790	329	1000
Chromium	400	93.1	500	43400	93.1	500
Copper	5400	115.3	2500	31600	115.3	2500

Notes:
MDL = maximum detection limit
RL = reporting limit
NS = no standard
1. The target reporting limits are based on wet weight. The actual reporting limits will vary based on sample weight and moisture content.
2. Action limits listed above are for comparison to MDLs and RLs only, and do not indicate their applicability to any specific sample location/event. Other action limits may be specified in work plans.
3. Lowest value of TACO Tier 1 Soil Remediation Objectives (for residential and commercial/industrial properties), USEPA Ecological Screening Levels, USEPA Region 5 Soil ESLs.
4. SVOCs may be analyzed by standard Method 8270C, 8270 SIM, or 8270 LL (MDLs and RLs specified above are f 8270 SIM).
5. MDLs and RLs are subject to variability.

TABLE 2C
PARAMETERS, METHODS, AND TARGET REPORTING LIMITS
(Biota)

BEAZER EAST, INC.
FORMER KOPPERS WOOD-TREATING SITE
CARBONDALE, ILLINOIS
QUALITY ASSURANCE PROJECT PLAN

Analyte	Biota [1] (ug/kg)		
	Action Limit	Laboratory MDL	Laboratory RL
Semivolatile Organic Compounds (8270)[2]			
Naphthalene	NS	0.4	5.0
Acenaphthylene	NS	0.069	5.0
Acenaphthene	NS	0.11	5.0
Fluorene	NS	0.15	5.0
Pentachlorophenol	NS	30	100
Phenanthrene	NS	0.36	5.0
Anthracene	NS	0.065	5.0
Fluoranthene	NS	0.09	5.0
Pyrene	NS	0.098	5.0
Benzo(a)anthracene	NS	0.066	5.0
Chrysene	NS	0.076	5.0
Benzo(b)fluoranthene	NS	0.07	5.0
Benzo(k)fluoranthene	NS	0.056	5.0
Benzo(a)pyrene	NS	0.081	5.0
Indeno(1,2,3-cd)pyrene	NS	0.064	5.0
Dibenz(a,h)anthracene	NS	0.059	5.0
Benzo(g,h,i)perylene	NS	0.073	5.0
Dioxins/Furans (8290)			
1,2,3,4,6,7,8-HpCDD	NS	0.000058	0.0025
HpCDDs (total)	NS	--	--
1,2,3,4,7,8,9-HpCDF	NS	0.000038	0.0025
1,2,3,4,6,7,8-HpCDF	NS	0.000043	0.0025
HpCDFs (total)	NS	--	--
1,2,3,4,7,8-HxCDD	NS	0.000031	0.0025
1,2,3,6,7,8-HxCDD	NS	0.000027	0.0025
1,2,3,7,8,9-HxCDD	NS	0.00004	0.0025
HxCDDs (total)	NS	--	--
1,2,3,4,7,8-HxCDF	NS	0.000045	0.0025
1,2,3,6,7,8-HxCDF	NS	0.000021	0.0025
1,2,3,7,8,9-HxCDF	NS	0.000033	0.0025
2,3,4,6,7,8-HxCDF	NS	0.000021	0.0025
HxCDFs (total)	NS	--	--
1,2,3,7,8-PeCDD	NS	0.000015	0.0025
PeCDDs (total)	NS	--	--
1,2,3,7,8-PeCDF	NS	0.000016	0.0025
2,3,4,7,8-PeCDF	NS	0.000014	0.0025
PeCDFs (total)	NS	--	--
2,3,7,8-TCDD	NS	0.000023	0.0005
TCDDs (total)	NS	--	--
2,3,7,8-TCDF	NS	0.000012	0.0005
TCDFs (total)	NS	--	--
OCDD	NS	0.00009	0.005
OCDF	NS	0.000042	0.005

TABLE 2C
PARAMETERS, METHODS, AND TARGET REPORTING LIMITS
(Biota)

BEAZER EAST, INC.
FORMER KOPPERS WOOD-TREATING SITE
CARBONDALE, ILLINOIS
QUALITY ASSURANCE PROJECT PLAN

Analyte	Biota [1] (ug/kg)		
	Action Limit	Laboratory MDL	Laboratory RL
Inorganics (6010/6020)			
Arsenic	NS	329	1000
Chromium	NS	93.1	500
Copper	NS	115.3	2500
Percent Lipids (3541)			
Percent Lipids	NS	--	--

Notes:
MDL = maximum detection limit
RL = reporting limit
NS = no standard (other action limits may be specified in work plans)
1. The target reporting limits are based on wet weight. The actual reporting limits will vary based on sample weight and moisture content.
2. SVOCs may be analyzed by standard Method 8270C, 8270 SIM, or 8270 LL (MDLs and RLs specified above are for 8270 SIM).

TABLE 3
ANALYTICAL QUALITY CONTROL LIMITS [1]

BEAZER EAST, INC.
FORMER KOPPERS WOOD-TREATING SITE
CARBONDALE, ILLINOIS
QUALITY ASSURANCE PROJECT PLAN

Parameter	Accuracy - % Recovery			Precision - RPD		
	Surrogate	MS/MSD	LCS	MS/MSD	Lab Duplicate	Field Duplicate
Soil/Sediment						
Semivolatile Organic Compounds	20-140	20-140	40-120	40	--	100
Dioxins/Furans	30-150	30-140	50-140	40	--	100
Metals	--	80-120	80-120	--	20	100
Total Organic Carbon (TOC)	--	--	--	--	--	--
Water						
Volatile Organic Compounds	75-115	60-145	70-140	20	--	30
Semivolatile Organic Compounds	20-140	20-130	40-120	40	--	50
Dioxins/Furans	30-150	40-130	50-140	20	--	50
Metals	--	75-125	75-125	--	20	50
Total Phenolics	--	75-125	75-125	--	20	50
Suspended Solids (TSS)	--	--	75-125	--	20	50
Biota						
Semivolatile Organic Compounds	20-140	20-140	40-120	40	--	100
Dioxins/Furans	30-150	30-140	50-140	40	--	100
Metals (6020)	--	77-127	48-151	--	20	100
Metals (6010)	--	70-130	85-115	--	30	100
Percent Lipids	--	--	--	--	--	--

Notes:
1. The listed QC limits are based on SW-846 guidance and are advisory. The actual limits are determined based on laboratory performance. Frequent failure to meet the QC limits may warrant investigation of the laboratory.

TABLE 4
SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

BEAZER EAST, INC.
FORMER KOPPERS WOOD-TREATING SITE
CARBONDALE, ILLINOIS
QUALITY ASSURANCE PROJECT PLAN

Parameter	Method[1]	Bottle Type	Preservation	Holding Time[2]
Soil/Sediment				
Semivolatile Organic Compounds	8270 [3]	1-4oz wide mouth glass jar	Cool to 4°C	14 days to extraction 40 days to analysis
Dioxins/Furans	8290	1-4oz wide mouth glass jar	Cool to 4°C	30 days to extraction 45 days to analysis
Metals	6010	1-4oz wide mouth glass jar	Cool to 4°C	180 days to analysis
Total Organic Carbon (TOC)	Walkley-Black/ASTM 2974	1-4oz wide mouth glass jar	Cool to 4°C	28 days to analysis
Total Solids	SM2540B	1-125ml plastic jar	Cool to 4°C	7 days to analysis
Water				
Semivolatile Organic Compounds	8270 [3]	2-1 liter amber glass bottle with Teflon®-lined lid	Cool to 4°C	7 days to extraction 40 days to analysis
Volatile Organic Compounds	8021B	2-40ml glass vials with Teflon®-lined lid	Cool to 4°C; HCL to pH<2	14 days to analysis
Dioxins/Furans	8290	2-1 liter amber glass bottle with Teflon®-lined lid	Cool to 4°C	30 days to extraction 45 days to analysis
Metals	6010	1liter plastic bottle	HNO_3 to pH<2	180 days to analysis
Total Phenolics	9066	500 ml glass container	Cool to 4°C, H_2SO_4 pH<2	28 days to analysis
Suspended Solids (TSS)	160.2	500ml plastic or glass bottle	Cool to 4°C	7 days to analysis
Biota				
Semivolatile Organic Compounds	8270 [3]	Wrap with aluminum foil and freezer paper	Cool to 4°C, store at laboratory at -20°C	14 days to extraction 40 days to analysis
Dioxins/Furans	8290	Wrap with aluminum foil and freezer paper	Cool to 4°C, store at laboratory at -20°C	30 days to extraction 45 days to analysis
Metals	6010/6020	Wrap with aluminum foil and freezer paper	Cool to 4°C, store at laboratory at -20°C	180 days to analysis
Percent Lipids	3541	Wrap with aluminum foil and freezer paper	Cool to 4°C, store at laboratory at -20°C	7 days to analysis

Notes:
1 . USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste. SW-846 3rd ed. Washington, D.C. 1996.*
USEPA. Methods for Chemical Analysis of Water and Waste. EMSL-Cincinnati. 1983:
APHA. *Standard Methods for the Examination of Water and Wastewater.* Washington, DC. 1998.
ASTM International. 2003. *Annual Book of ASTM Standards 2003 Section 4 Construction*, Volume 04.08. West Conshohocken, PA. ASTM International
Department of the Army. 1986. *Engineering Manual Laboratory Soils Testing*. Washington, D.C. Department of the Army, Office of the Chief of Engineers
2. All holding times are measured from date of collection.
3. SVOCs may be analyzed by standard Method 8270C, 8270 SIM, or 8270 LL.

TABLE 5
ELECTRONIC DATA DELIVERABLE (EDD) FORMAT

BEAZER EAST, INC.
FORMER KOPPERS WOOD-TREATING SITE
CARBONDALE, ILLINOIS
QUALITY ASSURANCE PROJECT PLAN

Field Name	Maximum Length	Data Type	Comments
FIELD SAMPLE ID	50	TEXT	From the chain of custody. Add "RE" or "DL" to differentiate reanalyses and dilutions.
SDG	50	TEXT	
LAB SAMPLE ID	50	TEXT	
MATRIX	10	TEXT	SOIL, WATER, SEDIMENT, etc.
SAMPLE TYPE	10	TEXT	FB, RB, TB, FD, FS for Field Blank, Rinse Blank, Trip Blank, Field Duplicate and Field Sample, respectively. DEFAULT TO FS
DATE COLLECTED	--	DATE/TIME	MM/DD/YY
TIME COLLECTED*	--	DATE/TIME	Military time
DEPTH START	--	NUMBER	
DEPTH END	--	NUMBER	
DEPTH UNITS	25	TEXT	FEET, INCHES, METERS, etc.
ANALYTICAL METHOD	50	TEXT	
CAS NUMBER	25	TEXT	
ANALYTE	100	TEXT	
RESULT VALUE	--	NUMBER	For non-detected results, enter Reporting Limit ("U" must be present in Lab Qualifier field).
LAB QUALIFIER	10	TEXT	"U" for non-detected, others as defined by laboratory.
REPORTING LIMIT	--	NUMBER	
RESULT UNIT	25	TEXT	
DILUTION FACTOR	--	NUMBER	
REPORTABLE RESULT	--	YES/NO	DEFAULT TO YES
FILTERED?	--	YES/NO	
DATE ANALYZED	--	DATE/TIME	MM/DD/YY
TIME ANALYZED*	--	DATE/TIME	Military time
DATE EXTRACTED*	--	DATE/TIME	MM/DD/YY
LABORATORY NAME*	50	TEXT	

Notes:
1 . This definition is for an "Excel-type" spreadsheet. Fields flagged with an "*" are optional and may be left blank if not available electronically from the laboratory.
2. Depth-related fields may be left blank for samples and matrices for which they are not applicable.

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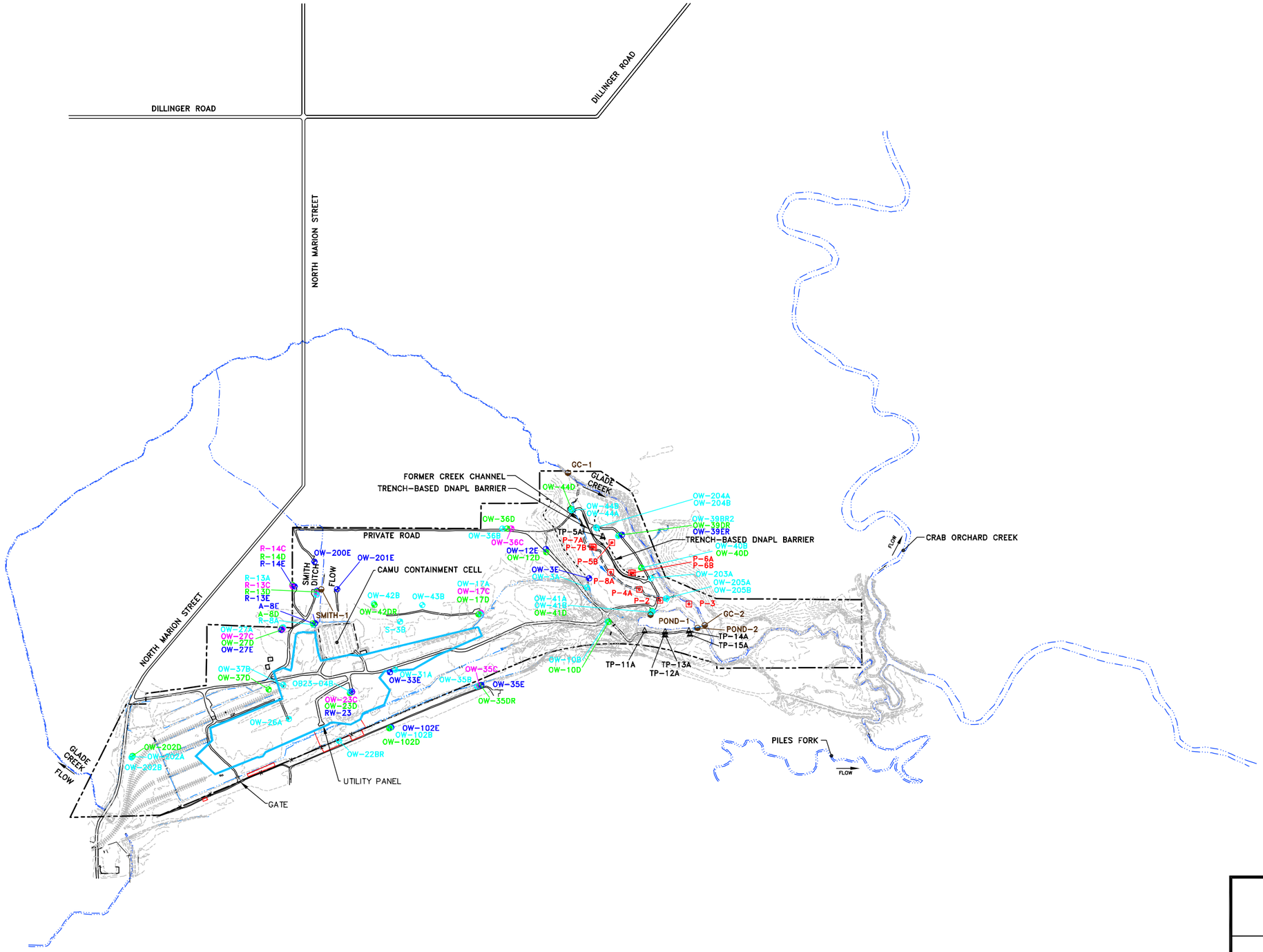
Figure



LEGEND:

- EDGE OF WATER
- DRAINAGE DITCH AND DIRECTION OF FLOW
- PROPERTY BOUNDARY (SEE NOTE 2)
- FENCELINE
- INDEX CONTOUR
- INTERMEDIATE CONTOUR
- FORMER PROCESS AREA SURFACE COVER
- SOIL REMOVAL AREA (SOIL/DEBRIS PILES AND SURFICIAL ASPHALT-LIKE MATERIALS)
- "A" AND "B" UNIT MONITORING WELL
- "C" UNIT MONITORING WELL
- "D" UNIT MONITORING WELL
- "E" UNIT MONITORING WELL
- PIEZOMETER
- TEMPORARY PIEZOMETERS
- SURFACE WATER GAUGE
- RECOVERY WELL

NOTES:

1. SITE FEATURES AND TOPOGRAPHY OBTAINED FROM PHOTOGRAMMETRIC MAPPING PROVIDED BY LOCKWOOD MAPPING COMPANY IN SEPTEMBER 2001 AND SURVEY DATA PROVIDED BY ENGINEERING DESIGN SOURCE, INC. IN JANUARY 2004, AND BASED ON AERIAL PHOTOGRAPHY PROVIDED BY LOCKWOOD MAPPING, INC. TAKEN ON NOVEMBER 22, 1996 AT AN APPROXIMATE SCALE OF 1'=500'. TOPOGRAPHY IN THE EASTERN SOUTHERN DRAINAGE DITCHES AREA BASED ON TOPOGRAPHIC FIELD SURVEY CONDUCTED IN AUGUST 2007.
2. PROPERTY BOUNDARY IS APPROXIMATE; OBTAINED FROM A COMBINATION OF SITE SURVEY DATA, HISTORICAL MAPS AND TAX MAPS.

0 500' 1000'
GRAPHIC SCALE

BEAZER EAST, INC.
FORMER KOPPERS WOOD TREATING SITE
CARBONDALE, ILLINOIS

SITE PLAN

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FIGURE
1

CITY: SYR DIV/GROUP: 85 DB: LAF LD: AM: PD: TM: TR: LYR:ON=*;OFF=*REF*
G:\CADACT\B0039093\0000\0008\DWG\SITE\39093G01.DWG LAYOUT: 1 SAVED: 2/7/2008 1:42 PM ACADVER: 17.0S (LMS TECH) PAGESETUP: C4.D2B-PDF PLOTSTYLETABLE: PLTFULL.CTB PLOTTED: 2/7/2008 1:43 PM BY: FORAKER, LYDIA
XREFS: 39093X01 39093X02 IMAGES: PROJECTNAME: —

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Appendices

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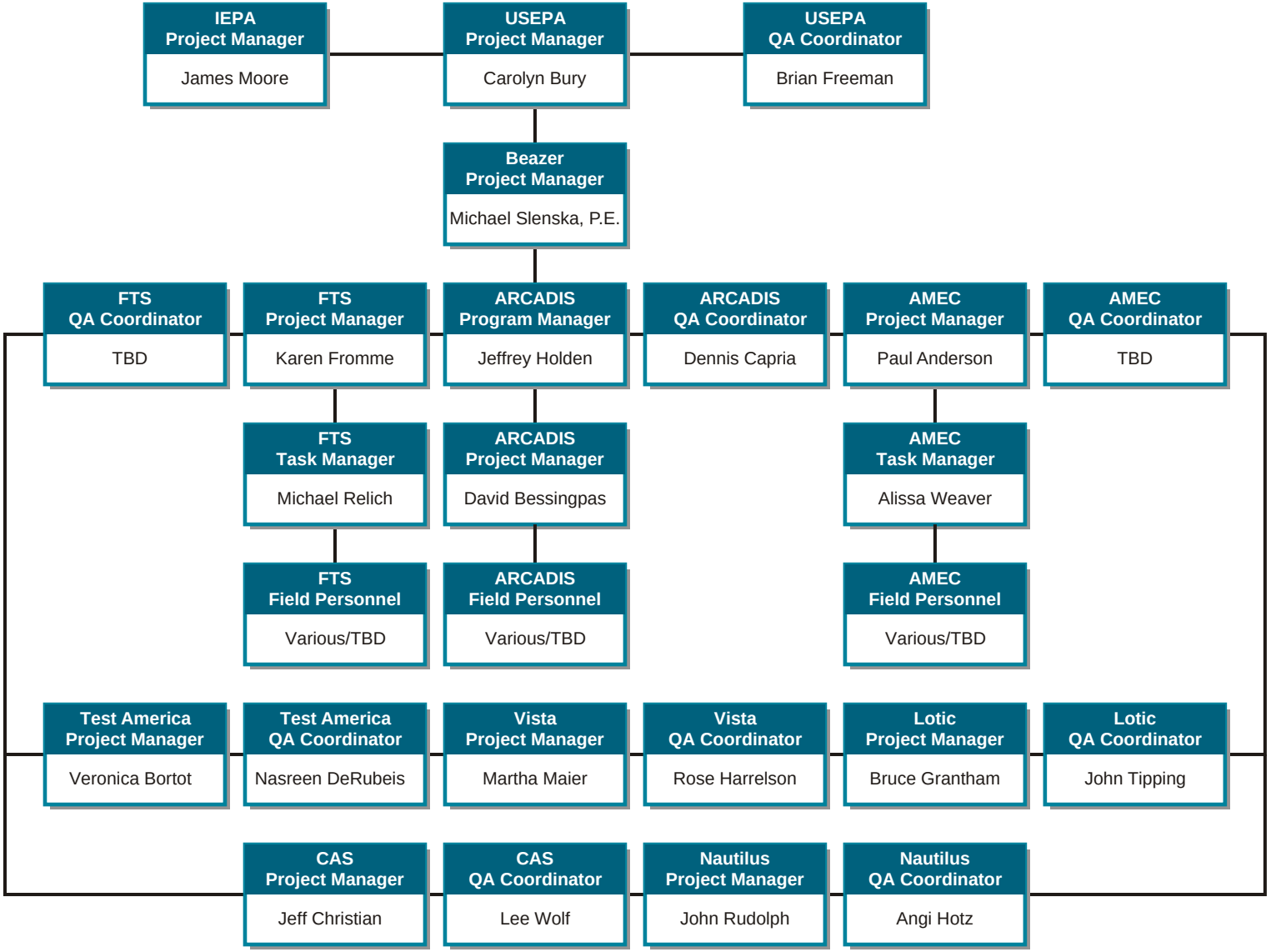
Appendix A

Project Organization Chart

Project Organization Chart



NOTE:
QA = Quality Assurance
TBD = To Be Determined

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Appendix B

Chain of Custody

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Infrastructure, environment, facilities

ID#:

CHAIN OF CUSTODY & LABORATORY ANALYSIS REQUEST FORM

Page ___ of ___

Lab Work Order #

Send Results to:

Contact & Company Name:	Telephone:
Address:	Fax:
City State Zip	E-mail Address:
Project Name/Location (City, State):	Project #:
Sampler's Printed Name:	Sampler's Signature:

Preservative							
Filtered (✓)							
# of Containers							
Container Information							

PARAMETER ANALYSIS & METHOD

Keys

Preservation Key:
A. H_2SO_4
B. HCL
C. HNO_3
D. NaOH
E. None
F. Other: _____
G. Other: _____
H. Other: _____

Container Information Key:
1. 40 ml Vial
2. 1 L Amber
3. 250 ml Plastic
4. 500 ml Plastic
5. Encore
6. 2 oz. Glass
7. 4 oz. Glass
8. 8 oz. Glass
9. Other: _____
10. Other: _____

Matrix Key:
SO - Soil, SE - Sediment, NL - NAPL/Oil
W - Water, SL - Sludge, SW - Sample Wipe
T - Tissue, A - Air, Other: _____

Sample ID	Collection Date	Collection Time	Type (✓) Comp	Type (✓) Grab	Matrix									REMARKS

Special Instructions/Comments:

☐ **Special QA/QC Instructions(✓):**

Laboratory Information and Receipt		Relinquished By	Received By	Relinquished By	Laboratory Received By
Lab Name:	Cooler Custody Seal (✓)	Printed Name:	Printed Name:	Printed Name:	Printed Name:
☐ Cooler packed with ice (✓)	☐ Intact ☐ Not Intact	Signature:	Signature:	Signature:	Signature:
Specify Turnaround Requirements:	Sample Receipt:	Firm:	Firm/Courier:	Firm/Courier:	Firm:
Shipping Tracking #:	Condition/Cooler Temp: _____	Date/Time:	Date/Time:	Date/Time:	Date/Time:

Distribution: WHITE – Laboratory returns with results YELLOW – Lab copy PINK – Retained by BBL

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Attachments

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Attachment A

Field Sampling Standard Operating Procedures

LOW-FLOW (MINIMAL DRAWDOWN) GROUNDWATER SAMPLING PROCEDURES

1.0 SCOPE AND PURPOSE

This standard operating procedure (SOP) provides the procedures to be used to collect groundwater samples using the low-flow (minimal drawdown) sampling method. This method reduces sample turbidity, mixing between the overlying stagnant casing water and water within the screened interval, and the volume of purge water generated. Typically, flow rates on the order of 0.1-0.5 liter/minute are used, however, the actual flow rate used at a given well will depend in large part upon the hydraulic conductivity of its screened interval.

2.0 REQUIRED MATERIALS

The following list identifies the types of equipment which may be used during groundwater sampling. Project-specific equipment should be selected based upon project objectives, the depth to groundwater, required analytical parameters, and well construction information. The types of groundwater sampling equipment are as follows:

- Purging/Sample Collection Equipment
  - Low-flow (e.g., 0.1-0.5 liter/minute) pumps such as peristaltic pumps, bladder pumps, electrical submersible pumps, and gas-driven pumps;
  - Pumps are to be constructed of stainless steel or equipped with disposable and/or dedicated Teflon™ hardware;
  - (Note that bailers are inappropriate devices for low-flow sampling.)

- Related sampling and field measurement equipment will include some or all of the following:
  - A multi-parameter measurement unit with in-line sampling capability such as a YSI 556 or equivalent;
  - A photoionization detector (PID) to monitor for volatile organic constituents upon opening the monitoring well cap (the need for this instrument will be specified in the project specific work plan or health and safety plan);
  - Turbidity meter;
  - An in-line filtration apparatus, 0.45 μm, if dissolved metals are required analytes;
  - A water level meter; and,
  - An interface probe, if light non-aqueous phase liquid (NAPL) or dense NAPL are potentially present on site (the need for this instrument will be specified in the project-specific work plan).

- General Equipment:
  - Safety Glasses or equivalent eye protection;
  - Distilled water and dispenser bottle;
  - Decontamination solutions (such as Alconox and solvents);
  - Field data sheets;
  - Sample preservation solutions;
  - Sample containers;
  - Buckets and intermediate containers;
  - Coolers;
  - Shipping labels;
  - Permanent markers/pens;
  - Packing tape;
  - First aid kit;
  - Graduated cylinder;
  - Key(s) for well locks; and,
  - Stopwatch.

- Disposable Materials:
  - Plastic sheeting/bags;
  - Pump tubing (and bladders if bladder pumps are used);
  - Gloves;
  - Filters;
  - Chemical-free paper towels; and,
  - Protective coveralls (e.g., Tyvek), if necessary.

3.0 METHODOLOGIES

3.1 Pre-Sampling Considerations

Water samples should not be collected immediately following well development. Sufficient time should be allowed for the groundwater flow regime in the vicinity of the monitoring well to stabilize and to approach chemical equilibrium with the well construction materials. Unless stated otherwise in appropriate project-specific documents, or required by the lead regulatory agency for the site, the time between developing and sampling a well will be one week.

Several preparatory activities need to be completed prior to sampling of each well. These preparatory activities can be summarized as follows:

- Log in sample bottles received from laboratory, prepare any deionized water or preservatives needed for the sampling;

- If non-dedicated sampling pumps are used, prepare pumps with standard decontamination procedures;

- Don the necessary personnel protective equipment (PPE) stipulated in the Site health and safety plan (HASP);

- Measure static water level prior to well purging. Water levels will be measured to the nearest hundredth of a foot with an electronic probe from the established measuring point of the well casing. If water levels will be used to determine groundwater flow direction and/or hydraulic gradients, water levels from all locations requiring measurement shall be collected over as short a time period as possible. Unless specified otherwise in project-specific documents, light/dense NAPL thickness and total well depth will be measured at each well during the initial round of water level measurements. All well gauging data will be recorded on a site specific well gauging sheet.

3.2 Equipment Calibration

Prior to purging and sampling, all sampling devices and monitoring equipment should be calibrated according to manufacturer's recommendations and project-specific documents such as a Quality Assurance Project Plan (QAPP) or Field Sampling Plan (FSP). Note that some types of dissolved oxygen measuring devices require that corrections be made for local barometric pressure and elevation during calibration.

3.3 Well Purging

The low-flow, minimal drawdown method relies on minimizing drawdown in the well being sampled (which minimizes increases to in-situ groundwater velocities). Groundwater is determined to be representative of in-situ conditions and therefore ready to sample when selected indicator parameters have stabilized, as described later in this section.

The procedures used to purge a well may vary slightly depending upon the construction of the well and the type of pump used. Details regarding selecting a pump are provided later in this section. General purging requirements are as follows:

- establish a flow rate (when attainable between 0.1L/min and 0.5 L/min), that maintains minimal drawdown in the well during both purging and sampling;
- maximize pump discharge tubing wall thickness and minimize tubing length;
- place the sampling device intake at the middle of the saturated portion of the screened interval (or open bedrock interval for open borehole bedrock wells), unless specified otherwise in the project-specific work plan
- minimize disturbance of the stagnant water column above the screened interval when measuring water levels and inserting the sampling device;
- make proper adjustments to stabilize the flow rate as soon as possible;
- monitor water quality indicator parameters during purging (as described below in Section 3.4);
- either dedicate pump tubing to each well, or properly dispose of it after use;
- handle and dispose of all purge water and other investigation-derived wastes as specified in the appropriate project-specific document(s).

PUMP AND DISCHARGE TUBING SELECTION

There are no unusual requirements for groundwater sampling devices when using low-flow, minimal drawdown techniques. The primary requirement is that the device give consistent results and minimal disturbance of the sample across a range of low flow rates (i.e., <0.5 liter/minute). Note that pumping rates that cause minimal to no drawdown in one well could easily cause significant drawdown in another well that has been installed in a less transmissive formation. Consistency in operation is critical to meet accuracy and precision goals. The goal is to achieve and maintain as low a flow rate as possible while maintaining non turbulent flow (i.e. no air bubbles in groundwater conveyance line with minimal flow rate variation).

There are several pumps which are used frequently for purging or sampling. These types include the peristaltic, bladder, and submersible pumps. It is desirable that the pump be easily adjustable and operates reliably at these lower flow rates. Gas-driven pumps should be of a type that does not allow the gas to be in direct contact with the sampled fluid. Bailers and other grab-type samplers are not suited for low-flow sampling and shall not be used.

A variety of tubing materials are available for use as pump discharge tubing. The type of tubing used to collect the sample will be contingent on the parameters of interest.

a. If conventional parameters (i.e., biological oxygen demand [BOD], total suspended solids [TSS], fecal coliform, pH, and oil and grease) are being analyzed, then standard Nalgene or polyethylene tubing is sufficient to collect the sample.

b. If volatile, semi-volatile, or metals parameters are the constituents of interest, Teflon™ or Teflon lined tubing will be used to collect the sample.

Unless specified otherwise in project-specific documents, or required by the lead regulatory agency for the site, the material used for sampling tubing shall be Teflon™ lined polyethylene (either high- or low-density). If a peristaltic pump is used, a short length of silicone tubing can be used through the pump head.

Peristaltic Pumps

Peristaltic pumps are operated above ground next to the well and are limited to water level depths of 20 to 30 feet below ground surface. The following procedure describes the use of peristaltic pumps for purging and sampling.

1. Carefully lower the pump tubing to the targeted depth down the well in a manner that minimizes mixing of the water column and disturbance of solids that may have collected at the well bottom.

2. Connect the tubing to a short length of silicone tubing in a manner that will allow the purge water to contact only polyethylene, Teflon or silicone materials (e.g., using connector fitting made of polyethylene or Teflon).

3. Run the silicone tubing through the pump head, connecting the discharge end to an in-line water-quality meter (these meters are described in Section 3.4). Place the discharge line from the water-quality meter into a container (e.g., 5-gallon bucket or 55-gallon drum) to collect the purged water.

4. Begin pumping at a low rate, while periodically monitoring the depth to water and the pumping rate. Measure the pumping rate using a calibrated container and a stopwatch. Record pumping-rate and water-level measurements.

5. Adjust the pumping rate as necessary to maintain minimal drawdown.

6. When the indicator parameters have stabilized (as described in Section 3.4), begin sampling (see section 3.5).

7. When sampling is completed, and before shutting the pump off, record the total pumping time and total volume of groundwater purged.

Bladder Pumps

The bladder pump is a compressed air or gas-operated, positive displacement submersible well pump that uses inert compressed gas, e.g., nitrogen, to inflate an internal bladder which pumps water up the discharge line. Usually these pumps are used on wells with diameters of 2 inches or greater and wells with depths up to 150 feet. For non-dedicated pumping assemblies, the air/water conveyance line will either be dedicated or properly disposed of. After use where practical, the tubing will be suspended within the well for future use in the well to which it is dedicated. For non-dedicated bladder pump assemblies, a new, disposable bladder will be used at each well

The following procedures should be followed for using the bladder pump:

1. Connect the line assembly to the pump by first attaching the cable and then connecting the sample and gas lines.

2. Lower the pump slowly down the well as to minimize excessive mixing of the stagnant water and solids that have collected at the bottom of the well by unrolling the line off the spool until the pump is located at the desired position inside the well.

3. Secure the cable to hold the pump at the desired depth.

4. Connect the gas line to the control box. Connect the discharge end to an in-line water-quality meter (these meters are described in Section 3.4). Place the discharge line from the water-quality meter into a container (e.g., 5-gallon bucket or 55-gallon drum) to collect the purged water.

5. Connect the gas supply to the control box and adjust the pressure according to the manufacturer's manual.

6. Begin pumping at a low rate, while periodically monitoring the depth to water and the pumping rate. Measure the pumping rate using a calibrated container and a stopwatch. Record pumping-rate and water-level measurements.

7. Adjust the pumping rate as necessary to maintain the lowest consistent flow rate with minimal drawdown.

8. When the indicator parameters have stabilized (as described in Section 3.4), begin sampling (see section 3.5).

9. When sampling is completed, and before shutting the pump off, record the total pumping time and total volume of groundwater purged.

10. Those non-disposable and/or non-dedicated portions of the pumping assembly that come in contact with water in the well must be decontaminated, as specified in the appropriate, project-specific document(s), before being used in another well.

Submersible Pumps

Submersible pumps that are specifically designed for groundwater sampling and can maintain the required low flow rates can be used for purging and sampling.

1. Connect tubing to the submersible pump and lower pump to the desired depth using a safety line that is secured to the well casing.

2. Connect the discharge line to the water quality meter or flow-through cell, with cell discharge line placed into a container (e.g., 5-gallon bucket or 55-gallon drum) to collect the purged water.

3. Connect the power cord to the power source and turn on the pump.

4. Begin pumping at a low rate, while periodically monitoring the depth to water and the pumping rate. Measure the pumping rate using a calibrated container and a stopwatch. Record pumping-rate and water-level measurements.

5. Adjust the pumping rate as necessary to maintain minimal drawdown.

6. When the indicator parameters have stabilized (as described in Section 3.4), begin sampling (see section 3.5).

7. When sampling is completed, and before shutting the pump off, record the total pumping time and total volume of groundwater purged.

8. Those portions of the pump and power line that come in contact with water in the well must be decontaminated, as specified in the appropriate, project-specific document(s), before being used in another well.

Continue to monitor the pumping rate and water level in the well, slowing the rate if drawdown occurs.

3.4 Monitoring Water Level and Water Quality Indicator Parameters

Check the water level periodically during purging and sampling to monitor drawdown in the well as a guide to flow rate adjustment. The goal is minimal drawdown (<0.3 feet) during purging. This goal may not be possible to achieve under some circumstances. If this goal cannot be met, pumping should continue at the lowest flow rate that can be maintained by the pump. In the event that the water level in the well reaches the pump intake, pumping will be discontinued, leaving the pump/tubing in place while the water level in the well recovers. When the water level has recovered sufficiently, pumping will be resumed at the same low flow rate and sampling will be completed. In this special case, the pumped groundwater can be directed into the sampling containers immediately.

The following indicator parameters will be monitored, unless specified otherwise in project-specific documents:

- temperature;
- pH;
- ORP;
- conductivity,
- dissolved oxygen; and,
- turbidity.

Unless specified otherwise in project specific documents, an in-line water-quality meter will be used to monitor all of these parameters, with the possible exception of turbidity. Turbidity can either be monitored with a flow-through cell or with a separate turbidity meter. Measurements of all of these parameters should be recorded every three to five minutes. The groundwater is considered representative and ready for sampling as soon as the indicator parameters meet the following criteria for three consecutive readings:

- $\pm 0.1^{\circ}$ C for temperature;
- ± 0.1 standard units for pH;
- $\pm 3\%$ for conductivity;
- ± 10 millivolts for ORP;
- $\pm 10\%$ for turbidity; and,
- $\pm 10\%$ for dissolved oxygen.

If one-or more of these criteria can not be met, field staff will contact the project manager, who will determine the appropriate course of action.

Pumping rate, drawdown, and the time or volume required to obtain stabilization of parameter readings can be used as a future guide to purging and sampling the well.

3.5 Groundwater Sampling

Once parameters have stabilized, begin collecting samples as soon as possible. Disconnect or bypass the in-line water-quality meter prior to collecting samples. The sampling flow rate should remain the same as that used during purging.

Samples will be collected in decreasing order of their volatility. This order is generally as follows:

- Volatile organic chemicals (VOCs);
- Total organic halogens (TOX);
- Gas sensitive parameters (e.g., Fe2+, CH4, H2S/HS-, alkalinity);
- Total organic carbon (TOC);
- Semivolatile organics chemicals (SVOCs);
- Inorganic parameters; and,
- If filtered samples are to be collected, these should be collected last. Filters are to be pre-rinsed following the manufacturer's recommendations. If there are no recommendations for pre-rinsing, when practical, a minimum of 1L or groundwater must pass through the filter prior to sample collection.

Samples collected for volatile organics should be carefully placed into 40 milliliter glass vials with Teflon septum lids. No air bubbles should be present in the vial after sealing the septum lid; if air bubbles are present, fill the vial more completely.

Samples collected for dissolved metals analyses, if required, will be filtered in the field. Filtering is performed using an in-line filtration device, hand vacuum pumps with transfer vessels, or peristaltic pumps with disposable funnels/filters. If using the vacuum pump method, a laboratory cleaned transfer vessel is used. If using a peristaltic pump, new silicone tubing is used in the pump head for each sample filtered and new Teflon tubing is used from the pump head to the filter. Samples are filtered through 0.45 micron filter unless specified otherwise in the appropriate, project specific document. Filtrate should be placed in a pre-preserved sampling container as soon as possible.

Sampling technicians must wear a clean pair of disposable gloves for each well.

4.0 QA/QC PROCEDURES

Refer to the appropriate, project-specific documents for the quality assurance/quality control procedures to be followed during low-flow (minimal drawdown) groundwater sampling.

5.0 DATA RECORDING AND MANAGEMENT

A written record of each monitoring event must be maintained. The record provides a summary of the sample collection procedures and conditions, shipment method, the analyses requested and the custody history. This record consists of the following:

- Groundwater Sample Collection Form (Example Form Attached);
- Chain of custody form; and,
- Shipping receipts.

Sample labels shall be completed at the time each sample is collected and will include the information listed below.

- Project name;
- Sample number;
- Time and date;
- Preservative (if applicable);
- Analyses to be performed; and,
- Sampler's name.

6.0 REFERENCES

U.S. EPA, Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures, by Robert W. Puls and Michael J. Barcelona, EPA/540/S-95/504, April 1996.

U.S. EPA, Region II, Ground Water Sampling Procedure - Low Flow Pump Purging and Sampling, March 1998.

ATTACHMENT A

LOW FLOW GROUNDWATER SAMPLE COLLECTION RECORD

FTS

WELL NO.:

GROUNDWATER SAMPLE COLLECTION RECORD

PERMIT NO.:

Project No.: _____ **Client:** Beazer East, Inc.
Project Name: _____ **Project Location:** _____
Weather Conditions: _____ **Sampling Date:** _____

1. WATER LEVEL DATA (measured from top of inner well casing)

a. **Depth to LNAPL:** _____ (ft) b. **Depth to Water:** _____ (ft)
c. **Depth to DNAPL:** _____ (ft) d. **Total Well Depth:** _____ (ft)
e. **LNAPL Thickness:** (a-b) _____ (ft) f. **DNAPL Thickness:** (c-d) _____ (ft)
g. **Length of Water Column:** _____ (ft) (b-d)
h. **Well Volume:** _____ (gal)

2. WELL PURGE DATA

a. **Purge Method:** _____
b. **Field Testing Equipment:** _____
d. **Total Volume and Number of Well Volumes Removed:** _____
e. **Begin Purge Time:** _____ **End Purge Time:** _____

Conversion Factors
(g x cf = h)

Well I.D.	Conv. Fact. (cf)
1	0.041
2	0.163
4	0.653
6	1.470

Read No.	Lapse Time (min.)	Purge Rate	Temp (deg. C) (±0.1°C)	pH (s.u.) (±0.1)	Spec. Cond. (ms/cm) (±3%)	Eh/ORP (mV) (±10mV)	Diss O2 (mg/L) (±10%)	TURB (NTU) (±10%)	Salinity %	TDS G/L	Water Level (ft)
PRE PURGE VALUES											
PURGING VALUES											

3. SAMPLE COLLECTION DATA **Sampling Personnel:** _____

Sampling Method(s) & Equip: _____
Sample I.D. (Name, Date, Time): _____
Sample Analytical Parameters/Method: _____

Sample Start Time: **End SampleTime:**
COMMENTS:

Standard Operating Procedure: Soil Sample Collection (Using Manual Methods)

I. Scope and Application

This Standard Operating Procedure (SOP) describes the procedures that will be followed to collect soil samples using manual methods at the Former Koppers Wood-Treating Site in Carbondale, Illinois.

II. Personnel Qualifications

ARCADIS BBL field sampling personnel will have current health and safety training, including 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response Standard (HAZWOPER) training, site supervisor training, Site-specific training, first aid and cardiopulmonary resuscitation (CPR), as needed. ARCADIS BBL field sampling personnel will be well versed in the relevant SOPs and possess the required skills ad experience necessary to successfully complete the field work.

III. Equipment List

Soil Sampling

The following materials will be needed during sediment sampling activities:

- copy of Work Plan;
- health and safety equipment, as required by the Site-specific Health and Safety Plan (HASP);
- sampling equipment (stainless steel hand-spades, Lexan tubes with end caps, stainless steel hand auger, and/or Macrocore sampler with liners);
- core driver (for Lexan tubes or Macrocore sampler);
- ruler/measuring tape;
- hacksaw;

- duct tape;
- aluminum or stainless steel trays/bowls and spoons/spatulas;
- transport container with ice;
- appropriate sample containers and forms;
- field notebook; and
- garbage bags.

Equipment Cleaning

The following materials will be needed for equipment cleaning:

- health and safety equipment, as required in the HASP;
- deionized or distilled analyte free water; non-phosphate soap (Alconox or equivalent);
- appropriate cleaning solvent (e.g., hexane);
- buckets for cleaning equipment in and containing cleaning fluids;
- brushes;
- aluminum foil; and spray bottles for cleaning fluids.

IV. Cautions

Uneven/slippery terrain and obstacles (e.g., brush, fallen tree, etc.) exist at the Site; sampling personnel should exercise caution to avoid slips, trips, and falls.

Prior to performing any intrusive activity, ARCADIS BBL will confirm that all underground utilities have been identified and marked out. The presence of some utility markings does NOT mean that all of the underground lines have been located. Do not allow drilling or excavating, or any other intrusive activity, to begin until you have confirmed that notified utilities have either responded to or cleared the work site.

V. Health and Safety Considerations

Refer to HASP.

VI. Procedures

Soil Sample Collection Procedures

Soil sampling may be conducted with stainless steel hand spades, Lexan tubes, hand augers, and/or Macrocore samplers; sample collection procedures are as follows:

1. Locate the targeted sampling location in the field and review Work Plan to confirm sampling scope.

2. Identify the sample location in the field notebook along with a general description of the sampling location.

3. Don personal protective equipment (PPE), as required by the HASP.

4. Clear the ground surface of brush, root mat, grass, leaves and other debris.

5. Use a stainless steel hand spade, Lexan tube, hand auger, or Macrocore sampler to obtain soil samples.

6. Use a ruler to verify the sample collection depths; record measurements in field book.

7. Place recovered soils onto an aluminum or stainless steel tray.

8. Characterize the entire soil core for color, texture, visual impacts, and odors as a function of depth.

9. Homogenize the soils from the targeted sample interval(s) with a stainless steel spoon or spatula.

10. Place each soil sample into the appropriate laboratory-supplied sample containers.

11. Label all sample containers with sample ID and depth interval, sample date/time, samplers' names, and requested analytical methods.

12. Handle, pack, and ship samples using standard chain-of-custody procedures.

13. Backfill hole with excess soils, or other suitable materials.

Equipment Cleaning Procedures

Non-dedicated and non-disposable sampling equipment will be cleaned prior to use at each sampling location. Equipment cleaning procedures will be as follows:

- wash with non-phosphate detergent and distilled/deionized water to remove visible particulate mater and oils;
- rinse with distilled/deionized water;
- triple rinse sequence of solvent (e.g. hexane, methanol, nitric acid) followed by distilled/deionized water; and
- air dry and wrap in aluminum foil.

All cleaning fluids will be collected and appropriately containerized for subsequent disposal

VII. Waste Management

Waste materials generated during the sediment sampling activities and disposable equipment will be placed into suitable containers for subsequent characterization and appropriate disposal by Beazer.

VIII. Data Recording and Management

The field supervisor will maintain a field notebook as a record of the field program and provide a copy to the Project Manager. With respect to sediment probing and sampling, the following details will be recorded (in addition to the information discussed above):

- project name and location;
- project objectives;
- daily log of activities;
- personnel present and involved in the sampling;
- weather conditions;
- rationale for field decisions, as appropriate;
- deviations from the stated procedures in the Work Plan or SOPs; and
- any health and safety incidents or “near-misses”.

IX. Quality Assurance

Refer to the Site-specific Quality Assurance Project Plan.

Standard Operating Procedure: Sediment Probing and Sample Collection

I. Scope and Application

This Standard Operating Procedure (SOP) describes the procedures that will be followed to collect sediment samples at the Former Koppers Wood-Treating Site in Carbondale, Illinois.

II. Personnel Qualifications

ARCADIS BBL field sampling personnel will have current health and safety training, including 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response Standard (HAZWOPER) training, site supervisor training, Site-specific training, first aid and cardiopulmonary resuscitation (CPR), as needed. ARCADIS BBL field sampling personnel will be well versed in the relevant SOPs and possess the required skills ad experience necessary to successfully complete the field work.

III. Equipment List

Sediment Sampling

The following materials will be needed during sediment sampling activities:

- copy of Work Plan;
- health and safety equipment, as required by the Site-specific Health and Safety Plan (HASP);
- aluminum boat with outboard motor (if needed, depending on water depths);
- chest and/or hip waders;
- metal push rod;
- 6-foot rule or survey rod;
- Lexan tubing with end caps and/or dredge sampler;

- core driver;
- vacuum pump;
- hacksaw;
- duct tape;
- aluminum or stainless steel trays/bowls and spoons/spatulas; transport container with ice;
- appropriate sample containers and forms;
- field notebook; and
- garbage bags.

Equipment Cleaning

The following materials will be needed for equipment cleaning:

- health and safety equipment, as required in the HASP;
- deionized or distilled analyte free water; non-phosphate soap (Alconox or equivalent);
- appropriate cleaning solvent (e.g., hexane);
- buckets for cleaning equipment in and containing cleaning fluids;
- brushes;
- aluminum foil; and spray bottles for cleaning fluids.

IV. Cautions

Sampling personnel should be cautious of working in and around water during sampling and sample processing activities. Sampling personnel must be aware of weather conditions at all times as creek water levels rise quickly during storm events.

Uneven/slippery terrain and obstacles (e.g., brush, fallen tree, etc.) exist in the uplands area (which will need to be traversed to reach the creeks), on the creek banks, and in the creeks themselves; sampling personnel should exercise caution to avoid slips, trips, and falls.

V. Health and Safety Considerations

Refer to HASP.

VI. Procedures

Sediment Probing Procedures

1. Locate the targeted probing location in the field.

2. Identify the probing location in the field notebook along with a general description of the creek conditions at the probing location.

3. Don personal protective equipment (PPE), as required by the HASP.

4. Prior to probing, measure water depth at probing location and record measurement in field notebook.

5. Push metal rod into sediments by hand until refusal; record sediment thickness in field book.

6. Repeat Steps 4 and 5 at various intervals along a transect aligned perpendicular to the creek flow, as required by the Work Plan.

Surficial Sediment Sampling Procedures (Using Dredge Sampler)

1. Locate the targeted sampling location in the field and review the Work Plan to confirm sampling scope.

2. Identify the sample location in the field notebook along with a general description of the creek conditions at the sampling location.

3. Don personal protective equipment (PPE), as required by the HASP.

4. At each sample location, lower the dredge sampler (attached to a rope) until it just reaches the top of sediment. Measure and record the depth of water.

5. Once the dredge has been allowed to settle into the sediment, pull hard on the rope to collect the sediment inside the dredge.

6. Retrieve the dredge and empty sediments into an aluminum or stainless steel tray or bowl and record general sediment descriptions, including color, texture, visual impacts, and odors.

7. Repeat Steps 4 through 6 at various locations across the creek, in accordance with the Work Plan, until sufficient sample volume is obtained for the required analyses.

8. Homogenize the sediments with a stainless steel spoon or spatula and record the sediment sample color, texture, visual impacts, and odors.

9. Place each sediment sample into the appropriate laboratory-supplied sample containers.

10. Label all sample containers with sample ID and depth interval, sample date/time, samplers' names, and requested analytical methods.

11. Handle, pack, and ship samples using standard chain-of-custody procedures.

Surficial Sediment Sampling Procedures (Using Lexan Tubes)

1. Locate the targeted sampling location in the field and review Work Plan to confirm sampling scope.

2. Identify the sample location in the field notebook along with a general description of the creek conditions at the sampling location.

3. Don personal protective equipment (PPE), as required by the HASP.

4. At each sample location, lower a section of Lexan tube until it reaches the top of sediment. Measure and record the depth of water.

5. Push the Lexan tube into the sediment by hand (or use a core driver if needed) to the targeted surficial sediment sample depth.

6. Pump the water out from inside the Lexan tube and place a vacuum pump on the top end of the Lexan tube and create a vacuum to prevent the sediments from escaping; or create a vacuum by filling the Lexan tube completely with water and place a cap on the top of the tube.

7. Slowly pull the tube from the sediment, twisting it slightly as it is removed (if necessary).

8. Before the tube is fully removed from the water, place a cap on the bottom end of the tube while it is still submerged.

9. Empty sediments from Lexan tube into an aluminum or stainless steel tray or bowl and record general sediment descriptions, including color, texture, visual impacts, and odors.

10. Repeat Steps 4 through 9 at various locations across the creek, in accordance with the Work Plan, until sufficient sample volume is obtained for the required analyses.

11. Homogenize the sediments with a stainless steel spoon or spatula and record the sediment sample color, texture, visual impacts, and odors.

12. Place each sediment sample into the appropriate laboratory-supplied sample containers.

13. Label all sample containers with sample ID and depth interval, sample date/time, samplers' names, and requested analytical methods.

14. Handle, pack, and ship samples using standard chain-of-custody procedures.

Deep Sediment Sampling Procedures (Using Lexan Tubes)

1. Locate the targeted sampling location in the field and review Work Plan to confirm sampling scope.

2. Identify the sample location in the field notebook along with a general description of the creek conditions at the sampling location.

3. Don personal protective equipment (PPE), as required by the HASP.

4. At each sample location, lower a section of Lexan tube until it just reaches the top of sediment. Measure the depth of water.

5. Push the Lexan tube into the sediment by hand (use a core driver if needed) to approximately three inches past the targeted sample depth, or until refusal. Measure the depth of sediment.

6. Pump the water out from inside the Lexan tube and place a vacuum pump on the top end of the Lexan tube and create a vacuum to prevent the sediments/plug from escaping; or create a vacuum by filling the Lexan tube completely with water and place a cap on the top of the tube.

7. Slowly pull the tube from the sediment, twisting it slightly as it is removed (if necessary).

8. Before the tube is fully removed from the water, place a cap on the bottom end of the tube while it is still submerged.

9. Keeping the tube upright, wipe the bottom end dry and seal the cap with duct tape and label. Measure the length of sediment recovered and evaluate the integrity of the core. If the core is not suitably intact, repeat coring procedure within 5 to 10 feet of the first location attempted.

10. While still keeping the core upright, use a hacksaw to make a horizontal cut in the tube approximately 1 inch above the sediment.

11. Re-cap the cut end of the tube, seal the cap with duct tape, and mark this end as “top.”

12. Wipe the tube dry.

13. Place a completed sample label on the tube.

14. Record the following information on both the tube and on the cap: 1) sample ID; 2) sampling date; and 3) sampling time.

15. Extrude the sediment core from the Lexan tube onto an aluminum or stainless steel tray.

16. Characterize the entire sediment core for color, texture, visual impacts, and odors as a function of depth.

17. Homogenize the sediments from the targeted sample interval(s) with a stainless steel spoon or spatula.

18. Place each sediment sample into the appropriate laboratory-supplied sample containers.

19. Label all sample containers with sample ID and depth interval, sample date/time, samplers' names, and requested analytical methods.

20. Handle, pack, and ship samples using standard chain-of-custody procedures.

Equipment Cleaning Procedures

Non-dedicated and non-disposable sampling equipment will be cleaned prior to use at each sampling location. Equipment cleaning procedures will be as follows:

- wash with non-phosphate detergent and distilled/deionized water to remove visible particulate mater and oils;
- rinse with distilled/deionized water;
- triple rinse sequence of solvent (e.g. hexane, methanol, nitric acid) followed by distilled/deionized water; and
- air dry and wrap in aluminum foil.

All cleaning fluids will be collected and appropriately containerized for subsequent disposal

VII. Waste Management

Waste materials generated during the sediment sampling activities and disposable equipment will be placed into suitable containers for subsequent characterization and appropriate disposal by Beazer.

VIII. Data Recording and Management

The field supervisor will maintain a field notebook as a record of the field program and provide a copy to the Project Manager. With respect to sediment probing and sampling, the following details will be recorded (in addition to the information discussed above):

- project name and location;
- project objectives;
- daily log of activities;
- personnel present and involved in the sampling;
- weather conditions;
- rationale for field decisions, as appropriate;
- deviations from the stated procedures in the Work Plan or SOPs; and
- any health and safety incidents or “near-misses”.

IX. Quality Assurance

Refer to the Site-specific Quality Assurance Project Plan.

Standard Operating Procedure: Surface Water Sample Collection

I. Scope and Application

This Standard Operating Procedure (SOP) describes the procedures that will be followed to collect surface water samples at the Former Koppers Wood-Treating Site in Carbondale, Illinois.

II. Personnel Qualifications

ARCADIS BBL field sampling personnel will have current health and safety training, including 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response Standard (HAZWOPER) training, site supervisor training, Site-specific training, first aid and cardiopulmonary resuscitation (CPR), as needed. ARCADIS BBL field sampling personnel will be well versed in the relevant SOPs and possess the required skills ad experience necessary to successfully complete the field work.

III. Equipment List

- copy of Work Plan;
- health and safety equipment, as required by the Site-specific Health and Safety Plan (HASP);
- aluminum boat with outboard motor (if needed, depending on water depths);
- chest and/or hip waders;
- 6-foot rule or survey rod;
- transport container with ice;
- appropriate sample containers and forms; and
- field notebook.

IV. Cautions

Sampling personnel should be cautious of working in and around water during sampling and sample processing activities. Sampling personnel must be aware of weather conditions at all times as creek water levels rise quickly during storm events. Uneven/slippery terrain and obstacles (e.g., brush, fallen tree, etc.) exist in the uplands area (which will need to be traversed to reach the creeks), on the creek banks, and in the creeks themselves; sampling personnel should exercise caution to avoid slips, trips, and falls.

V. Health and Safety Considerations

Refer to HASP.

VI. Procedure

1. Locate the targeted sampling location in the field and review the Work Plan to confirm sampling scope.

2. Identify the sample location in the field notebook along with a general description of the creek conditions at the sampling location.

3. Don personal protective equipment (PPE), as required by the HASP.

4. Measure and record the depth of water.

5. Fill sample containers by partially submerging into the water, taking care to avoid disturbing bottom sediment and entraining it in the sample. For pre-preserved sample containers, be careful not to release the preservative during sampling.

6. Record general surface water descriptions, including color, visual impacts, and odors.

7. Label all sample containers with sample ID, sample date/time, samplers' names, and requested analytical methods.

8. Handle, pack, and ship samples using standard chain-of-custody procedures.

VII. Waste Management

Waste materials generated during the surface water sampling activities will be placed into suitable containers for subsequent characterization and appropriate disposal by Beazer.

VIII. Data Recording and Management

The field supervisor will maintain a field notebook as a record of the field program and provide a copy to the Project Manager. With respect to surface water sampling, the following details will be recorded (in addition to the information discussed above):

- project name and location;
- project objectives;
- daily log of activities;
- personnel present and involved in the sampling;
- weather conditions;
- rationale for field decisions, as appropriate;
- deviations from the stated procedures in the Work Plan or SOPs; and
- any health and safety incidents or “near-misses”.

IX. Quality Assurance

Refer to the Site-specific Quality Assurance Project Plan.

Standard Operating Procedures

AMEC Earth & Environmental Ltd.

Stream Habitat Surveys

Purpose:

These techniques are used to sample fish habitat within study streams.

Methods:

Habitat surveys can involve two techniques: a general description of habitat types as per Beak (1980) and a more detailed survey of habitat parameters as per Scruton *et al*. (1992), Sooley et al. (1998), and McCarthey et al (2006).

Classification Schemes

1. Beak Classification System

This classification scheme tends to be more generalized and coarse than detailed quantitative information. This technique is most often utilized during aerial classification of salmonid habitat. The attached table provides an explanation of the habitat types (Table 1). This technique is based on describing physical habitat features over a defined area - most often a stream reach or segment of a reach.

Table 1. Characteristics of the four habitat classification types identified by Sooley *et al*. (1998) (originally Beak 1980).

Habitat Classification	Habitat Description
Type I	Good salmonid spawning and rearing habitat: often with some feeding pools for larger age classes: **flows:** moderate riffles; **current:** 0.1-0.3 m/s; **depth:** relatively shallow, 0.3-1.0 m; **substrate:** gravel to small cobble, some large rocks, boulders
Type II	Good salmonid rearing habitat with limited spawning usually only in isolated gravel pockets, good feeding and holding areas for larger fish in deeper pools, pockets or backwater eddies: **flows:** heavier riffles to light rapids; **current:** 0.3-1.0 m/s; **depth:** variable from 0.3-1.5 m; **substrate:** Larger cobble/rubble size rock to boulders, bedrock, some gravel pockets between larger rocks
Type III	Poor rearing habitat with no spawning capabilities, used for migratory purposes: **flows:** very fast, turbulent, heavy rapids, chutes, small falls; **current:** 1.0 m/s or greater; **depth:** variable, 0.3-1.5 m; **substrate:** Large rock and boulders, bedrock

Type IV	Poor juvenile salmonid rearing habitat with no spawning capability, provides shelter and feeding habitat for larger, older salmonid (especially brook trout): **flows:** sluggish; **current:** 0.15 m/s; **depth:** variable but often >1 m; **substrate:** Soft sediment or sand, occasionally large boulders or bedrock, aquatic macrophytes present in many locations

2. New Classification System

Habitat classes should be defined in an ecologically meaningful way (i.e., taking into account how fish utilize their habitat) that can be easily recognized by both field staff and habitat managers. Table 2 provides a description of each habitat type along with the range of parameter values associated with each.

The system is based on easily identifiable habitat characteristics. It is comprised of a series of three levels, each providing progressively more detail about the habitat. This three-level hierarchical system should provide the level of resolution needed for most, if not all, habitat management purposes. Although the third level doesn't provide a further breakdown in habitat characterization, it does add significant information regarding site-specific species utilization of the Intermediate Level habitat types, which may be required for more detailed assessments.

Table 2. Descriptions of riverine habitat classifications.

Habitat Type	Habitat Parameter	Description
Fast Water	**Mean Water Velocity** **Stream Gradient**	**> 0.5m/s** **Generally > 4%.**
Rapid	General Description Mean Water Velocity Mean Water Depth Substrate Stream Gradient	Considerable white water[1] present. > 0.5 m/s < 0.6 m Usually dominated by boulder (Coarse[2]) and rubble (Medium[2]) with finer substrates (Medium and Fine[2]) possibly present in smaller amounts. Larger boulders typically break the surface. Generally 4-7%
Falls/ Chute/ Cascade	General Description Mean Water Velocity Mean Water Depth Substrate Stream Gradient	Mainly white water present. The dominating feature is a rapid change in stream gradient with most water free-falling over a vertical drop or series of drops. > 0.5 m/s Variable and will depend on degree of constriction of stream banks. Dominated by bedrock and/or large boulders (Coarse). > 7% and can be as high as 100%.
Run	General Description Mean Water Velocity Mean Water Depth Substrate Stream Gradient	Relatively swift flowing, laminar[3] and non-turbulent. > 0.5 m/s > 0.3 m Predominantly gravel, cobble and rubble (Medium) with some boulder (Coarse) and sand (Fine) in smaller amounts. Typically < 4% (exception to gradient rule of thumb)
Moderate Water	**Mean Water Velocity** **Stream Gradient**	**0.2-0.5m/s** **>1 and < 4%**
Riffle	General Description Mean Water Velocity	Relatively shallow and characterized by a turbulent surface[4] with little or no white water. 0.2 – 0.5 m/s

Habitat Type	Habitat Parameter	Description
	Mean Water Depth	< 0.3 m
	Substrate	Typically dominated by gravel and cobble (Medium) with some finer substrates present, such as sand (Fine). A small amount of larger substrates (Coarse) may be present, which may break the surface.[5]
	Stream Gradient	Generally >1 and < 4%
Steady/ Flat	General Description	Relatively slow-flowing, width is usually wider than stream average and generally has a flat bottom.
	Mean Water Velocity	0.2 - 0.5 m/s
	Mean Water Depth	>0.2 m
	Substrate	Predominantly sand and finer substrates (Fine) with some gravel and cobble (Medium).
	Stream Gradient	> 1 and < 4%
Slow Water	**Mean Water Velocity**	**Generally < 0.2m/s (some eddies can be up to 0.4m/s).**
	Stream Gradient	**< 1%.**
Plunge / Trench / Debris Pools	General Description	Generally caused by increased erosion near or around a larger, embedded object in the stream such as a rock or log or created by upstream water impoundment resulting from a complete, or near complete, channel blockage. These pool types may be classified as an entire reach (e.g., pools greater than 60% of the stream width) or as sub-divisions of a fast water habitat.
	Mean Water Velocity	< 0.2 m/s
	Mean Water Depth	> 0.5 m depending on stream size (e.g., may be shallower in smaller systems).
	Substrate	Highly variable (i.e., coarse, medium or fine substrates)
	Stream Gradient	Generally < 1%
Eddy	General Description	Relatively small pools caused by a combination of damming and scour: however scour is the dominant forming action. Formation is due to a partial obstruction to stream flow from boulders, roots and/or logs. Partial blockage of flow creates erosion near obstruction. It is typically < 60% of the stream width and hence will be a sub-division of a faster-water habitat type (e.g., Run with 20% eddies).
	Mean Water Velocity	Typically < 0.4 m/s, but can be variable.
	Mean Water Depth	> 0.3 m. May vary depending on obstruction type, orientation, streambed and bank material and flows experienced.
	Substrate	Predominantly sand, silt and organics (Fine) with some gravels (Medium) in smaller amounts.
	Stream Gradient	Variable

White water is present when hydraulic jumps are sufficient to entrain air bubbles which disturb the water surface and reduces visibility of objects in the water.

[2] Coarse, Medium and Fine substrate types are classified according to the Standard Methods Guide for the Classification/Quantification of Lacustrine Habitat in Newfoundland and Labrador (Bradbury *et al*. 2001).

[3] Laminar describes the surface of the water as smooth and glass-like with no reduced visibility of objects in the water.

[4] Turbulence is present if there are local patches of white water or if water movement disturbs a portion of the surface.

[5] Pocket water often constitutes an important component of riffles and is characterized by a predominance of larger substrates (e.g., boulders) breaking the surface. The result is a riffle with many eddies around the boulders.

Stream surveys provide more detailed habitat than that stated above. A thorough, accurate, well-conducted stream survey provides the following information:

- the amount of habitat, both accessible and inaccessible, to migrating fishes which is available for fish production;

- the distribution and proportion of habitat types throughout a watershed including the key habitat attributes that determine habitat types (eg. Substrate type, water depths and velocities, etc.;
- the location and physical description of any barriers to migrating fish using the habitat;
- the locations, types and amounts of stream side vegetation and the stability of stream banks;
- the location and description of areas where habitat has been degraded or destroyed; and
- the accessibility of the watershed for future work.

Stream Notation and Sections:

It is generally standard practice for all habitat observations and records to be taken looking in an upstream direction (i.e., the left-hand side of the river is determined while facing upstream).

The location of the sampling site must be recorded. For large sampling areas or areas with multiple sampling locations on the same waterbody, each sampling site should be numbered. For a single waterbody, sample sites may be numbered sequentially (i.e., #1, #2, etc.). For multiple waterbodies, combining the number with an abbreviation for the waterbody (e.g., ER1 = Exploits River Site #1) may be useful. The type of sampling conducted may also be identified (e.g., GN1 = gill net set #1). All study site abbreviations must be clearly identified. At a minimum, all study sites should be recorded on a 1:50,000 scale topographical map. Other maps or aerial photos may also be used if they provide greater detail than the 1:50,000 map.

Study areas on flowing watercourses are often divided into homogeneous sections called reaches. A reach is a relatively homogenous section of stream having a uniform set of characteristics and habitat types. A reach is relatively uniform with respect to channel morphology, flow volume, gradient and habitat types and is separated from other reaches by changes in these characteristics. Conventionally, reach numbers are assigned in an upstream ascending order, starting at the mouth of the stream. Typically, reach lengths are too long to sample in their entirety, in which case representative study sections should be selected for determining species distribution and abundances. See Scruton *et al.* (1992) for a description of the standard tributary naming protocol used in most freshwater watersheds.

All sampling sites, whether they are point locations (such as a minnow trap site) or sections (such as a section of river that was electrofished), should be recorded on a map of the study area. The standard is to use a 1:50,000 National Topographical Service (NTS) map, but other maps or aerial photos can be used if they provide greater detail. The geographical position of sampling sites can also be recorded using Universal Transverse Mercator (UTM) grid coordinates or by degrees of latitude/longitude. UTM coordinates are particularly useful in case the map is lost as they can be used to pinpoint the sampling site on a new map.

Timing of Surveys:

The ideal time to conduct habitat surveys is during base-flow conditions (i.e., summer or early fall). This is the time of year when stream widths and depths are at their annual lows so measurements would reflect minimum values and water clarity is greatest. This is also the time of year when habitat variables are most limiting so that measurements are most representative of available habitat. Streams are usually wadable during this time, substrates can be readily classified and instream features are easily discernible. Another reason for conducting habitat surveys under base-flow conditions is that during

higher (floods) or lower (drought) flows, some habitat types may change in physical character. For example, under high flows a riffle may become a run and vice versa.

If habitat surveys are to be conducted over a number of days, they should be carried out under similar flow conditions. For instance, surveys should not be conducted after a period of heavy rain that produces a considerable change in discharge or flow. For safety reasons it is often advisable to delay further surveying until flows have subsided.

Photographic Records:

One of the more useful pieces of information collected during habitat surveys can be a site photo. Photos can provide a reference for future surveys as well as capture data that is not recorded on data sheets at the time of the survey. The first step in taking proper photos is accurate labeling. Photos of each habitat type/reach can be recorded on the survey sheet and each photo number clearly recorded (roll# and photo # for prints and photo # for digital). Any descriptive information regarding the photo should also be reported at this time. Other important information to record includes the date and time the photo was taken, the orientation of the camera (downstream/upstream) and any georeference information.

The second important step is the proper labeling of the actual photos as soon as possible. Many digital photos can be properly labeled immediately after they are downloaded on to a computer. Each habitat type/reach photo should be taken so that it captures the entire area. That is, the photographer should stand below the habitat type/reach of interest. If possible, there should be something in the photo for a size reference such as a meter stick or person. These steps are particularly important if photographing locations of proposed culvert crossings. When doing so it is important not to stand on the proposed crossing location when taking the photos. But rather stand below the area of any proposed crossing so that the areas upstream and downstream are clearly visible in the photo.

Parameters Measured:

Depth

Depth refers to the distance from the stream bottom to the water surface. For sampling purposes, rivers are categorized as small, moderate and large based on mean width (e.g., small rivers are < 5 m, moderate 5-20 m and large > 20 m). To measure stream depth in small rivers, it is recommended that at least three equidistant (¼, ½, and ¾) depth measurements be made across a transect. In moderate streams, depth measurements should be taken every 1-2 m along a transect and for large rivers, every 2-4 m, with a minimum of 10 more or less evenly-spaced measurements. To obtain the mean depth, simply sum the individual measurements and divide by the number of measurements plus 1. This is necessary in order to account for the number of segments being sampled (e.g., when taking three measurements there is effectively four segments being sampled: 0 - ¼; ¼ - ½; ½ - ¾; and ¾ - 1).

Cover

Cover is defined as those features within (or in close proximity to) the aquatic environment that provide refuge/protection from predators, competitors or adverse environmental conditions such as high current velocities and bright sunlight. Numerous studies have shown the critical importance of cover as a fish habitat component. What constitutes cover is sometimes ambiguous because cover requirements vary by species, life stage and season, however, it is generally accepted that cover includes substrate, woody debris, aquatic vegetation, overhanging terrestrial vegetation, undercut banks, water turbulence and concealing water depths (Bisson *et al.* 1982; Armantrout 1998).

Typically, woody debris is the most abundant cover in pools, while medium and coarse substrates are the primary cover in riffles. Depth is also an important cover in pools as maximum depth itself is a form of cover from non-diving terrestrial predators. Water turbulence often creates cover where falling water forms bubbles in pools, rapids and cascades thereby preventing a clear view of the water beneath. Substrate, such as rubble or boulders, function as cover by providing overhanging ledges or crevices for hiding.

Large woody debris (LWD) in the form of logs is an important habitat component for a variety of fishes. LWD increases channel stability, creates pools, increases structural complexity, traps gravel for spawning and invertebrate production, holds other organic matter, serves as habitat for aquatic invertebrates, and provides cover for fish. The typical definition for LWD is pieces greater than 1-2 m in length and 10 cm in diameter. Woody debris accumulates naturally in streams and plays an important role in stream mechanics and fish habitat.

Equipment:

-maps(s) 1:50,000 scale or more detailed)
- clinometer
- 50 or 100 m measuring tape (preferable cloth or plastic)
-metre sticks
-stream survey instructions (this SOP or Scruton *et al*. 1992)
-stream survey sheets (see attached)
-waterproof notebook, pencils, clipboard (weatherproof if possible)
-camera and film
-stopwatch and floating ball (hockey ball or orange) or flowmeter
- Rangefinder
- Thermometer
-rain gear
-fly repellant
-waders (hip boots and possibly chest waders for deep stretches)
-first aid kit
-change of clothing, footwear
-emergency rations
-compass and GPS unit
-communication device if necessary

For completeness, pages from Scruton *et al.* (1992) explain the methodology utilized so that data is standardized for each survey.

References:

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Scruton, D.A., T.C. Anderson, C.E. Bourgeois, and J.P. O'Brien. 1992. Small Stream Surveys for Public Habitat Improvement and Enhancement Projects, Can. Manuscr. Rep. Fish. Aquat Sci. No. 2163:v + 49p.

PURPOSE

To establish a standard protocol for all AMEC field personnel to ensure acceptable, consistent and clean sampling techniques are followed during the sampling of macroinvertebrates using artificial substrates, specifically leaf packs, for identification.

RESPONSIBILITIES

A qualified individual, preferably a technician with training in biology or a related field, is responsible for the deployment, monitoring, and retrieval of the artificial substrates for macroinvertebrate sampling. Field personnel shall familiarize themselves with the site in question and the sampling locations, looking for the most efficient and safe entry and exit locations along the water body. They will also make note of river flow and velocity so sampling success will be maximized. That is, substrates should not be deployed during or immediately after a flood event. Field personnel shall also acquire all sampling equipment, sample bottles, decontamination equipment (if necessary), and arrange for sample shipping following the sampling event. They will be responsible for locating the pre-determined length of stream (reach) and for potentially moving the reach location due to site-specific reasons (e.g. no access point or a tree blowdown) Lastly, a sample log describing habitat characteristics, sampling methods and efficiency shall be kept.

Field personnel are also expected to adhere to relevant Health and Safety protocols specific to the Site and to the sampling methodology. Sampling equipment shall be inspected for proper operation prior to the use of the equipment.

PROCEDURE

Macroinvertebrate samples can be collected using different methods, depending on the depth, turbidity, and flow of the water. One such sampling technique involves passive collection through the use of artificial substrates. Macroinvertebrate sampling by use of artificial substrates measures the colonization potential and community assemblage for a location over a set period of time.

The procedures for implementing artificial substrate sampling are described below.

MACROINVERTEBRATE SAMPLING BY USE OF ARTIFICIAL SUBSTRATES

Listed below is key information on macroinvertebrate sampling by use of artificial substrates.

- Rocks are used to incur significant weight to individual rock baskets or mesh bags for the artificial substrate setup. It is important to assess the degree of effort to transfer heavy artificial substrates from one location to another (boats or floating rafts/sleds are recommended).
- Vandalism of artificial substrates is not uncommon. Colonization time requires 6 – 8 weeks so it is important that the artificial substrates are “invisible” from above water yet “visible” upon retrieval (e.g. with the use of flags or markers on nearby trees).

Artificial substrates: Equipment

- Charbroil baskets or polypropylene mesh bags
- Mesh bags
- Artificial substrate (natural rocks, road grade stone, leaves, etc.)
- Polypropylene twine or string
- ¼” or ⅜” polypropylene rope (3-ply)

- Knife or LeatherMan (to cut twine, string or rope)
- Butane lighter (used to "seal" ends of polypropylene twine, string or rope)
- Cable ties (assorted sizes)
- Electrical tape
- Duct tape
- Wash sieve bucket with 0.5 mm mesh
- Filter paper or waterproof paper (e.g. "Rite-in-the-Rain")
- No. 2 Pencil
- Hip boots and/or waders
- Prelabelled sample jars or Ziploc bags
- Soft-bristled brush (e.g. plastic grout brush)
- Preservative (typically 80% ethanol/0.01% rose Bengal or 10% buffered formalin)

Artificial substrates: Planning/Mobilization Phase

a) Identify the length of reach needed to be sampled. A minimum length of 100 feet is typically recommended for an "individual" reach.
b) Identify the number of reaches needed to be sampled.
c) Once (a) and (b) are determined, identify the location of each reach to be sampled. When doing this, first make sure Health & Safety requirements are met (e.g. flow velocity/discharge will not overwhelm samplers). If sampling is biased to determine a "trend" (e.g. decrease in tissue concentration with increase in distance from a site-related source), it is very important to make sure there is ample distance in between individual reaches.
d) Based on (a) through (c) above, rent or buy Charbroil baskets or mesh bags through a reputable vendor (e.g. Wildco, Memphis Net & Twine, Forestry Suppliers; or, because baskets or mesh bags may be ordered in significant quantities, possibly "Google" for best price)

Artificial substrates: Field Sampling Procedure

Collecting

a) Fill all Charbroil baskets or mesh bags with the same approximate volume of artificial substrate. If using Charbroil baskets, use cable ties as "hinges" and also to cable tie the trap door shut (these will be cut upon retrieval). If using a mesh bag, simply pull the drawstring tight and tie an overhand knot to keep bag closed (cable ties can also be woven through the mesh to ensure bag does not open in current).
b) Deploy substrate samplers in duplicate or triplicate at each station. The number of replicates depends on the data needed and the stream characteristics. Ensure that each substrate is secured either to the bottom substrate between large rocks, with rebar driven into sediment, or tied to an object on land (e.g. tree trunk or branch).
c) Make sure substrates are not too visible from above water. Also make sure that on-shore flagging tape is used identify the general location of each station.

Field processing

d) After a predetermined period of colonization, identify location of each substrate.
e) To retrieve, stand downstream of each substrate with a wash sieve bucket, immediately downstream of the substrate. Pick up the substrate while, at the same time, keeping the wash sieve bucket "in line" with the substrate so that the sieve will catch any organisms that drift away from the substrate material.
f) If the field personnel are processing the samples in the field, gently place the substrate in the wash bucket and move to the bank of the river to process the substrate material.

g) Gently brush organisms from rocks into wash bucket and discard rocks. Once all gross material is processed, thoroughly wash all debris and organisms in the sieve bucket in the stream.
h) If the substrate samples are to be cleaned and identified in a taxonomy laboratory, collect samples, making sure the entire leaf pack is collected and preserved without any separation.

MACROINVERTEBRATE HANDLING PROCEDURE

If the samples are processed in the field, preserve whatever material that is retained on the sieve bucket screen in a sample jar or Ziploc bag ((include leaves, unless you are sure all organisms have been brushed or washed from the leaf surface). Include an “internal label”, written in pencil on a piece of filter paper or waterproof paper, inside the preservative jar or bag. If shipping preserved sample(s) in Ziploc bags, make sure most of the air is removed. Then double-bag, and ship the Ziploc bag upright in the cooler.

If the samples are processed in a taxonomy laboratory, place the entire leaf pack in a sample jar or Ziploc bag. Then fill out the appropriate Chain-of-Custody forms. Remove AMEC copy of the COC form. Place laboratory copy of COC form in a one gallon Ziploc bag and seal. Place COC form in cooler. Seal cooler with COC Seal and secure both the cooler and the COC Seal with clear packing tape. Ship cooler overnight to appropriate laboratory address. If sampling occurs on a Friday, make sure to mark “Saturday Delivery” on packing slip and notify lab to ensure that a lab technician will be there to receive the cooler.

PURPOSE

To establish a standard protocol for all AMEC field personnel to ensure acceptable, consistent and clean sampling techniques are followed during the sampling of fish and/or crayfish for chemical analysis of the biological tissue.

RESPONSIBILITIES

A qualified individual with training in biology or a related field is responsible for fish sampling and oversight. Field personnel shall familiarize themselves with the site in question and the sampling locations, looking for the most efficient and safe entry and exit locations along the water body. Field personnel shall also acquire all sampling equipment, sample bottles, decontamination equipment (if necessary), and arrange for sample shipping following the sampling event. Lastly, a sample log describing habitat characteristics, sampling methods and efficiency shall be kept.

The sampling equipment shall be inspected for proper operation prior to use.

PROCEDURE

Fish samples are collected using a variety of methods and equipment, depending on the depth, turbidity and/or flow of the water, and the type of fish being sampled (larger sport or game fish versus smaller forage fish). Fish are collected either actively using trawls, umbrella nets, cast nets, dip nets, seines, electroshocking units, or rod & reel methods; or passively using minnow traps, trap nets, hoop nets, set lines, or gill nets. Crayfish have been most successfully caught using minnow traps or dip nets.

States typically require Scientific Collection Permits in which the methods for collection are identified. Upon calling the State regulatory agency for such information, it helps to talk to a State biologist who is familiar with the area as to whether seining is the best SOP for the type of fish that require capture.

The methods that AMEC typically employs are described below.

SEINING

Listed below is key information on implementing seining for fish and/or crayfish.

- Nylon seines are recommended because they are lighter and more easily maintained.
- Seines and nets are difficult to maneuver in water w/o “handles”. It is therefore recommended that each seine be tied onto long wooden dowels or broomsticks, which makes them easier to pay out and haul in.
- Seining is not effective in deep water because the fish can escape over floats or under lead lines.
- Seining is not effective in areas that have snags and sunken debris.
- Although results of seining are sometimes expressed as number of fish per unit seined, this type of sampling is generally not a quantitative method.
- This method is most efficient with three people: two in the water to handle paying out either side of the seine, and one on shore to assist with hauling in the seine, handling fish, and processing samples.
- “Straight” or “minnow” seines (without bags) are generally used for shallow water, whereas “haul” or “bag seines are generally used for deeper water (e.g. ponds or lakes).

Seining: Equipment

- Proper size, depth and width seines (see "Planning/Mobilization Phase" below)
- ¾" or larger diameter dowels, with holes bored in both ends for tying off seines (broomsticks from Home Depot or Lowes are fine)
- Polypropylene twine or string
- ¼" or ⅜" polypropylene rope (3-ply)
- Knife or Leatherman (to cut twine, string or rope)
- Butane lighter (used to "seal" ends of polypropylene twine, string or rope)
- Cable ties (assorted sizes)
- Electrical tape
- Duct tape
- Iron rebar (if desired for in-stream staking)
- Small sledge hammer
- Hacksaw
- Sample Cooler (because of lability, dry ice is recommended when sampling fish)
- Secondary container for dry ice inside sample cooler to keep dry ice longer, and to protect samples

Seining: Planning/Mobilization Phase

a) Identify the length of reach needed to be seined. The minimum length of an "individual" reach typically recommended is 100 feet for quantitative assessments.
b) Identify the number of reaches needed to be sampled.
c) Once (a) and (b) are determined, identify the location of each reach to be sampled. When doing this, first make sure Health & Safety requirements are met (e.g. flow velocity/discharge will not overwhelm samplers). Also bear in mind that fish are migratory and that sampling of similar habitat(s) may yield similar types/species of fish. It is important to make sure there is ample distance in between individual reaches if any quantitative comparisons will be made between reaches.
d) Before ordering the seine, identify the "average" river width for each reach. Ideally, a site "walk-over" will give an indication of this, as well as the ideal locations of access roads/areas to conduct the seining activity. If a walkover cannot be performed, widths can be determined using aerial photographs and GIS scaling.
e) Based on (a) through (d) above, order the seines required from a reputable fishing or netting supply vendor (e.g. Memphis Net & Twine; Forestry Suppliers; Wildco). Ensure that i) each seine is long enough to span the width of river/stream at each reach (see "d") and ii) the minimum mesh size ordered is small enough to catch the smallest fish identified in the Quality Assurance Project Plan (QAPP).
f) Attach handles to each end of the seine

Seining: Field Sampling Procedure

a) If using a two-seine methodology: At each reach, secure the "downstream" seine first. Tying off the corners of the seine to a tree or branch on the bank

of the river/stream can do this. Metal rebar can also be driven into the river/stream substrate and/or through the lower corner of the seine.

b) Move (via land) to the "upstream" portion of the reach and, with a crew member, carefully extend the second seine across the width of the upper reach. Then carefully "walk" the upper seine down to the lower seine. Care must be taken to avoid large rocks or snags on the bottom of the river/stream as the upstream seine is moved toward the downstream seine.
c) Once the samplers have arrived at the downstream seine, the upper seine is pulled on to shore in a "U-shaped" fashion, making sure the lower rope of the seine is pulled in faster than the upper rope to prevent fish from swimming underneath the lead line.
d) Select fish that are required for samples (according to the QAPP and/or work plan) and then release any remaining live fish back into the water body.

e) If using a single-seine transect methodology: Seine the reach by using "sweeps" of the creek with a seine net in a brisk paced downstream to upstream format. After the sweep, the ends of the seine are brought together and pulled up on one side of the creek bank to prevent any fish from escaping. Each of the sweeps is considered a "seine". Several seines may be needed per reach to either a) obtain enough fish for analysis or b) to sweep the entire length of reach, if quantifying the population of fish per reach.

ELECTROSHOCKING

This method requires at least two people in the water and is most efficient with three people (one on land to receive fish and/or process samples). ALL persons conducting the field sampling procedure(s) should read through the Instruction Manual for any Backpack or Boat-mounted electroshocker.

Electroshocking: Equipment

- Backpack Electroshocker (ask rental vendor for all recommended equipment)
- Elbow-length rubber gloves
- Capture Nets (w/ insulated poles)
- 12V Battery Recharger (may be included with electroshocker)
- Polaroid Sunglasses (required, non-polarized will not work!)
- Hipboots and/or waders
- Sample Cooler (because fresh fish deteriorates rapidly, dry ice is recommended for rapidly preserving and/or shipping fish)

Electroshocking: Planning/Mobilization Phase

a) Identify the length of reach needed to be electroshocked. The minimum length of "individual" reach typically recommended is 100 feet.
b) Identify the number of reaches needed to be sampled.
c) Once (a) and (b) are determined, identify the location of each reach to be sampled. When doing this, first make sure Health & Safety requirements are met (e.g. flow velocity/discharge will not overwhelm samplers). Also bear in mind that fish are migratory and that sampling of similar habitat(s) may yield similar types/species of fish.

d) Before renting the electroshocker, identify whether the water quality in the area to be sampled may affect the efficiency of the electroshocker. For example, brackish water requires much more power (amps) than freshwater, even though the voltage drops may be identical. Similarly, some electrolytes, in the form of salt blocks, may be required for unusually soft waters.
e) Based on (a) through (d) above, rent the electroshocker from a reputable rental company, or a vendor that both rents and sells electroshockers (e.g. Smith-Root). Before sampling, ensure,that the electroshocker is working according to the Instruction Manual supplied by the renter/vendor.

Electroshocking: Field Sampling Procedure

a) Optional: secure a seine across the "upstream" limit of the reach being sampled (to block fish that may be startled upstream by wading/shocking activity).
b) Crew member "A" who will not be wearing the electroshocker will assist crew member "B" in putting on the Backpack Electroshocker.
c) Once "A" and "B" are in the water, take a few minutes to ensure that hipboots or waders are not leaking. If leaks are observed, that crew member must exit the stream to prevent electrical shock and either fix the leak or obtain new waders before continuing with the shocking.
d) Depending on the yield or success rate, crew member "A" will adjust the voltage, frequency/amplitude and amperage according to the Backpack Electroshocker Instruction Manual.
e) Both "A" and "B" should start at the "downstream" segment of each reach. "A" will attempt to "net" any fish that may float to the surface after being stunned by the current provided by the submerged electrodes ("triggered" by "B"). Some electroshockers, however, may include a net at the end of one of the two electrodes).
f) Crew member "B" generally will walk slightly ahead of "A" and trigger electric currents while walking. Undercut banks, snags, pools and other optimal fish habitat are the best places to shock.
g) If unsuccessful when arriving at the upstream limit of the reach, return to the downstream reach boundary via land. Wait about 15 minutes and repeat the downstream to upstream electroshocking procedure (c – f, above).
h) Select fish that are required for samples (according to the QAPP and/or work plan) and then release any remaining live fish back into the water body. Most stunned fish, if kept in site water, will return to normal after about 2-10 minutes.

PASSIVE TRAPPING/NETTING

Many states have outlawed trot lines and/or gill nets. It is therefore necessary to disclose this method on any permit applications if the method may be used. Experimental gill nets, which have five different mesh sizes, are generally the most "versatile" in terms of the successful sampling of all types of fish.

Gill netting requires at least two people in the water and may be most efficient with three people (one on land to receive fish and/or process samples).

Netting: Equipment

- Passive trapping device (e.g. gill net, hoop net, fyke net, trot line, minnow net)
- Bait (Note: asking local bait vendor/store what to use to attract fish or crayfish may save many hours in the field!)
- Passive traps or nets
- Polypropylene twine or string
- ¼" or ⅜" polypropylene rope (3-ply)
- Knife or LeatherMan (to cut twine, string or rope)
- Butane lighter (used to "seal" ends of polypropylene twine, string or rope)
- Cable ties (assorted sizes)
- Electrical tape
- Duct tape
- Hip boots and/or waders
- Sample Cooler (because fresh fish deteriorates rapidly, dry ice is recommended for rapidly preserving and/or shipping fish)

Netting: Planning/Mobilization Phase

a) Identify the length of reach needed to be sampled. The minimum length of "individual" reach typically recommended is 100 feet.
b) Identify the number of reaches needed to be sampled.
c) Once (a) and (b) are determined, identify the location of each reach to be sampled. When doing this, first make sure Health & Safety requirements are met (e.g. flow velocity/discharge will not overwhelm samplers). Also bear in mind that fish are migratory and that sampling of similar habitat(s) may yield similar types/species of fish. Additionally, if sampling if biased to determine a "trend" (e.g. decrease in fish tissue concentration with increase in distance from a site-related source), it is very important to make sure there is ample distance in between individual reaches.
d) Before buying or renting passive traps, identify the whether the method is permitted in the state/city/water body to be sampled. For example, some southern states have outlawed trot lines and gill nets.
e) If ordering gill nets, ensure that the "top" line floats (or has floats attached) and that the "bottom" line sinks (or has sinkers attached). Both floating and sinking rope is now available and therefore deploying of gill nets containing these types of rope is much easier.
f) Based on (a) through (e) above, rent or buy passive traps and/or nets through a reputable vendor (e.g. Memphis Net & Twine, Forestry Suppliers; or, because traps may be ordered in significant quantities, may want to "Google" for best price)

Netting: Field Sampling Procedure

a) If gill netting, deploy the net in an area that would logically entrain fish. For example, across the inlet or outlet of a pond or lake. If the water is deeper

than the gill net is tall, attach a visible float or lobster buoy to ensure that the net can be retrieved the following morning. Do not leave gill nets unattended for more than 12 hours (if fish die, they will begin to decompose).

b) If using a trap, bait the trap and, if necessary, use a weight to ensure it stays on the bottom (e.g. stones placed in a minnow trap). If trapping in flowing water, tie (if possible) a rope to the trap and tie the other end to an object on shore (e.g. tree, branch, culvert, etc.). If trapping in standing water, ensure that the trap is upright, or in the "correct" position, when placing on the bottom of the water body.
c) If using a hoop or fyke net in flowing water, ensure that the opening of the net faces upstream. If the net has "wings", ensure that each side wing is staked in the correct position to keep the net open and/or operable.
d) If using a trot line, ensure all hooks are properly baited, that both ends of the line are secure, and that very little slack remains in the line after deployment.
e) After a predetermined trapping period (typically overnight), return and retrieve the trap or traps of interest.
f) Select fish or organisms that are required for samples (according to the QAPP and/or work plan) and, if any remain alive and are not needed, toss live back into the water body. Select fish that are required for samples (according to the QAPP and/or work plan) and, if any remain alive and are not damaged, release them back into the water body.

OTHER METHODS

Several other methods may be used for fish collection. Two methods typically used as supplemental methods are discussed below.

A minnow trap is a black rectangular steel mesh trap approximately 18 inches long, 8 inches high, and 8 inches wide with a sliding mesh door and a funnel at one end. When it is placed in the water, it will be weighted down with rocks if needed, and a float will be tied to the handle so that it is easily found and retrieved the following morning. It will be kept in place for approximately 24 hours at each sampling location. The minnow trap is baited with bread or some other suitable fish bait and placed in the creek in the area of best available fish habitat in the vicinity of the sample location (e.g., aquatic vegetation, backwaters, submerged debris).

Rod and reel sampling (baited or unbaited) is an active method that can be used as a supplement to other methods. This method is meant to target larger fish when sampling fish to evaluate human health consumption. Sampler should have knowledge of what types of bait and tackle would make the most efficient sampling at a given location.

FISH HANDLING PROCEDURE

When fish are collected, all fish are identified, enumerated, if necessary, and wrapped in aluminum foil. The foil-wrapped fish will be put inside a Ziploc® bag and labeled, then placed in a cooler on ice for transport to the analytical laboratory.

Once field sampling is complete, fill out the appropriate Chain-of-Custody forms. Remove AMEC copy of the COC form. Place laboratory copy of COC form in a one gallon Ziploc® bag and seal. Place COC form in cooler. Seal cooler with COC Seal and secure both the cooler and the COC Seal with clear packing tape. Ship cooler overnight to appropriate laboratory address. If sampling occurs on a Friday, make sure to mark "Saturday Delivery" on packing slip and notify lab to ensure that a lab technician will be there to receive the cooler.

ARCADIS

Attachment B

Laboratory Standard Operating Procedures

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Title: Extraction and Cleanup of Organic Compounds from Waters and Solids

Method(s): SW846 3500 Series, 3600 Series, 8151A and EPA 600 Series Methods

Approvals (Signature/Date):

Brian A. Pino 10/17/07 Brain Pino — Date Technical Manager	Steve Jackson 10/16/07 Steve Jackson — Date Health & Safety Manager / Coordinator
Nasreen K. DeRubeis 10/16/07 Nasreen DeRubeis — Date Quality Assurance Manager	Larry Matko 10/16/07 Larry Matko — Date Laboratory Director

This SOP was previously identified as SOP No. PITT-OP-0001, Rev. 9.

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Title: Semivolatile Organic Analysis by GCMS

Method(s): 8270C and 625

Approvals (Signature/Date):

Sharon Bacha Technical Manager	11/16/07 Date	Steve Jackson Health & Safety Coordinator	11/16/07 Date
Nasreen K. DeRubeis Quality Assurance Manager	11/16/07 Date	Larry Matko Laboratory Director	11/16/07 Date

This SOP was previously identified as SOP No. PITT-MS-0001, Rev. 7

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Title: Acid Digestion of Soils

Method: SW846 Method 3050B

Approvals (Signature/Date):

	10/5/07		9/19/07
William Reinheimer	Date	Larry Matko	Date
Technical Manager		Acting Health & Safety Manager	
	9/19/07		9/19/07
Nasreen DeRubeis	Date	Larry Matko	Date
Quality Assurance Manager		Laboratory Director	

This SOP was previously identified as SOP No. PITT-IP-0002, Rev.6.

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Title: ACID DIGESTION OF AQUEOUS SAMPLES

Method(s): SW846 Methods 3005A and 3010A and EPA Methods 200.7 and 200.8

Approvals (Signature/Date):

	10/12/07		10/15/07
William Reinheimer Technical Manager	Date	Steve Jackson Health & Safety Manager / Coordinator	Date
	10/12/07		10/12/07
Nasreen DeRubeis Quality Assurance Manager	Date	Larry Matko Laboratory Director	Date

This SOP was previously identified as SOP No. PITT-IP-0003, Rev. 7.

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Title: Inductively Coupled Plasma-Atomic Emission Spectroscopy, Spectrometric Method for Trace Element Analyses

Method(s): SW-846 Method 6010B and EPA Method 200.7

Approvals (Signature/Date):

	1/29/08		1/29/08
William Reinheimer Technical Manager	Date	Steve Jackson Health & Safety Manager / Coordinator	Date
Nasreen K. DeRubeis	1/29/08	Nasreen K. DeRubeis	1/30/08
Nasreen DeRubeis Quality Assurance Manager	Date	for Larry Matko Laboratory Director	Date

This SOP was previously identified as SOP No. PITT-MT-0001, Rev. 8.

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Title: Total Organic Carbon Analysis for Solid Matrices

Method: Walkley-Black

Approvals (Signature/Date):

Michael Wesoloski — 10/18/07 Michael Wesoloski — Date Technical Manager	Steve Jackson — 10/18/07 Steve Jackson — Date Health & Safety Manager / Coordinator
Nasreen K. DeRubeis — 10/17/07 Nasreen K. DeRubeis — Date Quality Assurance Manager	Larry Matko — 10/18/07 Larry Matko — Date Laboratory Director

This SOP was previously identified as SOP No. PITT-WC-0058, Rev. 3.

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TESTAMERICA KNOXVILLE

PROCEDURE SUMMARY

TITLE: Solid-Phase Microextraction and Analysis of Dissolved Parent and Alkyl Polycyclic Aromatic Hydrocarbons in Sediment Pore Water

(SUPERSEDES: None)

The Sections and Section numbers in this summary will not directly correspond to the controlled, complete standard operating procedure.

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Title: GAS CHROMATOGRAPHIC ANALYSIS BASED ON METHODS 8000B, 8021B, 8081A, 8082, 8151A, 8015B, AND 615

Approvals (Signature/Date):

Angela McGregor	1/18/08		1-18-08
Technology Specialist	Date	Health & Safety Coordinator	Date
Dorothy J. Gleason	1/18/08		1/17/08
Quality Assurance Manager	Date	Laboratory Director	Date
Mark Burns	1/18/08		
Technical Director	Date		

This SOP was previously identified as SOP CORP-GC-0001NC, Rev 5.8, dated 02/06/06

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Title: Phenolics (Automated)

Method(s): SW846 Method 9065 and 9066 and EPA Methods 420.1 and 420.4

Approvals (Signature/Date):

Michael Wesoloski — 10/29/07 — Date Technical Manager	Steve Jackson — 10/3/07 — Date Health & Safety Manager / Coordinator
Nasreen K. DeRubeis — 10/1/07 — Date Quality Assurance Manager	Larry Matko — 10/2/07 — Date Laboratory Director

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Title: Determination of Solids in Waters and Wastes

Method(s): EPA 160.1, 160.2, 160.3, 160.4, 160.5 and Standard Methods 2540 B through F

Approvals (Signature/Date):

Michael Wesoloski	1/29/08		1/29/08
Michael Wesoloski Technical Manager	Date	Steve Jackson Health & Safety Manager / Coordinator	Date
Nasreen K. DeRubeis	1/29/08	Nasreen K. DeRubeis	1/30/08
Nasreen DeRubeis Quality Assurance Manager	Date	for Larry Matko Laboratory Director	Date

This SOP was previously identified as SOP No. C-WC-0002, Rev. 4.

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STL

Implementation Date: April 30, 2007

STL PITTSBURGH STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF SOLIDS IN WATERS AND WASTES
(METHODS 160.1, 160.2, 160.3, 160.4, 160.~~0~~5, SM 2540 B-F)
(**SUPERSEDES:** C-WC-0002, REV. 4, 10/12/05)

Prepared By: _____

Reviewed By: _____
Technical Specialist

Approved By: _____
Quality Assurance Manager

Approved By: _____
Environmental Health and Safety Coordinator

Approved By: _____
Laboratory Director

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Vista Analytical Laboratory
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Analytical Laboratory

SOP 24	Revision: 2	Supersedes: Rev1
POLYCHLORINATED DIBENZO DIOXIN/FURANS BY USEPA METHOD 8290		
Analyst Review: 3/14/07		
Management:		
Quality Assurance:		
Effective Date: 14 March 2007		

Revision	Description of Revision
0	Renumbered and reformatted.
1	[illegible]
2	[illegible] Added Penta-HS to Table 1. Update company name.

Vista Analytical Laboratory
El Dorado Hills, CA 95762

Laboratory Procedures

Sample Receipt

Samples will be checked against the chain of custody and logged in the sample log book upon receipt. All samples will also be checked for proper preservation and re-preserved in 70% ethanol if found to be less than adequately preserved. Any containers received leaking or broken will be replaced, and also noted in the sample log book. A copy of the chain of custody will be retained by Lotic.

Sample Sorting

Samples will be poured through a 500 micron sieve and rinsed with water to remove as much sediment as possible. Large objects (sticks, leaves, pinecones, rocks etc.) will be rinsed in the sieve and set aside. Samples will be sorted using a stereo microscope. Sorted organisms are stored in a 4-dram vial with neoprene stopper and preserved in 70% ETOH. Sample labels are placed inside and outside the vial with all pertinent sample information. Sorted sample material is disposed of unless requested.

In the event that the samples contain large numbers of organisms (>1000) subsampling may be performed. This involves placing the sieved samples in a gridded tray and randomly sorting grids until a 300 organism subsample is collected (Methods follow Rapid Bioassessment Protocols for Use in Wadeable Streams and Rivers, US EPA 841-B-99-002). The unsorted sample portion is retained for a period of 5 years.

Organism Identification

Macroinvertebrates will be identified using state-of-the-art stereo microscopes and the most recently published taxonomic references. Chironomidae are cleared by immersion in a 10% solution of room-temperature KOH for 24-48 hours. Once cleared the specimens are neutralized in 5% glacial acetic acid, rinsed in distilled water and slide mounted in a solution of CMCP-10. Once the mountant has dried, the coverslips are ringed with clear nail polish. Oligochaeta are mounted in polyvinyl lactophenol. All slide material is identified with a compound microscope. Slides are labeled with the site location, date and sample number.

Organisms will be identified to genus/species unless the condition of the organism or lack of workable keys prevent it. All identified organisms will be retained in the original sample containers, with the exception of the voucher collection.

Sample Disposal

Sample remnants will be retained by Lotic for 5 years or until notified, at which time they will be safely disposed of.

Selected Taxonomic References

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10 TO 21-DAY STATIC FRESHWATER SEDIMENT TOXICITY TESTS WITH THE MIDGE LARVAE (*CHIRONOMUS TENTANS*)

I. PURPOSE

This method is designed to measure the toxicity of freshwater sediments to the midge larvae *Chironomus tentans* using survival and growth as the endpoints. Standard test durations range from 10 to 21 days and are project dependent.

II. EQUIPMENT

- Proven source of clean freshwater water (e.g., Culligan filtered water)
- Environmental chamber maintained at 20 ± 1 degrees celsius (°C) or 23 ± 1°C
- Cold room maintained at 4°C to hold sediment samples prior to test initiation
- Thermometer, pH meter, dissolved oxygen (DO) meter, conductivity meter, dissecting light microscope, and analytical balance
- 1-liter (L) glass jars with lids (or equivalent) to serve as exposure chambers (5 jars per test site, reference, and control); with holes in lids to permit aeration
- 0.5-millimeter (mm) and 1.0-mm Nitex mesh screens, transfer pipets, and glass holding bowls
- Aeration source, 1.0-milliliter (ml) pipets, and airline tubing
- Muffle furnace for ash free dry weight (AFDW) if project-applicable
- Test, control, and reference sediments
- 30-ml disposable polystyrene cups for holding animals; freshwater squirt bottle
- Safety equipment - lab coats, eye protection, gloves, and respirator as required

III. TEST PROCEDURE

A. Pre-test Set-up

1. *Chironomus* are purchased from a reliable, experienced dealer and shipped to the lab via next day delivery service. Upon arrival and test initiation, *Chironomus* are 2nd to 3rd instar (approximately 10-day old larvae). Organisms are held in plastic bowls and allowed to acclimate to laboratory conditions for at least six hours prior to test initiation.

Approved By:	
Laboratory Manager (Date)	QA Supervisor (Date)

2. During the holding period, the bowls are aerated and animals are acclimated to lab conditions by slowly replacing culture water with laboratory water. Water is

replaced by adding a slow drip using lab water. During acclimation temperature changes should not exceed 2°C per 24-hr period.

3. Samples are logged in upon receipt. See procedural SOP for details on sediment sample check-in procedures.

4. Interstitial porewater is collected from each sample for the measurement of ammonia, pH, and conductivity. Interstitial water is collected by centrifuging a subsample of whole sediment at 3000 rpm for 15 min. Interstitial water may also be collected from overlying water in sediment collection containers. During shipment, sediments quickly settle, often leaving a layer of interstitial water on the sediment surface.

5. Sediment samples are sieved through a 0.5-mm screen to filter out any resident organisms and/or debris. The sieved sediment is placed in a clean, labeled, plastic bag, and stored at 4°C until tests are initiated.

6. A randomization key is made for all concurrently tested samples using Microsoft Excel

<u>24 Hrs Prior to Test Initiation</u>

7. Label test chambers according to the randomization sheet and add the appropriate site sediment to the labeled jars to a depth of 2 centimeters (cm).

8. Prepare an additional test chamber for each test site. This surrogate will be used for water quality measurements during the test period.

9. Fill jars with 900 ml laboratory test water.

10. Place jars in the 20 or 23°C environmental chamber to settle and equilibrate overnight.

11. Aerate test chambers using 1-ml glass pipettes placed through a hole in lid and at mid-depth in the water column with special care taken to prevent disturbing the sediment. Aeration should be performed at a rate of 2-3 bubbles per second.

12. Adjust light cycle in environmental chamber to a 16:8 hour light:dark cycle.

B. Test Initiation - Day 0

1. Measure and record water quality parameters (DO, conductivity, temperature, and pH) in each surrogate test chamber. Remove a subsample of overlying water for ammonia analysis from each site and preserve with 1 ml of 10% sulfuric acid.

2. Count 5 animals into a 30-ml polystyrene plastic cup containing clean laboratory water at the appropriate test temperature. All animals should be of similar size. Place 5 randomly chosen cups aside to obtain a mean time zero weight.

3. A second count is made by a different employee to verify that there are 5 animals in each cup and in healthy condition.

4. Randomly distribute 10 test animals (two cups) into each test chamber by gently rinsing the contents of each cup into a test chamber with a squirt bottle containing Culligan water.

5. All test chambers are inspected within two hours of initiation to ensure that no *Chironomu*s are trapped in the surface tension of the water. Any floating animal observed is replaced with a new animal.

C. Daily Monitoring and Feeding Regime

1. Measure and record pH, DO, conductivity, and temperature daily in surrogate chambers.

2. Observe all chambers and correct obvious problems (e.g., aeration rate) as required.

3. Adjust aeration as needed to maintain the DO above 4.0 mg/L.

4. Feed test chambers every 48-hours with 1 ml of Tetramin® solution per test chamber. Food consists of 2g TetraMin® blended into 100 ml Culligan water. If there is an accumulation of excess food on the sediment surface, suspend feeding to avoid fungal growth and degraded water quality in the test chambers.

D. Test Termination - Day 10 to 21 (project dependent)

1. Measure and record water quality (DO), pH, conductivity, and temperature) in surrogate test chambers. Remove a subsample for ammonia analysis from each site and preserve with 1 ml of 10% sulfuric acid.

2. Gently suspend sediment in the test chambers and pour onto 0.5-mm cylindrical screen. Rinse away remaining sediment using laboratory water within 2°C of the test temperature.

3. Count and record the number of surviving *Chironomus* making note of any dead animals or parts of animals found.

4. Collect and rinse surviving *Chironomus* with deionized water. Using forceps, gently place on an appropriately labeled and tared aluminum pan.

5. Dry in an oven at 60°C for 24 hours.

6. Remove animals from oven. Weigh to the nearest 0.0001g and record on the data sheet.

7. If requested, place dried and weighed organisms in a muffle furnace at 550°C for 2 hours to obtain ash-free dry weight.

E. Reference Toxicant Testing

1. A 96-hour reference toxicant is run with the same batch of animals with sediment tests as a quality assurance procedure to assess the health of the test organisms and soundness and consistency of procedures. Copper chloride is used as a reference toxicant. The testing concentrations for the reference toxicant are 0 (Control), 100, 200, 400, 800, and 1600 µg/L.

2. The LC_{50} for survival is calculated and should be within two standard deviations of the historical control chart mean for AMEC.

IV. TEST ACCEPTABILITY CRITERIA

Mean control sediment survival at test termination must be 80 percent or greater. There is no acceptability criterion for the weight endpoint as it is not specified in the protocol.

V. HEALTH AND SAFETY

Health and safety precautions and applicable regulations should be considered at all times. Gloves must always be worn when handling test sediments.

VI. PERSONNEL

Only qualified technicians who have been properly trained and can demonstrate competency with these techniques are permitted to conduct this test.

VII. QUALITY ASSURANCE REQUIREMENTS

Quality assurance practices encompass all aspects of testing including the collection, handling, and preparation of test organisms, samples, and dilution waters. Proper record keeping is required and concentrated efforts are made for complete documentation on a real-time basis.

VIII. REFERENCES

"Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S.-Testing Manual." Environmental Protection Agency Office of Water/Department of the Army, US Army Corps of Engineers. EPA-823-B-98-004,1998.

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"Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates (Second Edition)." EPA 600/R-99/064, 2000.

I. PURPOSE

This method is designed to measure the toxicity of freshwater sediments to the amphipod *Hyalella azteca* using survival and growth as endpoints.

II. EQUIPMENT

- Proven source of clean freshwater water (e.g., Culligan filtered water)
- Environmental chamber maintained at 20 ± 1 degrees Celsius (°C) or 23 ± 1°C
- Cold room maintained at 4°C to hold sediment samples prior to test initiation
- Thermometer, pH meter, dissolved oxygen (DO) meter, conductivity meter, dissecting light microscope, and analytical balance
- 1-liter (L) glass jars with lids (or equivalent) to serve as exposure chambers (5 jars per test site, reference, and control); with holes in lids to permit aeration
- 0.5-millimeter (mm) and 1.0-mm Nitex mesh screens, transfer pipets, and glass holding bowls
- Aeration source, 1.0-milliliter (ml) pipets, and airline tubing
- Test, control, and reference sediments
- 30-ml disposable polystyrene cups for holding animals; freshwater squirt bottle
- Safety equipment - lab coats, eye protection, gloves, and respirator as required

III. TEST PROCEDURE

A. Pre-test Set-up

1. *Hyalella*, 7 to 14 days in age, are purchased from a reliable, experienced dealer and shipped to the lab via next day delivery service. All animals in a single batch shall be within a two-day age range. Organisms are held in plastic bowls and allowed to acclimate to laboratory conditions for at least six hours prior to test initiation.
2. During the holding period, the bowls are aerated and animals are acclimated to lab conditions by slowly replacing culture water with laboratory water. Water is replaced by adding a slow drip using lab water. During acclimation temperature changes should not exceed 2°C per 24-hr period.

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Laboratory Manager (Date) QA Supervisor (Date)

4. Samples are logged in upon receipt. See procedural SOP for details on sediment sample check-in procedures.

5. Interstitial porewater is collected from each sample for the measurement of ammonia, pH, and conductivity. Interstitial water is collected by centrifuging a subsample of whole sediment at 3000 rpm for 15 min. Interstitial water may also be collected from overlying water in sediment collection containers. During shipment, sediments quickly settle, often leaving a layer of interstitial water on the sediment surface.

6. Sediment samples are sieved through a 0.5-mm screen to filter out any resident organisms and/or debris. The sieved sediment is placed in a clean, labeled, plastic bag, and stored at 4°C until tests are initiated.

6. A randomization key is made for all concurrently tested samples using Microsoft Excel

<u>24 Hrs Prior to Test Initiation</u>

7. Label test chambers according to the randomization key and add the appropriate site sediment to the labeled jars to a depth of 2-centimeters (cm).

8. Prepare an additional test chamber for each test site. This surrogate will be used for water quality measurements during the test period.

9. Fill jars with 900 ml laboratory test water.

10. Place jars in the 20 or 23°C environmental chamber to settle and equilibrate overnight.

11. Aerate test chambers using 1-ml glass pipettes placed through a hole in lid and at mid-depth in the water column with special care taken to prevent disturbing the sediment. Aeration should be performed at a rate of 2-3 bubbles per second.

12. Adjust light cycle in environmental chamber to a 16:8 hour light:dark cycle.

B. Test Initiation - Day 0

1. Measure and record water quality parameters (DO, conductivity, temperature, and pH) in each surrogate test chamber. Remove a subsample of overlying water for ammonia analysis from each site and preserve with 1 ml of 10% sulfuric acid.

2. Count 10 animals into a 30-ml polystyrene plastic cup containing clean laboratory water at the appropriate test temperature. All animals should be of similar size. Place 5 randomly chosen cups aside to obtain a mean time zero weight.

3. A second count is made by a different employee to verify that there are 10 animals in each cup and in healthy condition.

4. Randomly distribute 20 test animals (two cups) into each test chamber by gently rinsing the contents of each cup into a test chamber with a squirt bottle containing Culligan filtered water.

5. All test chambers are inspected within two hours of initiation to ensure that no amphipods are trapped in the surface tension of the water. Any floating animal observed is replaced with new animal.

C. Daily Monitoring and Feeding Regime - Days 1 to 10

1. Measure and record pH, DO, conductivity, and temperature daily in surrogate chambers.

2. Observe all chambers and correct obvious problems (e.g., aeration rate) as required.

3. Adjust aeration as needed to maintain the DO above 4.0 mg/L.

4. Feed test chambers every 48-hours with 1 ml of Tetramin® solution per test chamber. Food consists of 2g TetraMin® blended into 100 ml Culligan water. If there is an accumulation of excess food on the sediment surface, suspend feeding to avoid fungal growth and degraded water quality in the test chambers.

D. Test Termination - Day 10

1. Measure and record water quality (DO), pH, conductivity, and temperature) in surrogate test chambers. Remove a subsample for ammonia analysis from each site and preserve with 1 ml of 10% sulfuric acid.

2. Gently suspend sediment in the test chambers and pour onto 0.5-mm cylindrical screen. Rinse away remaining sediment using laboratory water within 2°C of the test temperature.

3. Count and record the number of surviving amphipods making note of any dead amphipods found.

4. Collect and rinse surviving amphipods with deionized water. Using forceps, gently place on an appropriately labeled and tared aluminum pan.

5. Dry in an oven at 60°C for 24 hours.

6. Remove animals from oven. Weigh to the nearest 0.0001g and record on the data sheet.

E. Reference Toxicant Testing

1. A 96-hour reference toxicant is run with the same batch of animals with sediment tests as a quality assurance procedure to assess the health of the test organisms and soundness and consistency of procedures. Copper chloride is used as a reference toxicant. The testing concentrations for the reference toxicant are 0 (Control), 100, 200, 400, 800, and 1600 µg/L.

2. The LC_{50} for survival is calculated and should be within two standard deviations of the historical control chart mean for AMEC.

IV. TEST ACCEPTABILITY CRITERIA

Mean control sediment survival at test termination must be 80 percent or greater. There is no acceptability criterion for the weight endpoint as it is not specified in the protocol.

V. HEALTH AND SAFETY

Health and safety precautions and applicable regulations should be considered at all times. Gloves must always be worn when handling test sediments.

VI. PERSONNEL

Only qualified technicians who have been properly trained and can demonstrate competency with these techniques are permitted to conduct this test.

VII. QUALITY ASSURANCE REQUIREMENTS

Quality assurance practices encompass all aspects of testing including the collection, handling, and preparation of test organisms, samples, and dilution waters. Proper record keeping is required and concentrated efforts are made for complete documentation on a real-time basis.

VIII. REFERENCES

"Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S.-Testing Manual." Environmental Protection Agency Office of Water/Department of the Army, US Army Corps of Engineers. EPA-823-B-98-004, February 1998.

"Standard Test Methods for Measuring the Toxicity of Sediment-Associated Contaminants with Freshwater Invertebrates." ASTM E 1706-00, 2002.

"Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates (Second Edition)." EPA 600/R-99/064, 2000.

STANDARD OPERATING PROCEDURE

for

TISSUE SAMPLE PREPARATION

SOP Code: MET-TISP
Revision: 5

February 12, 2007

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Approved By:

Supervisor		02/12/07 Date
QA Manager		2-12-07 Date
Laboratory Manager		2/12/07 Date

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Annual review of this SOP has been performed and the SOP still reflects current practice.
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Standard Operating Procedure for Tissue Sample Preparation

1. PURPOSE

This standard operating procedure describes procedures for the initial preparation of tissue samples prior to sample analysis. Customer-specific contracts or statement of works (SOWs) with alternate procedures will take precedence over this SOP.

2. APPLICABILITY

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This SOP is intended to provide guidance for the preliminary preparation of tissue samples prior to the sample aliquoting and analytical preparation described in individual analytical SOPs. The procedures described in this SOP also apply to compositing and subsampling of tissue samples for analyses to be subcontracted.

3. DEFINITIONS

Sample: The material presented to the laboratory for analysis or testing.

Sample Aliquot: A representative part or portion of a sample for analysis which is a fraction of the whole sample. See subsampling also.

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Compositing: The process by which sample aliquots from two or more samples are united to form a combined sample which is subsequently analyzed.

Subsampling: The process by which a representative portion is obtained from a whole sample.

Service Request: The service request (SR) is a document prepared at the time of sample receipt and summarizes sample analysis and reporting instructions about a customer's sample(s).

QAPP: Quality Assurance Project Plan document provided by the client specifically written for their project.

VOC Analyses: Volatile organic compounds (VOC) analyses, including halogenated and aromatic volatile organic compounds and gasoline range organics (GRO) analyses.

Non-VOC Analyses: Any analysis other than a VOC analysis.

4. DISCUSSION

4.1. Tissue samples are inherently heterogeneous requiring special considerations in order to obtain a truly representative sample aliquot for analysis. This SOP provides guidance for handling tissue samples prior to the sample preparation steps described in analytical SOPs. This SOP applies to samples delivered to the lab in whole body form or in the form of pre-resected tissues.

4.2. The sample handling strategy must consider

- what analyses are to be performed (metals, organics, or both, and VOC or non-VOC),
- how much sample is available
- are the analyses to be performed on individual samples or composite homogenates,
- are the analyses to be performed on whole body, edible portions or specific organs, and
- are any of the analyses going to be subcontracted which may require subsampling.

4.3. Proper preparation and handling of tissue samples is required to obtain a representative sample, avoid contamination, and to ensure loss of sample and target constituents is minimized.

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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

Refer to the determinative method.

7. APPARATUS AND EQUIPMENT

Note: Refer to the Procedure section for specific equipment used based on the determinative analysis to be performed. The use of implements and surfaces may vary depending on the analyses to be performed.

7.1. Hobart Food Chopper, or comparable device.

7.2. Tissumizer.

7.3. Waring blender, or similar device.

7.4. Freeze-drier, Labonco or equivalent.

7.5. Glass or PTFE cutting boards.

7.6. Knives and cutting implements – refer to Procedure section.

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7.7. Standard laboratory glassware (beakers, scintillation vials, etc.)

7.8. VOA vial – pre-cleaned, 40ml with Teflon-lined cap.

7.9. Pre-cleaned glass jars with PTFE lined lids, various sizes.

7.10. Gloves: Should be talc free and of non-contaminating materials.

7.11. Heavy duty aluminum foil.

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8. RESPONSIBILITIES

8.1. Sample custodians, together with project chemists and department supervisors, are responsible for documenting any required sample preparation (including the percent solids or percent lipids determination if required) on the service request. All personnel preparing tissue samples should be familiar with the contents of this document prior to commencing work.

8.2. Tissue sample preparation is to be performed only by lab analysts instructed in the proper handling techniques outlined in this SOP. It is the responsibility of the analyst to perform this procedure to complete all documentation required for data review.

9. PREVENTIVE MAINTENANCE

9.1. No specific maintenance steps are needed other than normal cleaning and inspection of apparatus.

9.2. For organics samples, polypropylene and polyethylene (plastic) surfaces, implements, and containers are a potential source of adsorption and contamination and should not be used. Gloves should be talc free and of non-contaminating materials.

10. PROCEDURE

10.1. Sample Login

Any special sample handling must be noted on the service request and on a label attached to the sample itself. During sample receiving, a sample custodian must follow the procedures described in Section 10.1.1.

10.1.1. Tissue Samples with Limited Quantity

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An assessment of the required sample quantity should be made by the project chemist when taking delivery of the sample. This assessment must take into consideration the tests, the required detection limits and the necessary quality assurance samples. If the quantity of sample given to the laboratory is insufficient for the analyses requested, the sample custodian will, along with the project chemist estimate the total amount of sample available. A "LIMITED SAMPLE VOLUME" tag is attached to the sample on which is recorded the estimated sample quantity.

In some cases it may be beneficial to perform sample preparations as described in this SOP prior to estimating the sample amount. In this case, the analyst prepring the sample will provide the project chemist with an estimate of the amount available.

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The project chemist must determine if limited sample quantity exists and set the priorities for the analyses and, if possible, estimate the quantity of sample to be *used for each test*. This information is to be documented and placed in the project file and on the service request to communicate to the laboratory staff. For example, 8080 use 10 g; metals use 1g.

NOTE. Samples that are quantity limited and require multiple analyses must be identified as soon as possible. Optimally, this should happen during sample login; however, discovery at any time should trigger appropriate actions as described in Section 10.1.1.

10.2. Sample Homogenization

This section outlines the steps for preparing homogenous samples of whole fish, edible fish (fillets), edible shellfish, worm composite homogenates, eggs, and plant tissues.

10.2.1. Samples for Organics Analyses:

Equipment used for the processing of tissue samples for organics analyses should be of stainless steel, anodized aluminum, glass or polytetrafluorethylene (PTFE).

Polypropylene and polyethylene (plastic) surfaces, implements, and containers are a potential source of adsorption and contamination and should not be used. Gloves should be talc free and of non-contaminating materials. Filleting should be done on glass or PTFE cutting boards that are cleaned properly between samples or on cutting boards that are covered with heavy duty aluminum foil (hexane rinsed) that is changed between samples. Tissue should be handled with precleaned, high quality, corrosion-resistant stainless steel instruments. Fillets or homogenate should be stored in cleaned glass jars of suitable dimensions with PTFE lined lids. If the sample is to be analyzed for VOCs, the homogenization steps should be performed on sample tissue that is partially frozen or chilled. An aliquot of the homogenate should be placed in a clean 40ml voa vial and labelled "FOR VOA ANALYSIS ONLY".

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Prior to handling each sample, utensils, cutting boards and containers should be washed in a detergent hot water solution and rinsed with tap water, hexane, and DI water. Precleaned, certified sample containers may be used without further cleaning. If the sample is to be analyzed for VOCs, methanol is substituted for the rinsing of implements with acetone and the hexane rinsing of the aluminum foil. Exposure to solvent vapors must be minimized.

10.2.2. Samples for Metals Analyses:

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Equipment used in the processing of samples for metals analyses should be of PTFE, ceramic, polypropylene or polyethylene. Filleting should be performed on PTFE cutting boards which are cleaned after each sample. Knives with titanium or high quality stainless steel blades may be used for tissue resections. Tissue should be stored in glass jars with PTFE lined lids.

Prior to sample handling, utensils, cutting boards and containers should be washed in a detergent hot water solution, rinsed with tap water, 25% HCL (except metal utensils), and DI water. Precleaned, certified sample containers may be used without further cleaning.

10.2.3. Samples for both Metals and Organics Analyses:

If the sample is to be prepared for both organics and metals, care must be taken to use equipment and cleaning procedures that are non-contaminating for both.
Quartz, ceramic, glass and PTFE are recommended materials for sample processing equipment. Knives with titanium or high quality stainless steel blades may be used for tissue resections. Glass or PTFE cutting boards should be used.

If the sample is to be analyzed for VOC's, the homogenization steps should be performed on sample tissue that is partially frozen or chilled. An aliquot of the homogenate should be placed in a clean 40ml voa vial and labelled "FOR VOA ANALYSIS ONLY".

Prior to handling each sample, utensils, cutting boards and containers should be washed in a detergent hot water solution and rinsed with tap water, acetone, methanol, or hexane (as appropriate), and DI water. Precleaned, certified sample containers may be used without further cleaning. Non-metallic surfaces and utensils should also be rinsed with 25% HCL followed by DI water. If the sample is to be analyzed for VOCs, methanol is substituted for the rinsing of implements with acetone. Exposure to solvent vapors must be minimized.

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10.2.4. Sample Preparation

Each tissue sample may be homogenized in the original glass bottle container if there is sufficient space to allow thorough mixing. If homogenization is not achievable in the original container, place the entire sample contents into a clean glass jar. Generally, liquids contained in the container are to be considered part of the sample. If the sample requires size reduction prior to homogenization, chop the sample into the 1-2" chunks using a titanium or stainless steel bladed knife. Large samples may require the use of industrial food processors such as a Hobart Food Chopper, or comparable device. Size-reduced chunks of tissue are thoroughly homogenized to a paste-like consistency using a Tissumizer, Waring blender, or similar device until it reaches a paste-like consistency. Transfer the sample paste to a glass jar for storage and freeze until ready for sample extraction. The new sample container is labeled with the sample I.D., the word "homogenized", initialed, and dated.

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10.2.4.1. Whole Fish Tissue

Samples may be frozen in the field or in the laboratory. While still partially frozen, rinse the fish with DI water to remove extraneous materials and liquids. Cut the fish into appropriate size chunks and mechanically macerate the sample using cutting tools appropriate for the size of the sample and the analysis type. If necessary, process fish tissue chunks through the Hobart Food Chopper. To ensure thorough mixing, divide the ground sample into quarters, mix opposite quarters and then mix halves. Homogenize sample using a Tissumizer or Waring blender until it reaches a paste-like consistency. Transfer the sample paste to a glass jar for storage and freeze until ready for sample extraction. The new sample container is labeled with the sample I.D., the word "homogenized", initialed, and dated.

10.2.4.2.Edible Fish Tissue

If the client or QAPP indicates that only edible tissue be analyzed, the fish must be filleted. If the sample arrives prefilleted, the sample tissue may be frozen before processing. If the sample is not yet filleted, the sample should remain chilled until the filleting is completed. Freezing can result in the contamination of edible tissues from the bursting of internal organs. Fish having ruptured internal organs should be noted on the prep benchsheet and the Project Chemist consulted. Rinse the fish with DI water to remove extraneous materials and liquids. Remove scales from scaled fish or skin from non-scaled fish. Rinse the fish again prior to filleting. A separate or clean cutting board should be used for filleting. Gloves should be changed between samples. Carefully remove the fillets from the carcass by following the steps outlined in Appendix A. Care should be taken to avoid contaminating fillet with inadvertent puncture of internal organs. Cut the fillet tissue into appropriate size chunks and mechanically macerate the sample using cutting and grinding tools appropriate for the size of the sample and the analysis type. Proper selection of maceration equipment must consider the potential contaminants, sample size/volume and amount of tissue likely to be lost in using the equipment.

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Divide the ground sample into quarters, mix opposite quarters and then mix halves. Again homogenize the sample using an appropriate blending mixer. Continue repeating this process until the sample is truly homogenous and no chunks of tissue remain. Freeze sample until ready for extraction.

10.2.4.3.Shellfish Tissue

Shellfish should be frozen as soon as possible after receipt by the laboratory unless samples can be prepared within 48 hours of sampling. Edible portions of various shellfish are described below and resection described in Appendix B. Thawing of frozen shellfish samples should be kept to a minimum during tissue removal to avoid loss of liquids. Shellfish should be rinsed with DI water prior to tissue removal to dislodge external debris. When multiple organisms constitute a single sample, the edible tissues are collected, composited and homogenized.

10.2.4.3.1.Bivalve mollusks (oysters, clams, mussels, and scallops).

Bivalves are typically prepared by severing the adductor muscle, prying open the shell, and removing all of the soft tissue. The soft tissue includes viscera, meat, and body fluids.

10.2.4.3.2.Crabs

Edible tissue includes all leg and claw meat, back shell meat and body cavity meat. Internal organs generally are removed. If the crab is soft shelled, the entire crab is used in the sample.

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10.2.4.3.3.Shrimp and Crayfish - Edible tissue includes the tail meat.

10.2.4.3.4.Lobster - Edible tissue includes the tail and claw meat.

10.2.4.4.Worms

Samples are typically supplied to the lab in sample jars containing multiple organisms. Liquid and specimens constitute the entire sample and are blended together typically in the sample container. When a worm sample containing dirt particles or significant amounts of water is encountered, the technician should contact the project chemist to seek guidance from the client.

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10.2.4.5.Eggs

Avian eggs are typically removed from the shell and blended. Aquatic eggs are blended including the soft shell.

10.2.4.6.Internal Organs Extraction

Organs such as livers or kidneys must be identified and removed by an experienced sample technician following clear written resection procedures or other guidance provided by the client.

10.2.4.7.Plant Tissue

Plant tissue should be handled using the size reduction, homogenization and implement cleaning steps outlined in Sections 10.2.1, 10.2.2, 10.2.3, and 10.2.4. Where these procedures are inappropriate, specific written procedures or guidance from the client is recommended.

If drying is requested by the client or is project-specified, a subsample for mercury analysis is taken from the wet sample, then the plant tissue is dried at 60°C prior to homogenization.

10.2.4.8.Small Mammals and Rodents

10.2.4.8.1.There are two primary concerns in working with small mammals and rodents: safety and sample homogenization.

10.2.4.8.2.Small mammals are potential carriers of lethal viruses, such as hantavirus and rabies, and bacteria that can be contracted through inhalation or direct contact. Typically, these organisms are excreted in the feces and distributed on the air as the fecal matter dries. During the sample preparation process, tissue is typically freeze-dried in order to calculate a percent solids value and to analyze for metals. As such, it is possible to increase the potential for dispersion of the bacteria or viruses after the sample is homogenized and processed. Prior to processing, all samples should be stored frozen and shipped to a subcontractor for irradiation. Minimum irradiation dosage is 3 - 4 Mrads. The samples should be frozen and shipped next day priority in an insulated cooler packed on ice or dry ice. Use enough ice to ensure the samples remain frozen until return of the samples (use a three day time line in estimating the amount of ice needed to ensure samples remain frozen). Care should be take to minimize air space in the cooler thereby maximizing insulating potential. It is recommended that the cooler be chilled prior to packing.

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10.2.4.8.3.Prior to sample homogenization, instructions should be received from the client regarding the processing of the hide. For organics, it is recommended that the hides be left on the carcass and the entire sample be homogenized. For metals, there is a potential for accumulation in the hair. As a non-digestible portion of the rodent, inclusion of the hair may result in a high bias if the data is to be used in estimating bioaccumulation up the food chain. Skinning may be a preferred alternative when metals are the primary chemicals of concern.

10.2.4.8.4.Homogenization should be done while the carcass is still partially frozen.

10.2.4.8.5.If the hide is to be included in the homogenization, snip the feet from the animal using stainless steel scissors.

The tail should be removed if it will prevent complete homogenization of the sample (e.g., the tail of a mouse or rat may result in incomplete homogenization and should not be included with the sample). Remove seeds, grasses, and mud from the hide.

10.2.4.8.6.If the hide is to be removed from the carcass, make an incision through the skin on the back of the neck (do not cut into the muscle). In most cases, the hide can be removed by pulling the incision horizontally along the back in one direction, and over the ears, head and snout in the opposite direction. The eyes are usually lost during this procedure. Continue to skin the animal by peeling the hide over the hind legs, off the underside of the animal, and around the front legs. The hide is removed at the hind legs and the snout. Care should be taken not to tear the connective tissue under the hide. Fat should be scraped from the hide when possible and included with the sample. Rinse the skinned carcass with DI water to remove any hair or dirt that has accumulated during the skinning procedure.

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10.2.4.8.7.Homogenize the sample using a stainless steel Waring blender. Select a blender cup that is sized in accordance with the amount of sample to be homogenized. That is, small samples should be homogenized using small blender cups. This will improve the overall homogenization and recovery of the sample. Continue to mix the sample into a paste like consistency. Make sure no chunks of muscle, hide, or bone are distributed in the sample. Transfer the sample paste to a glass jar for storage and freeze. The new sample container is labeled "homogenized", initialed, and dated.

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10.2.5. General Provision for Handling Large Sample Mass

In some cases, large specimens will be received by the laboratory for homogenization prior to chemical analysis. For the purpose of this SOP, 'large' is defined as requiring preliminary size reduction to allow sequential processing of the sample. Sub-samples of the whole specimen should be cut to a size appropriate for the blender, mixer, or grinder that will be used. After each individual fraction is processed, the homogenized material is added to a reservoir large enough to hold all fractions as they accumulate. The reservoir will be constructed of a material suitable for the analytical application as defined under Section 10.2.3. For very large specimens (i.e. >20 pounds), high grade stainless steel containers are used (large bowls or small drums).

Blending of the combined fractions to achieve a whole homogenous material is achieved via manual mixing. In general, this is accomplished using a high grade

stainless steel paddle or spoon of appropriate size (i.e. relative to the whole homogenate). Very large specimens (i.e. >20 pounds) generally require secondary processing through the grinder, particularly when large amounts of skin, bone, and/or cartilage is present. In these cases, the Hobart grinder is generally used.

10.3. Compositing

Each sample is to be logged in and receive a lab code. Additionally, the sample composite also is assigned a lab code. The compositing process is to be performed by trained staff. It is to be performed in an area free of contamination. It is imperative that the samples are treated in a manner consistent with the requirements of the tests to be performed on the composited sample. Compositing of homogenates should be performed according to this SOP or specific instructions provided by the client.

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10.3.1. Documentation

The analyst preparing the composite will document

- that homogenization was done before removing an aliquot,
- the quantity of each (field or discrete) sample used for the composite,
- the date and time of compositing, and
- any other pertinent observations.
- An example of a data sheet that can be used for this purpose is given in Appendix C.

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10.3.2. Tissue Samples with Limited Quantity

Samples and sample composites that are quantity limited will be handled by the same procedure as described in Section 10.1.1

10.3.3. Compositing Procedure

10.3.3.1. Each tissue sample is first homogenized as per instructions in Section 10.2.

10.3.3.2. An equal weight of sample aliquot from each of the homogenized samples is weighed into a clean glass sample bottle. The amount to be weighed of each sample will depend upon the number of analyses to be performed on the composite and if the quantity of any individual sample is limited.

10.3.3.3. The mixture of the individual sample aliquots is thoroughly homogenized in the glass container. The composite sample bottle is labeled with:

- the name of the composite,
- the lab code of the composite,
- the analyst's initials
- the date of composite preparation
- The composite sample and the remaining (individual, discrete) samples are stored frozen until analysis.

10.3.4. Tissue Samples Requiring VOC Analyses

A separate aliquot of the composite homogenate should be placed in 40ml voa vial container for later analysis by the VOA department. Each container should be labelled with the lab identifier, date, initials, and "FOR VOA ANALYSIS ONLY". To minimize losses of volatile constituents, the sample should be kept as cold as possible, the work should be completed as quickly as possible, and the VOA vial filled to the top to minimize head space.

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10.4. Subsampling

The sample is first thoroughly homogenized as per Section 10.2. A sample aliquot is removed and placed into a clean glass container of appropriate size and labelled as follows:

COPY

- the name of the sample,
- the lab code of the sample,
- "homogenized" written on the label,
- the purpose of the sub-sample (e.g. "dioxin subsample")
- the analyst's initials
- the date.

10.5. Freeze-Drying

10.5.1. Depending on project specifications, samples may require freeze-drying. Freeze-drying may be performed on a separate portion of sample to determine % Freeze-Dried Solids, or may be done on the analytical subsample for certain tests. The analyst should obtain direction from the supervisor and/or Project Chemist.

10.5.2. Weigh 5-8 g of sample (wet weight) into a scintillation vial. Freeze the sample for at least 2 hours.

10.5.3. Remove the sample from the freezer and place in the freeze drier for at least 24 hours, or longer if necessary for the particular sample matrix.

10.5.4. Record the measurements on the benchsheet in Appendix C.

11. QA/QC REQUIREMENTS

A rinsate blank should be prepared to accompany each batch of tissue samples. The blank is comprised of a collection of DI water rinses of cleaned equipment (knives, cutting boards and mixers/grinders) prior to the commencement of sample batch preparation. If contamination of the samples is suspected, the rinsate blank is extracted and analyzed for contaminants. The rinsate blank should be labeled with the extraction date and the associated SR numbers and stored at 4 C. In the event that contamination is suspected, the rinsate blank can be analyzed to confirm the presence of contaminants in the tissue preparation process.

12. RECORDS

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Sample handling documentation must include information about sample homogenization (was it done or not), compositing, and subsampling. The data benchsheets in Appendix C provide a means for recording this information. This sample handling information is to be filed in the project file with the raw data.

13. REFERENCES

Kateman and L. Buydens, *Quality Control in Analytical Chemistry*, Second Edition, John Wiley & Sons, Inc., New York, NY, 1993: Chapter 2 on Sampling and especially sections 2.5 (Sample Quality) and 2.7 (Handling of Samples).

COPY

Guidance For Assessing Chemical Contaminant Data For Use In Fish Advisories; Volume 1; Fish Sampling and Analysis, 3rd Edition; USEPA Office of Water; EPA 823-B-00-007; Nov 2000.

Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound; Tetra Tech, Inc.; final report TC-3991-04 Recommended Guidelines for Measuring Organic Compounds in Puget Sound Sediment and Tissue Samples Revision April 1996.

PCB's and Mirex In Fish Tissue and Clams New York State Department of Health Wadsworth Center For Laboratories and Research; Albany, N.Y. 10/6/81

Draft Method 1613-Tissue; Determination of PCDDs and PCDFs in Fish and Other Tissue Using Method 1613; USEPA Office of Water June 1993.

APPENDIX A

Fish Filleting Procedure

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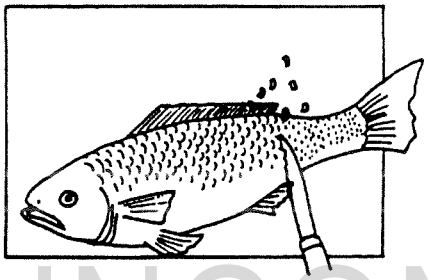
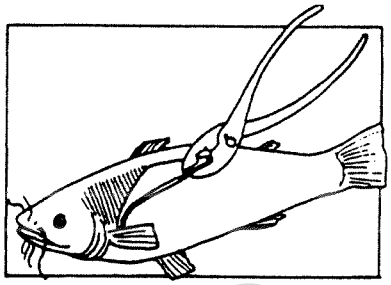
1 Scaled Fish

After removing the scales (by scraping with the edge of a knife) and rinsing the fish:

1b Scaleless Fish

Grasp the skin at the base of the head (preferably with pliers) and pull toward the tail.





Note: This step applies only for catfish and other scaleless species.

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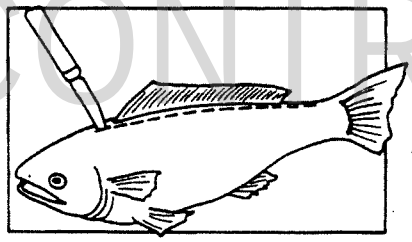
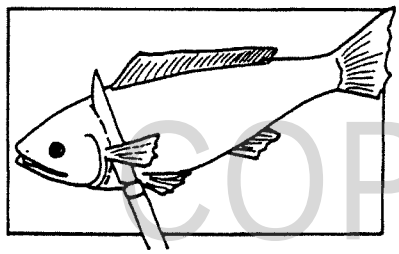
2



Make a shallow cut through the skin (on either side of the dorsal fin) from the top of the head to the base of the tail.

3



Make a cut behind the entire length of the gill cover, cutting through the skin and flesh to the bone.

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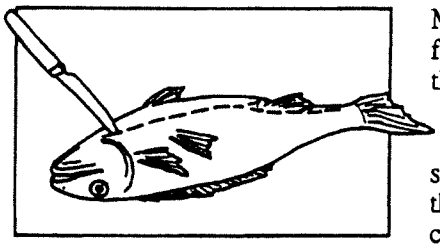
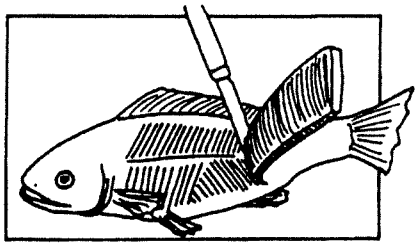
4



Make a shallow cut along the belly from the base of the pectoral fin to the tail. A single cut is made from behind the gill cover to the anus and then a cut is made on both sides of the anal fin. Do not cut into the gut cavity as this may contaminate fillet tissues.

5



Remove the fillet.

Source: U.S. EPA, 1991d.

Figure 7-3. Illustration of basic fish filleting procedure.

APPENDIX B

General Procedure for Removing Edible Tissues From Shellfish

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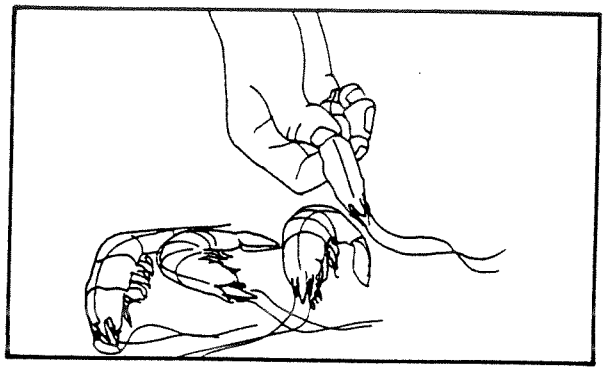
Heading, peeling and deveining shrimp

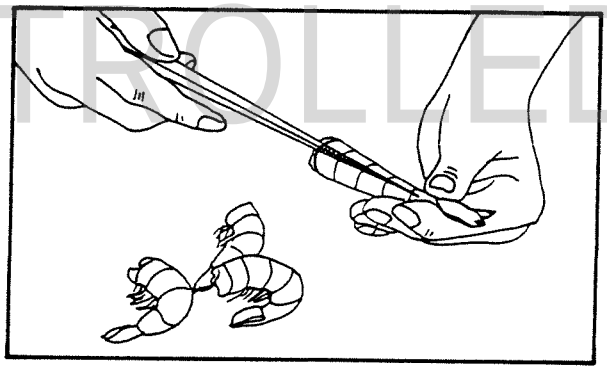
1

To head a shrimp, hold it in one hand. With your thumb behind shrimp head, push head off. Be sure to push just the head off so that you do not lose any meat.



2

If using a deveiner, insert it at head end, just above the vein.



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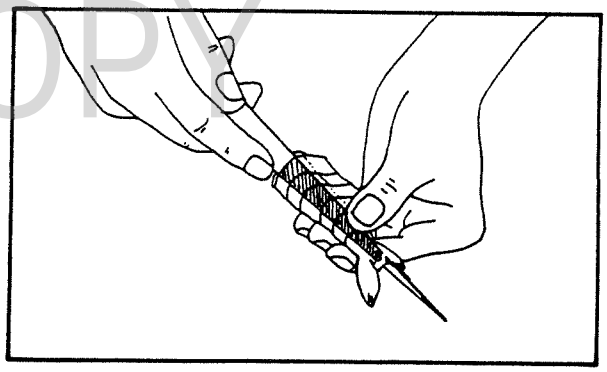
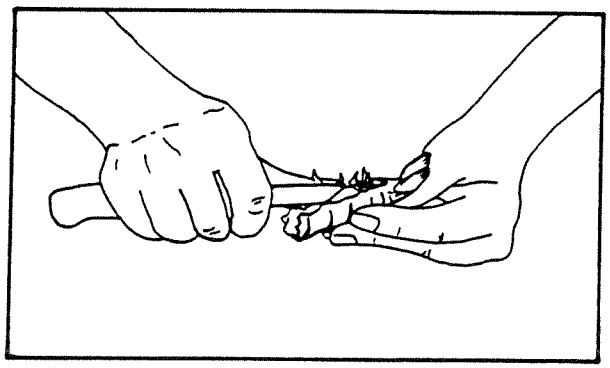
3

Push through shrimp to the tail and split and remove shell. This removes vein at the same time.



4

If you prefer to use a paring knife, shell shrimp with your fingers or knife. Then use knife to gently remove vein.

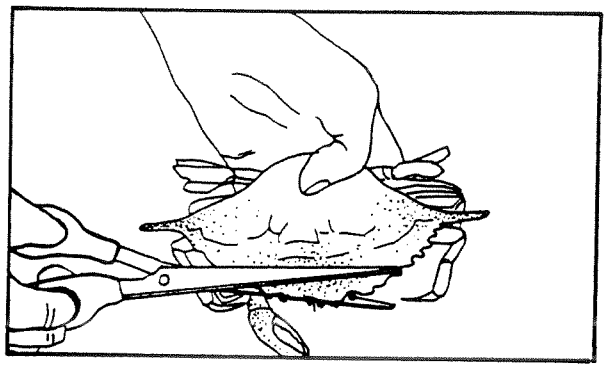


Source: UNC Sea Grant Publication UNC-SG-88-02

Cleaning soft-shell crabs

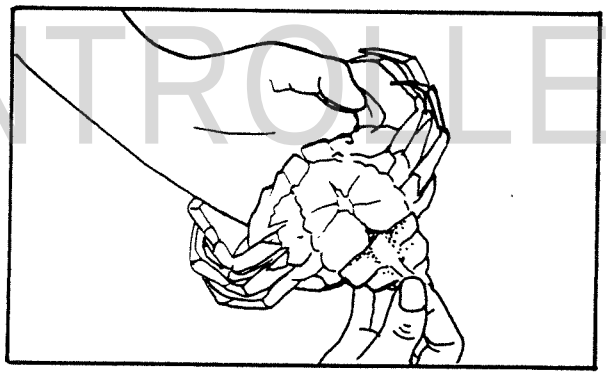
1

Hold crab in one hand and cut across body just behind eyes to remove eyes and mouth.



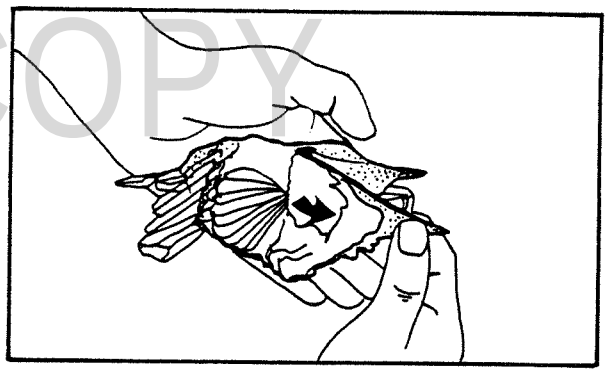
2

Turn crab on its back. Lift and remove apron and vein attached to it.



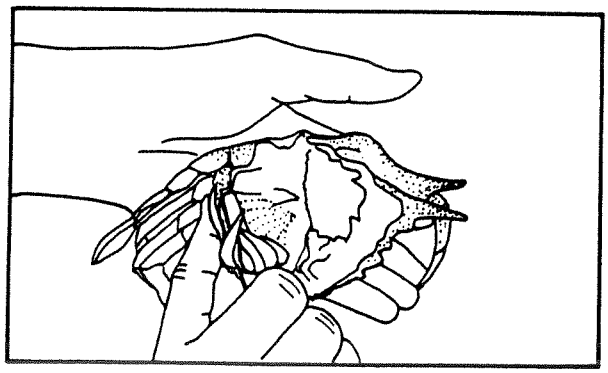
3

Turn crab over and lift one side of top shell.



4

With a small knife, scrape off grayish-feathery gills. Repeat procedure on other side.



Source: UNC Sea Grant Publication UNC-SG-88-02

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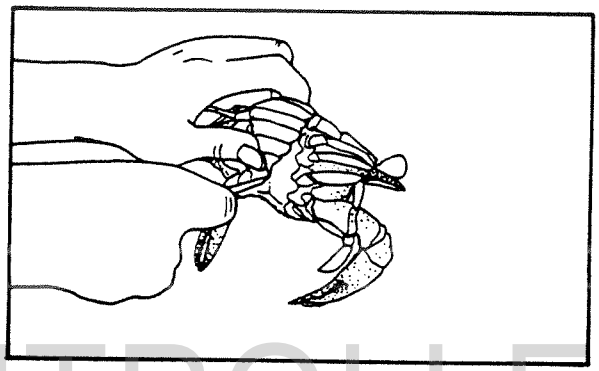
Cleaning hard-shell crabs

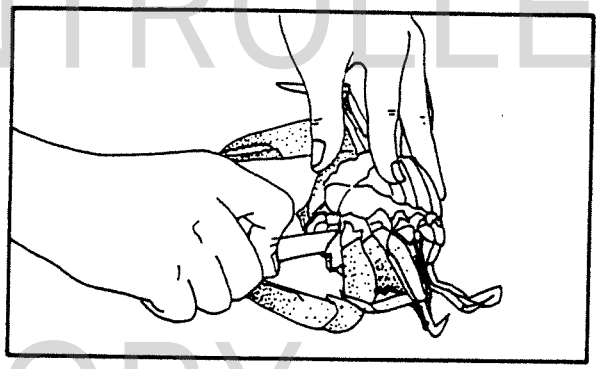
1

Hold crab in one hand. Turn crab over and stab straight down at point of apron with a knife.



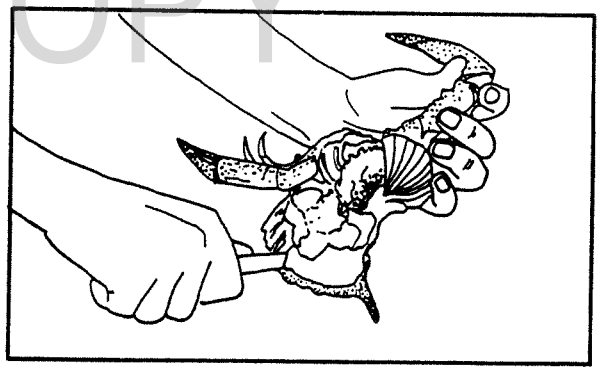
2

Make two cuts from this point to form a V-pattern that will remove mouth.



3

Do not remove knife after making second cut. Firmly press crab shell to cutting surface without breaking back shell. With other hand, grasp crab by legs and claws on the side where you are holding knife, and pull up. This should pull crab body free from back shell.



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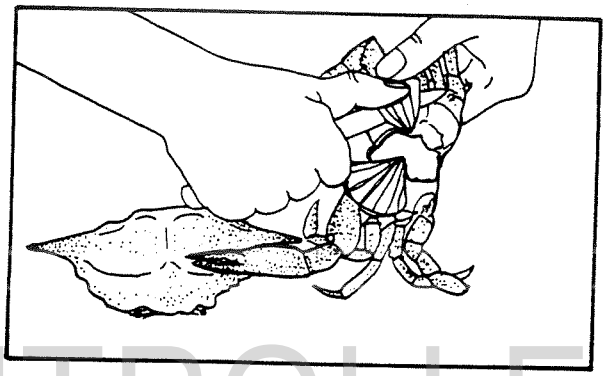
4

Remove gray, feathery gills, which are attached just above legs. Cut and scrape upward to remove gills.



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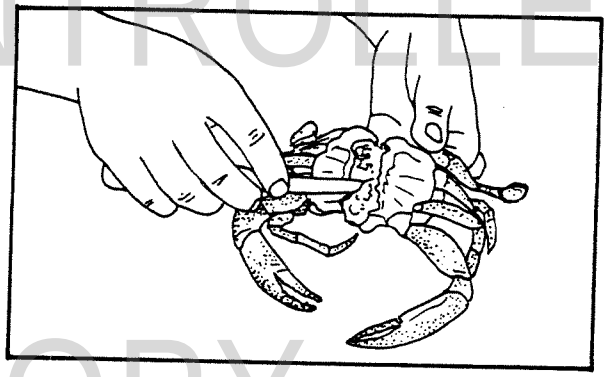
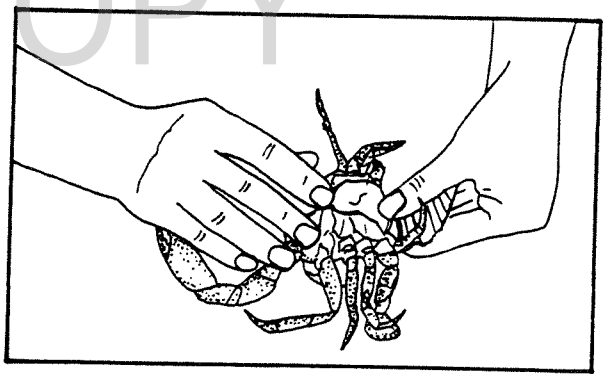
5

Remove all loose material—viscera and eggs—from body cavity.



6

If apron did not come loose with shell, remove it.

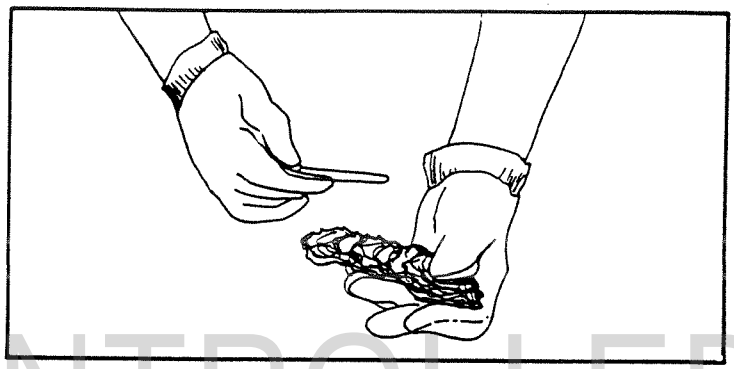


Source: UNC Sea Grant Publication UNC-SG-88-02

Shucking oysters

1

Oyster shells are especially sharp; be sure to wear gloves to protect your hands. Chip off a small piece of shell from the thin lip of the oyster until there is a small opening.



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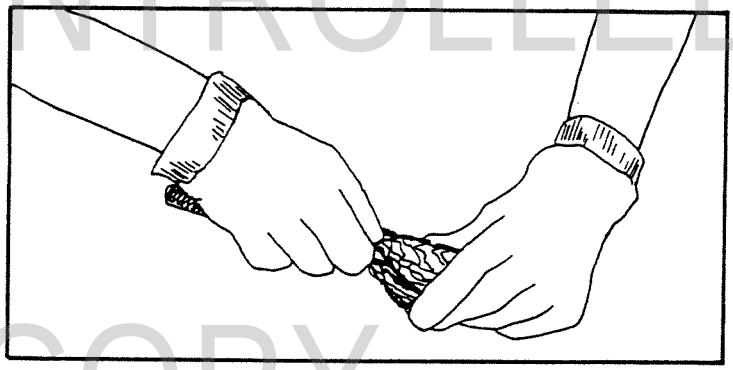
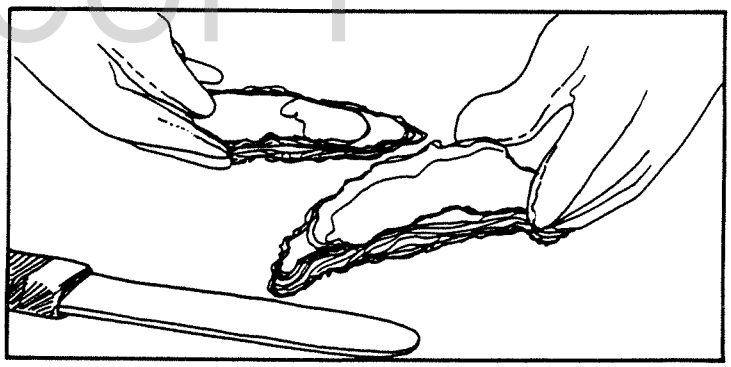
2

Insert knife blade into the opening and cut muscle free from top and bottom shells.



3

Remove oyster meat from the shell.

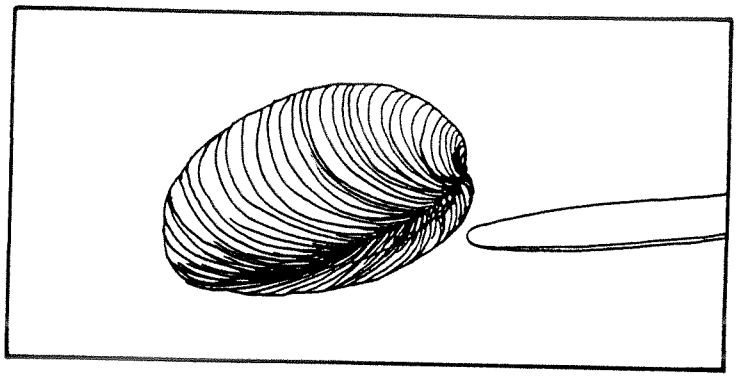


Source: UNC Sea Grant Publication UNC-SG-88-02

Shucking clams

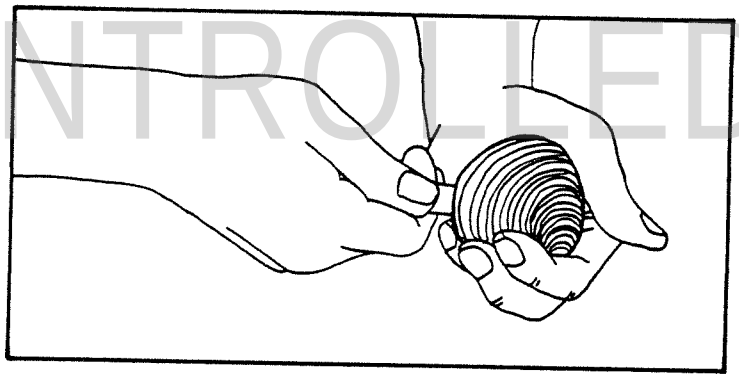
1

In the back of clam near the hinge is a black ligament. Toward the front where ligament ends is a weak spot. Insert your knife at this spot.



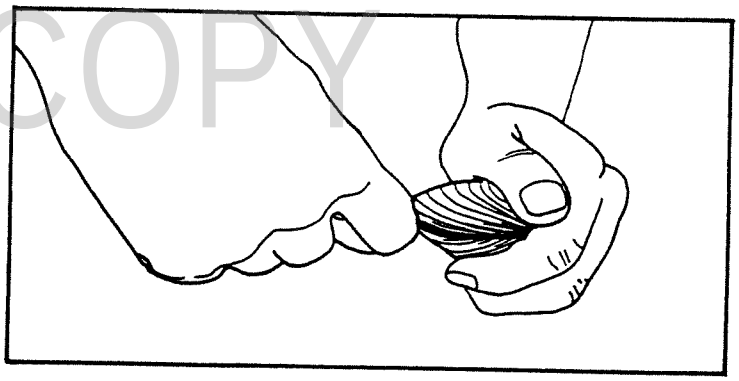
2

Inside are two muscles. Run the knife around the shell to sever both muscles.



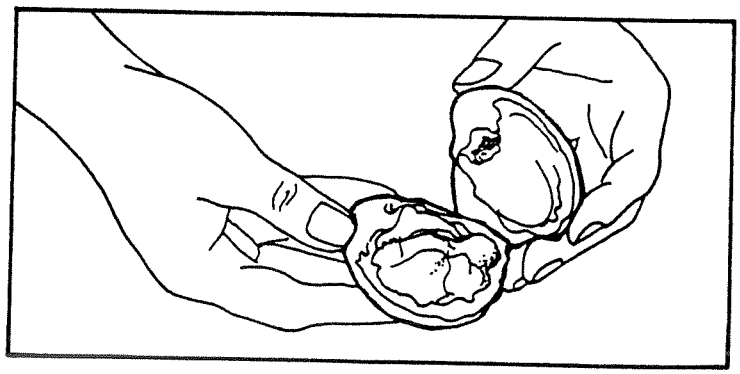
3

Now insert the knife blade into the front of the shell and separate the two shells.



4

Scrape the meat free from the top and bottom shell.



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APPENDIX C

Compositing and Freeze-Drying Bench Sheets

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COLUMBIA ANALYTICAL SERVICES, INC.

Work Request #:_____ Date Composited:_____ Analyst:_____

Tissue Composition Data

Lab ID	Weight Before Composition	Length Before Composition	Observations/ Comments

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Comments:_____

_____

_____

Date Samples Homogenized:	Analyst:

COLUMBIA ANALYTICAL SERVICES, INC.

Service Request #: _____

Analysis For: Freeze Dried Solids

Lab Code	Wet Weight (g)	Tare (g)	Tare + Dry Wt.(g)	Dry Weight (g)	% Total Solids

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Time In : _____ Time Out: _____

Comments:

$\bar{x}$ = RPD =

Analyst:	Date:
Reviewed By:	Date:

STANDARD OPERATING PROCEDURE

POLYCYCLIC AROMATIC HYDROCARBONS BY GC/MS SELECTIVE ION MONITORING

EPA Method 8270C SIM

SOC-8270P
Revision 6
March 8, 2007

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Approved By: _____ Supervisor — 3/8/07 Date

_____ QA Manager — 3-8-07 Date

_____ Laboratory Manager — 3/8/07 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: _____
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NUMBER: _____

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POLYCYCLIC AROMATIC HYDROCARBONS BY GC/MS SELECTIVE ION MONITORING
Method 8270C SIM

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine the concentrations of Semi-Volatile Organic Compounds in water, soil, and tissue matrices using EPA Method 8270C SIM. This procedure may also be applicable to various miscellaneous waste samples. Table 1 lists compounds that may be determined by this method and the method reporting limits (MRLs) in water, soil, and tissue. Alkylated PAHs listed in Table 1A may also be determined using this procedure. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL) and Practical Quantitation Limit (PQL). Therefore, MRL=EQL=PQL. Table 1 lists Method Detection Limits (MDLs) that have been achieved, however, MDLs may change as MDL studies are performed,

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1.2. The procedure is intended for samples containing trace-level amounts of target compounds. Samples containing high concentrations of target analyte will not be analyzed undiluted. Extracts may be screened using GC/FID to estimate the hydrocarbon content and concentrations of individual polynuclear aromatic hydrocarbons (PAHs). Samples containing PAHs in excess of five times the high calibration standard will be diluted prior to analysis. All MRLs will be adjusted in accordance with this dilution. Therefore, samples containing high levels of PAHs will not be analyzed to achieve the optimum MRLs for the analysis.

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1.3. This procedure can be used to quantitate most neutral 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. This procedure is optimized for the analysis of polynuclear aromatic hydrocarbons.

1.4. Other compounds than those listed in Tables 1 and 1A may be analyzed. However, analytes not summarized in Table 1 have not been validated with a method detection limit study. Therefore, the lab will not use this procedure to analyze for non-routine analytes unless a similar analyte has been validated with a MDL study. As a general rule, the MRL for these compounds will equal the MRL of a similar compound in the routine analyte list. Results will not be reported below this estimated MRL.

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, water

samples are extracted using either method 3520 (SOP EXT-3520) or 3535 (SOP EXT-3535) and soil/solid samples are extracted using 3541 (SOP EXT-3541).

2.2. All soil, tissue, and colored water extracts will be cleaned using method 3630 (silica gel cleanup, SOP SOC-3630) prior to analysis. In cases where project-specified analytes are not amenable to silica gel cleanup, gel permeation chromatography (SOP SOC-3640) will be used for cleanup prior to analysis.

2.3. A 5.0 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 in the selective ion mode. 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.

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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) acquired in full scan mode followed by initial calibration standard(s) acquired in SIM mode. Once calibrated, a CCV is evaluated and extracts can be run. The sequence ends after 12 hours based on the DFTPP acquisition time.

3.2. **Laboratory Control Sample (LCS)** - In the LCS analysis, predetermined quantities of standard solutions of all analytes are added to a blank matrix prior to sample extraction and analysis. The purpose of the LCS is to monitor analytical control for the sample batch. Percent recoveries are calculated for each of the analytes.

3.3. **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, then spiked and analyzed. Percent recoveries are calculated for each of the controlled analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.4. **Standard Curve** - A standard curve is a plot of concentrations of a known analyte standard versus the instrument response to the analyte.

3.5. **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 matrix spikes prior to extraction. Percent recoveries are calculated for each surrogate.

3.6. **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.

3.7. **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.

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3.8. **Second Source Verification Standard or Independent Verification Standard (SSV or 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.

3.9. **Selective Ion Monitoring (SIM)** – Mass spectrometry technique where ions resulting from fragmentation are selectively monitored, therefore excluding other ions. The technique enhances sensitivity as compared to full scan analysis. Because the analysis results in significantly less mass spectral information, this gain in sensitivity is made at the expense of analyte selectivity. Therefore, the use of SIM results in significantly lower instrument detection limits, but increases the uncertainty associated with the analysis.

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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. 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 an instrument blank to check for carryover.

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 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. Certified clean containers should be purchased for sample collection. Alternatively, containers used to collect samples for the determination of semivolatile organic compounds should 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. Water and soil samples should be iced or refrigerated at 4 ± 2°C from time of collection until extraction. Tissue samples are stored frozen until extraction.

6.3. Water samples must be extracted within 7 days. Soil samples must be extracted within 14 days. Holding times for tissues are typically defined by project specifications, otherwise tissue samples may be held frozen up to one year before extraction. Extracts are stored at -10°C and must be analyzed within 40 days following extraction.

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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. An injector capable of large volume injection must be attached to the GC system. Optic 2 and Optic 3 systems are recommended.

7.1.2. Column: 5% Dipenyl, 95% Dimethyl Polysiloxane - 30 m x 0.25 mm ID x 0.25 µm film thickness silicone-coated fused-silica capillary column or equivalent. Recommended: Restek XTI-5 with Integra-guard, catalog #12223-124.

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, and capable of operating in the SIM mode.

7.1.4. GC/MS Interface - Any GC-to-MS interface that gives acceptable calibration points 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.

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7.1.6. Instrumental systems are identified as follows:

Instrument I.D.	Analytical System	Routine Matrix
MS11	6890/5973	Water/Soil
MS14	6890/5973	Tissue
MS06	5890/5972	Water/Soil (overflow capacity)*
MS17	6890/5973	Soil/Tissue

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* MDL studies are not maintained for overflow capacity instrumentation. Prior to any sample analyses on this instrument, MDL studies for each matrix and preparation method will be analyzed.

7.2. Appropriate analytical balance (0.0001 g), volumetric flasks, syringes, vials, and bottles for standards preparation.

8. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

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

8.2.1. Commercially prepared stock standards are typically used when available at a concentration of 100 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.

8.2.2. Transfer stock standard solutions into amber Teflon-sealed crimp top autosampler vials. Store at -10°C and protect from light, or store as recommended by the manufacturer. Standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

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8.2.3. Unopened stock standards and neat materials have an expiration date equal to the manufacturer's recommendation. Neat material that does not have a manufacturer's recommended expiration date should be replaced after three years. Stock standard solutions received in sealed ampules with manufacture expiration dates in excess of 1 year have an expiration date of 1 year from the date of opening the sealed ampule.

8.2.4. For the alkylated PAH option, a PAH-ALKH SRM is used. This SRM is a petroleum crude oil which has been cleaned up using GPC and silica gel cleanup procedures. All of the homologs are present in the SRM.

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8.3. Internal Standard Solutions - The internal standards are naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12} (See Table 3 for corresponding compounds). The nominal concentration of the standard is 20 ng/µL. See Table 5 for standard preparation instructions. Each 1 ml of sample extract undergoing analysis should be spiked with 10 µL of the internal standard solution, resulting in a concentration of 0.2 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 - A methylene chloride solution containing 50 ng/µL of decafluorotriphenylphosphine (DFTPP). The standard should also contain 50 ng/µL of pentachlorophenol and benzidine to verify injection port inertness and GC column performance. This injection is acquired in full scan mode and evaluated in accordance with the method specified criteria. Store at -10°C or less when not being used, or store according to the manufacturer's recommendations.

8.5. Calibration Standards

8.5.1. Prepare an intermediate surrogate standard by diluting 40uL of the 5000ppm stock to 2.0mL in DCM, resulting in 100ug/mL. An intermediate standard is prepared to combine the PAHs and surrogates into a standard that is used to prepare the calibration standards. See Table 5 for preparation instructions.

8.5.2. A minimum of six initial calibration standards should be prepared from stock solutions (note that a seven point calibration is recommended). 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 samples, but should not exceed the working range of the GC/MS system. Each 1 ml aliquot of calibration standards should be spiked with 10 µL of the internal standard solution prior to analysis.

8.5.3. All calibration standards should be stored at -10°C or less and should be freshly prepared from stocks every 365 days, or sooner if check standards indicate a problem.

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8.5.4. The following calibration standards are recommended: 0.002 ng/µl, 0.004 ng/µl, 0.008 ng/µl, 0.02 ng/µl, 0.1 ng/µl, 0.2 ng/µl, 0.4 ng/µl, 1.0 ng/µl, 1.6 ng/µl and 2.0 ng/µl. See Table 5 for preparation instructions.

8.5.5. The independent calibration verification (ICV) standard is prepared at a nominal 0.4 ng/µL concentration from stock solutions (see Table 5). The ICV is prepared at the time of initial calibration and can be stored at 4°C ± 2°C.

8.5.6. The daily calibration standard (CCV) is prepared at a nominal 0.4 ng/µL concentration from stock solutions (see Table 5). The CCV is prepared weekly and can be stored at 4°C ± 2°C.

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8.6. QC Standards

8.6.1. Surrogates: Prepare a working spiking solution in methanol containing Fluorene-d10, Fluoranthene-d10, and Terphenyl-d14 at 100 ng/µL. This solution may be combined with the surrogate solution used to monitor analyses for 8270 full list. Aliquots of the solution are spiked into all extracted samples, blanks, and QC samples according to the extraction SOP used.

8.6.2. Matrix Spike Standards: Prepare a working spiking solution in methanol containing all analytes of interest ("full list spike"). All analytes are prepared at 25 ng/µl. Aliquots of the solution are spiked into the selected QC aliquots according to the extraction SOP used.

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.

9.4. Mass Spectrometer

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9.4.1. 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.

9.4.2. 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.

9.4.3. Tune the MS as needed to result in consistent and acceptable performance while meeting the required ion abundance criteria.

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, as described in 8270C, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. Water, soil, tissue and waste samples are prepared using the appropriate extraction and cleanup methods (refer to SOPs) and may be screened by GC/FID (see SOP SOC-SCR).

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11.1.2. An appropriate cleanup procedure may be performed depending on the sample matrix and target analytes. Perform cleanups on all soil, tissue, and colored water extracts using method 3630 (silica gel cleanup, SOP SOC-3630) prior to analysis. In cases where project-specified analytes are not amenable to silica gel cleanup, perform method 3640 GPC cleanup (SOP SOC-3640) prior to analysis.

11.1.3. 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:

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Ion dwell time:	10 – 50 msec per ion
Initial temperature:	40°C, hold for 1 minutes
Temperature program:	40-140°C at 30°C/min hold for 0 minutes
Temperature program:	140-270°C at 10°C/min hold for 4 minutes
Final temperature:	270-320°C at 20°/min, hold for 1.10 minutes
Injector temperature:	320°C
Detector interface temp:	300°C
Injector:	splitless, electronic pressure control with pulse
Sample volume:	5.0 µL
Carrier gas:	helium at 1.2ml/min (constant flow)

11.3. Selected Ion Acquisition

11.3.1. Determine the ions to be monitored for the compounds of interest. Refer to Table 2 for characteristic ions. At a minimum, 2 ions should be monitored for each compound, and 3 monitored for compounds with more complex fragmentation patterns. Set the SIM windows in order to monitor the correct ions at the correct time, based on chromatographic elution of the compounds. This can be setup by analyzing a standard using a full scan analysis and using the GC conditions of the SIM analysis. This analysis will give retention time and spectral information for

determining the location of start times for the SIM groups or windows. This is often referred to as a "locator" analysis.

11.3.2. Select the dwell times to be used for each group of ions to be monitored. Dwell times should be selected in order to provide a sufficient number of measurements across the chromatographic peak to accurately define the peak shape. Too few measurements across the peak will result in poor definition of the peak and subsequently result in poor accuracy and precision of results. Too many measurements across the peak may result in inconsistent detector behavior over the calibration range. Significant differences in dwell times may also affect sensitivity. Typical dwell times are listed in section 11.2.

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11.4. Initial Calibration

Refer to the SOP for *Calibration of Instruments for Organics Chromatographic Analysis* (SOC-CAL) for general calibration procedures. The calibration procedure and options chosen must follow SOP SOC-CAL and this SOP. In general, the calibration procedure is as follows:

11.4.1. 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:

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- 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 elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tune peak or part of any other peak eluting close to the tune peak.
- 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 tune peak or part of any other peak eluting close to the tune 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 tune peak or part of any other peak eluting close to the tune 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 tune peak or part of any other peak eluting close to the tune peak.

11.4.2. Evaluate the spectrum obtained for DFTPP against the tuning criteria in Table 4 or 4A (dependent upon instrumentation). The GC/MS must meet the DFTPP ion abundance criteria prior to further analyses. Pentachlorophenol must be present at the normal response, with no visible peak tailing, as demonstrated by the peak tailing factors.

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The acceptance criteria for the peak tailing factor for pentachlorophenol is < 5.0. If excessive tailing or poor chromatography 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 cannot be met, the source may need cleaning, filaments replaced or other maintenance.

11.4.3. 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 3 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 2). If interferences are noted, use the next most intense ion as the quantitaion ion (i.e. for acenaphthene-d_{10}, use 162 m/z for quantitation).

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11.4.4. Analyze 5.0 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 2). 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).

11.4.5. A system performance check must be performed to ensure that minimum average RFs are met before the calibration curve is used. The minimum acceptable average

RF for these compounds is 0.10. If they are not acceptable, perform GC maintenance (see section 9).

11.4.6. The percent relative standard deviation (%RSD) should be less than 15% for each compound. 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.
RF = mean of 5 initial RFs for a compound.
SD = standard deviation of average RFs for a compound.

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$$SD = \sqrt{\sum_{i=1}^{N} \frac{(RF_i - RF)^2}{N - 1}}$$

where:

RF_i = RF for each of the 5 calibration levels
N = Number of RF values (i.e., 5)

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11.4.7. 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.

11.4.8. 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:

11.4.8.1. The mean of the RSD values for all analytes in the calibration is $\leq$ 15% and the %RSD does not exceed 30% for any compound.

11.4.8.2. 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.

11.4.8.3. The data user must be supplied with a list of compounds which exceed 15% RSD and the result of the mean RSD calculation. For tier III and higher deliverables, an initial calibration summary may be used.

11.4.9. If all of the conditions in Section 11.4.8 are met, then the average response factor may be used to determine sample concentrations as described in Section 11.4.4.

11.4.10. When analysis for alkylated PAHs is to be performed, follow the calibration procedure given in Appendix A.

11.4.11. If the RSD of any target analyte is greater than 15%, refer to SOP SOC-CAL, section 11.2.3 and sections 7.5.2 and 7.5.3 in Method 8000B for additional calibration options. One of the options must be applied to initial calibration in this situation, or a new initial calibration must be performed.

11.4.12. 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. For each compound of interest, the calculated value must be ± 20% of the true value for the initial calibration to be valid.

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11.5. Continuing Calibration

11.5.1. Following an acceptable tune, a calibration standard, or standards, at mid-concentration containing all PAH analytes, and all required surrogates, must be analyzed every 12 hours during analysis.

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11.5.2. For each daily calibration, a system performance check must be made. For each compound in the daily calibration standard, a minimum response factor of 0.10 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. 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.

11.5.3. If the percent drift for each compound of interest 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 compound, corrective action must be taken. Problems similar to those listed in 11.5.2 could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new initial calibration must be generated. This criterion must be met before sample analysis begins.

Calculate the percent drift using:

$$\% \, Drift = \frac{C_I - C_c}{C_I} \times 100$$

where:

C_1 = Compound standard concentration.
C_c = Measured concentration using selected quantitation method.

11.5.4. When analysis for alkylated PAHs is to be performed, follow the calibration procedure given in Appendix A.

11.5.5. 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.

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11.5.6. 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.

11.6. GC/MS Analysis

11.6.1. Evaluate FID screen and make proper dilution (See SOP SOC-SCR).

11.6.2. 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.

11.6.3. 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 0.2ng/µL of each internal standard in the extracted volume. The diluted extract must be reanalyzed.

11.6.4. Store the extracts at -10°C or less, protected from light in vials equipped with unpierced Teflon lined septa. Archive extract in freezer for 3 months, or longer if required by client, after analysis in the instrument/date specific storage boxes.

12. QA/QC REQUIREMENTS

12.1. 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 analysis of samples begin, or whenever significant changes to the procedures have been made. To do this, four deionized 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.

12.2.2. 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 (ADM-MDL)*.

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12.2.3. 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.

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. If the lab control sample (LCS) fails acceptance limits for any of the compounds, the analyst must evaluate the system and calibration. Corrective action must be taken.

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:

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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:

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$$RPD = \frac{R1 - R2}{(R1 + R2)/2} \times 100$$

Where R1 = % recovery of the MS
R2 = % recovery of the DMS

The acceptance limits for the MS/DMS 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.

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.

12.3.5. The acceptance criteria listed in Attachment A are current criteria, but are subject to change as control limits are updated.

12.3.6. Additional QA/QC measures include control charting of QC sample results.

12.3.7. Corrective action – When a data quality objective is not met, the initial corrective action will include a review of the raw data for potential calculation and/or integration errors. If this review does not correct the problem, the following corrective actions will be performed.

12.3.7.1. Method Blank – No target analyte should be detected in a method blank at or above the method reporting limit (1/2 the MRL for DoD projects). If target analytes are detected in the method blank, the sample data must be reviewed for possible laboratory contribution. Detections of target analytes greater than the MRL require a Nonconformity and Corrective Action Report (NCAR). A decision to reextract the associated samples will depend on the level of the contamination, data quality implications, and the intended use of the data. At a minimum, all positive detections in the associated samples that are not more than 20X the concentration in the blank will be qualified with a "B". Also, as part of the corrective action, the problem will be discussed with the appropriate sample prep personnel in an effort to identify the contamination source.

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12.3.7.2. Laboratory Control Sample – The analysis should include a full list LCS spike. All target analytes will be evaluated. The following cases require corrective action:

- If any analytes do not meet acceptance criteria, the analytical batch should be considered out of control for that analyte. Corrective action may include reinjection to verify the result. If the result is confirmed, a NCAR will be filed and the problem investigated to determine that cause. A decision to reextract the associated samples will depend on the data quality implications and the intended use of the data, and should involve the Project Chemist and client. If reextraction is not feasible, all reported results for that analyte will be qualified and the implications will be discussed in the case narrative.
- In cases where a result is outside the upper control criterion, corrective action is only required if that analyte was also detected in field samples. The associated samples with positive results should be reextracted. In cases where a result is outside the lower control criterion, the associated samples should be reextracted. If reextraction is not feasible, all reported results for that analyte will be qualified and the data quality implications will be discussed in the case narrative. Hoever, investigation into the cause of the failure should still be performed.

12.3.7.3. Matrix Spike and Duplicate Matrix Spike Samples – If a recovery is outside of control criteria, review the consistency between the two analyses. If the result is supported between the two analyses, the outlier can be attributed to matrix inteference. Do not reanalyze the extract. If the results do not support each other, reanalyze the extracts to verify the results. If the results confirm, review the LCS recovery and take corrective action accordingly. If the LCS recovery is acceptable, flag the matrix spike data and discuss potential data quality implications in the case narrative.

12.3.7.4. Relative Percent Difference – For MS/DMS or LCS/DLCS, no corrective action is required based on RPD data alone. However, the data should be reviewed for information that will help determine if the RPD problem is the result of a sample specific issue (e.g., the DMS was concentrated to dryness), or if the problem is representative of the entire analytical batch. When the problem is apparently universal to the batch, a NCAR will be filed and the batch will be reextracted. If results of the reextraction confirm the original analyses of the field samples, the original data is reported and the RPD problems are discussed in the case narrative. If results of the reextraction confirm a problem in the original data, only the reextracted data is reported.

12.3.7.5. Surrogates – Corrective action includes reinjection to verify the result. If the result is confirmed, a NCAR will be filed and the sample will be reextracted. If the reextraction confirms the original results are biased due to matrix interferences, report the original data. If reextraction is not feasible, the surrogate will be qualified and the data quality implications will be discussed in the case narrative.

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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 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 ions monitored in the SIM mode and typically are the two or three ions of greatest relative intensity. Compounds are identified as present when the criteria below are met.

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.

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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 the gas chromatographic peaks appear to represent more than one 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.

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13.1.6. Evaluate internal standard areas in each sample. If the area in the sample is less than 50% or greater than 200% the area of in the CCV, corrective action must be taken. Depending on the analysis, this corrective action may include reinjection or dilution of the extract followed by reinjection.

13.2. Tentatively identified compounds (TICs) cannot be reported using this method.

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. 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:

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Aqueous Samples: $Concentration\ (\mu g / L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$

Nonaqueous Samples: $Concentration\ (ug/Kg) = \frac{(Cex)\ (Vf)\ (D)}{(W)}$

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Where
Cex = Concentration in extract in ng/mL
Vf = Final volume of extract in mL
D = Dilution factor
Vs = Volume of sample extracted, liters
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.

13.5. Reporting

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 which 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.

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* (ADM-NCAR) for correct procedures for identifying and documenting such data. Procedures for applying data qualifiers are described in the SOP for *Report Generation* (ADM-RG) 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* (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the CAS Quality Assurance Manual.

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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.

18. TRAINING OUTLINE

18.1. The following items provide guidelines for training analysts.

18.1.1. Review applicable literature (method references, etc.) 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.

18.2. Following this training period, the analyst is expected to complete an Initial Precision and Recovery (IPR) study as described in Section 12. Documentation of the IPR study should be forwarded to the analyst's training file.

19. REFERENCES

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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. Exxon Valdez Spill Assessment Procedure EV89-2, Revision 2.0, June 1989.

19.3. Standard Methods Manual for Environmental Sampling and Analysis in San Francisco Bay, US Army Corps of Engineers, November 1992 Draft, Volume 2 of 3.

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19.4. CAS SOPS

19.4.1. Continuous Liquid-Liquid Extraction, EXT-3520

19.4.2. Solid Phase Extraction, EXT-3535

19.4.3. Automated Soxhlet Extraction, EXT-3541

19.4.4. Silica Gel Cleanup, SOC-3630

19.4.5. Gel Permeation Chromatography, SOC-3640

TABLE 1

Target Analytes, Method Reporting Limits, and Method Detection Limits

	WATER µg/L (ppb)		SOIL µg/Kg Dry Weight (ppb)		Tissue µg/Kg Wet Weight (ppb)	
Analytes	**MRL**	**MDL**	**MRL**	**MDL**	**MRL**	**MDL**
Naphthalene	0.02	0.004	5	0.2	5	5
2-Methylnaphthalene	0.02	0.003	5	0.09	5	5
1-Methylnaphthalene	0.02	0.003	5	0.2	5	5
Biphenyl	0.02	0.004	5	0.1	5	5
2,6-Dimethylnaphthalene	0.02	0.003	5	0.1	5	5
Acenaphthylene	0.02	0.002	5	0.08	5	4
Acenaphthene	0.02	0.002	5	0.1	5	4
Dibenzofuran	0.02	0.008	5	0.3	5	4
2,3,5-Trimethylnaphthalene	0.02	0.002	5	0.09	5	4
Fluorene	0.02	0.003	5	0.2	5	3
Dibenzothiophene	0.02	0.02	5	0.6	5	5
Phenanthrene	0.02	0.004	5	0.3	5	2
Anthracene	0.02	0.002	5	0.2	5	2
1-Methylphenanthrene	0.02	0.002	5	0.08	5	2
Fluoranthene	0.02	0.003	5	0.2	5	2
Pyrene	0.02	0.003	5	0.2	5	2
Benz(a)anthracene	0.02	0.003	5	0.2	5	2
Chrysene	0.02	0.002	5	0.2	5	3
Benzo(b)fluoranthene	0.02	0.002	5	0.2	5	3
Benzo(k)fluoranthene	0.02	0.002	5	0.2	5	3
Benzo(e)pyrene	0.02	0.002	5	0.2	5	2
Benzo(a)pyrene	0.02	0.002	5	0.1	5	3
Perylene	0.02	0.002	5	0.2	5	3
Indeno(1,2,3-cd)pyrene	0.02	0.003	5	0.2	5	2
Dibenz(a,h)anthracene	0.02	0.002	5	0.2	5	3
Benzo(g,h,i)perylene	0.02	0.004	5	0.2	5	2

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TABLE 1A
Additional Analytes
Target Analytes and Method Reporting Limits

	WATER µg/L (ppb)		SOIL µg/Kg Dry Weight (ppb)		Tissue µg/Kg Wet Weight (ppb)	
Analytes	**MRL**		**MRL**		**MRL**	
C2-Naphthalenes	0.02		5		5	
C3-Naphthalenes	0.02		5		5	
C4-Naphthalenes	0.02		5		5	
C1-Fluorenes	0.02		5		5	
C2-Fluorenes	0.02		5		5	
C3-Fluorenes	0.02		5		5	
C1-Dibenzothiophenes	0.02		5		5	
C2-Dibenzothiophenes	0.02		5		5	
C3-Dibenzothiophenes	0.02		5		5	
C1-Phenanthrenes/Anthracenes	0.02		5		5	
C2-Phenanthrenes/Anthracenes	0.02		5		5	
C3-Phenanthrenes/Anthracenes	0.02		5		5	
C4-Phenanthrenes/Anthracenes	0.02		5		5	
C1-Fluoranthenes/Pyrenes	0.02		5		5	
C2-Fluoranthenes/Pyrenes	0.02		5		5	
C3-Fluoranthenes/Pyrenes	0.02		5		5	
C1-Chrysenes	0.02		5		5	
C2-Chrysenes	0.02		5		5	
C3-Chrysenes	0.02		5		5	
C4-Chrysenes	0.02		5		5	

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TABLE 2
TARGET COMPOUNDS AND CORRESPONDING PRIMARY AND SECONDARY IONS

Compound	Approximate Retention Time (min)	Primary Ion	Secondary Ion
Naphthalene-d 8 (I.S.)	5.86	136	68
Naphthalene	5.87	128	127
2-Methylnaphthalene	6.62	141	142
1-Methyl naphthalene	6.74	141	142
Biphenyl	7.18	154	153
2,6-Dimethylnaphthalene	7.37	156	155
Acenaphthlene-d 10 (I.S.)	7.95	164	162
Acenaphthylene	7.75	152	153
Acenaphthlene	8.00	154	153
Dibenzofuran	8.22	168	139
2,3,5-Trimethylnaphthalene	8.53	170	155
Fluorene	8.70	166	165
Phenanthrene-d 10 (I.S.)	10.10	188	94
Dibenzothiophene	9.95	184	152
Phenanthrene	10.13	178	179
Anthracene	10.20	178	176
1-Methylphenanthrene	11.10	192	150
Fluoranthene	11.99	202	203
Chrysene-d 12 (I.S.)	14.51	240	236
Pyrene	12.35	202	203
Benz (a) anthracene	14.48	228	226
Chrysene	14.57	228	226
Perylene-d 12 (I.S.)	18.48	264	260
Benzo(b)fluoranthene	17.50	252	126
Benzo(k)fluoranthene	17.56	252	126
Benzo(e)pyrene	18.22	252	126
Benzo(a)pyrene	18.33	252	126
Perylene	18.52	252	126
Indeno (1,2,3-cd) pyrene	20.42	276	277
Dibenz (a, h) anthracene	20.47	278	276
Benzo(g,h,i)perylene	20.86	276	277
Fluorene-d10 (surr.)	8.67	176	175
Fluoranthene-d10 (surr.)	11.97	212	213
Terphenyl-d 14 (surr.)	12.63	244	122

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TABLE 3

SEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

Naphthalene-d8

Naphthalene
2-Methylnaphthalene
1-Methylnaphthalene
Biphenyl
2,6-Dimethylnaphthalene

Acenaphthene-d10

Acenaphthene
Acenaphthylene
Dibenzofuran
2,3,5-Trimethylnaphthalene
Fluorene
Fluorene-d_{10} (surr.)

Phenanthrene-d10

Dibenzothiophene
Phenanthrene
Anthracene
1-Methylphenanthrene
Fluoranthene
Fluoranthene-d_{10} (surr.)

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Chrysene-d12

Pyrene
Benzo(a) anthracene
Chrysene
Terphenyl-d_{14} (surr.)

Perylene-d12

Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(e)pyrene
Benzo(a)pyrene
Perylene
Indeno (1,2,3-ccd)pyrene
Dibenz(a,h)anthracene
Benzo(g,h,i)perylene

TABLE 4
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA
for **5973** GC/MS systems

Mass	Ion Abundance Criteria
51	10-80% of mass 198
68	0-2% of mass 69
70	0-2% of mass 69
127	10-80% of 198
197	0-2% of 198
198	30-100% of 442 (alternate base)
199	5-9% of 198
275	10-60% of 198
365	1-50% of 442
441	0.01-100% of 443
442	30-100% of 198 (alternate base)
443	15-24% of 442

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TABLE 4A
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA
for **5972** GC/MS systems

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Mass	Ion Abundance Criteria
51	30-80% of mass 198
68	0-2% of mass 69
69	Present
70	0-2% of mass 69
127	25-75% of 198
197	0-1% of 198
198	100% Relative abundance, Base Peak
199	5-9% of 198
275	> 0.75% of 198
365	1-50% of 442
441	0.01-99.99% of 443
442	40-110% of 198
443	15-24% of 442

TABLE 5

STANDARD PREPARATION

Internal Standard Working Standard

Parent Solution	Initial Concentration	Aliquot	Final Volume	Final Concentration	Solvent
AccuStandard Z-014J	4000 ug/mL	50 uL	10 mL	20 ug/mL	DCM

Initial Calibration Intermediate Standard

Parent Solution	Initial Concentration	Aliquot	Final Volume	Final Concentration	Solvent
Absolute PAH Mix	100 ug/mL	80 uL	2 mL	4 ug/mL	DCM
PAH Surr. Intermediate	100 ug/mL	80 uL	↓	4 ug/mL	↓

Initial Calibration Standards

Initial Cal. Intermediate Std. 4ug/mL	Internal Std Working Std 20ug/mL	Final Volume	Solvent	Final Concentration PAH	Final Concentration Int Std
5 uL	100 uL	10 mL	DCM	0.002 ug/mL	0.2 ug/mL
5 uL	50 uL	5 mL	↓	0.004 ug/mL	↓
4 uL	20 uL	2 mL	↓	0.008 ug/mL	↓
5 uL	10 uL	1 mL	↓	0.02 ug/mL	↓
25 uL	↓	↓	↓	0.1 ug/mL	↓
50 uL	↓	↓	↓	0.2 ug/mL	↓
100 uL	↓	↓	↓	0.4 ug/mL	↓
250 uL	↓	↓	↓	1.0 ug/mL	↓
400 uL	↓	↓	↓	1.6 ug/mL	↓
500uL	↓	↓	↓	2.0 ug/mL	↓

Independent Calibration Verification (ICV) Standard

Parent Solution	Initial Concentration	Aliquot	Final Volume	Final Concentration	Solvent
Cerilliant PAH Mix CSQ-4049	100 ug/mL	20 uL	5 mL	0.4 ug/mL	DCM
PAH Surr. Intermediate	100 ug/mL	20 uL	↓	0.4 ug/mL	↓
Int. Std Working Std	20 ug/mL	50 uL	↓	0.2 ug/mL	↓

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APPENDIX A

Procedure for quantifying alkylated homologs of polynuclear aromatic hydrocarbons

1. Analyze a PAH ICAL using the A_PAHK method. These acquisition parameters will acquire data for the parent PAHs as well as the homologs.

Process the data and update the calibration table. Go into EDIT COMPOUNDS which is under the INITCAL menu. Enter the peak areas from the parent compounds into the corresponding levels in the associated homologs calibration table as listed below.

PARENT	HOMOLOG
Naphthalene	C2-Naphthalenes
	C3-Naphthalenes
	C4-Naphthalenes
Fluorene	C1-Fluorenes
	C2-Fluorenes
	C3-Fluorenes
Dibenzothiophene	C1-Dibenzothiophenes
	C2-Dibenzothiophenes
	C3-Dibenzothiophenes
Phenanthrene	C1-Phenanthrenes/Anthracenes
	C2-Phenanthrenes/Anthracenes
	C3-Phenanthrenes/Anthracenes
	C4-Phenanthrenes/Anthracenes
Pyrene	C1-Fluoranthenes/Pyrenes
	C2-Fluoranthenes/Pyrenes
	C3-Fluoranthenes/Pyrenes
Chrysene	C1-Chrysenes
	C2-Chrysenes
	C3-Chrysenes
	C4-Chrysenes

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Use the same Curve Fit for the Homolog as is used for the Parent compound. Use the average RF if the %RSD is less than 20%. Use a quadratic curve if the %RSD exceeds 20%.

2. Analyze the PAH-ALKH SRM. This SRM is a petroleum crude oil which has been cleaned up using GPC and silica gel cleanup procedures. All of the homologs are present in the SRM.

3. Process the SRM data against the ICAL. Review the SRM data in QEDIT and manually integrate the homologs using the example in the SOP book as a guide. Print a graphics report for each homolog.

4. Update the calibration table using the PAH CCV. Enter the average retention time for each of the homologs into the processing method.

5. Analyze the sample extracts and process the data against the ICAL. Using the SRM data as a guide, manually integrate the homologs. Print graphics reports for each sample hit.

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Attachment A
QC Acceptance Criteria

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SEMIVOLATILE ORGANICS ANALYSES						
Method	Prep Method	Matrix	Analyte	LCS Accuracy (% Rec.)	Matrix Spike (% Rec.)	Precision (RPD)
8270-SIM	3541	Soil	1,2,4-Trichlorobenzene	70-130	70-130	40
8270-SIM	3541	Soil	1,2-Dichlorobenzene	70-130	70-130	40
8270-SIM	3541	Soil	1,3-Dichlorobenzene	70-130	70-130	40
8270-SIM	3541	Soil	1,4-Dichlorobenzene	70-130	70-130	40
8270-SIM	3541	Soil	1-Methylnaphthalene	46-119	23-121	40
8270-SIM	3541	Soil	1-Methylphenanthrene	59-118	52-114	40
8270-SIM	3541	Soil	2,3,4,5-Tetrachlorophenol	44-134	48-141	40
8270-SIM	3541	Soil	2,3,4,6-Tetrachlorophenol	46-121	70-130	40
8270-SIM	3541	Soil	2,3,4-Trichlorophenol	49-131	35-155	40
8270-SIM	3541	Soil	2,3,5,6-Tetrachlorophenol	40-135	27-168	40
8270-SIM	3541	Soil	2,3,5-Trichlorophenol	45-134	26-166	40
8270-SIM	3541	Soil	2,3,5-Trimethylnaphthalene	53-116	47-105	40
8270-SIM	3541	Soil	2,3,6-Trichlorophenol	46-134	25-165	40
8270-SIM	3541	Soil	2,4,5-Trichlorophenol	39-131	59-118	40
8270-SIM	3541	Soil	2,4,6-Trichlorophenol	39-132	53-121	40
8270-SIM	3541	Soil	2,4-Dichlorophenol	70-130	70-130	40
8270-SIM	3541	Soil	2,4-Dimethylphenol	70-130	70-130	40
8270-SIM	3541	Soil	2,4-Dinitrophenol	70-130	70-130	40
8270-SIM	3541	Soil	2,4-Dinitrotoluene	70-130	70-130	40
8270-SIM	3541	Soil	2,6-Dimethylnaphthalene	36-136	36-107	40
8270-SIM	3541	Soil	2,6-Dinitrotoluene	70-130	70-130	40
8270-SIM	3541	Soil	2-Chloronaphthalene	70-130	70-130	40
8270-SIM	3541	Soil	2-Chlorophenol	70-130	70-130	40
8270-SIM	3541	Soil	2-Methyl-4,6-dinitrophenol	70-130	70-130	40
8270-SIM	3541	Soil	2-Methylnaphthalene	42-121	13-126	40
8270-SIM	3541	Soil	2-Methylphenol	70-130	70-130	40
8270-SIM	3541	Soil	2-Nitroaniline	70-130	70-130	40
8270-SIM	3541	Soil	2-Nitrophenol	70-130	70-130	40
8270-SIM	3541	Soil	3,3'-Dichlorobenzidine	70-130	70-130	40
8270-SIM	3541	Soil	3,4,5-Trichlorophenol	10-155	10-182	40
8270-SIM	3541	Soil	3-Nitroaniline	70-130	70-130	40
8270-SIM	3541	Soil	4-Bromophenyl Phenyl Ether	70-130	70-130	40
8270-SIM	3541	Soil	4-Chloro-3-methylphenol	70-130	70-130	40
8270-SIM	3541	Soil	4-Chloroaniline	70-130	70-130	40
8270-SIM	3541	Soil	4-Chlorophenyl Phenyl Ether	70-130	70-130	40
8270-SIM	3541	Soil	4-Methylphenol	70-130	70-130	40
8270-SIM	3541	Soil	4-Nitroaniline	70-130	70-130	40
8270-SIM	3541	Soil	4-Nitrophenol	70-130	70-130	40
8270-SIM	3541	Soil	Acenaphthene	50-110	18-125	40
8270-SIM	3541	Soil	Acenaphthylene	50-111	21-121	40
8270-SIM	3541	Soil	Aniline	70-130	70-130	40
8270-SIM	3541	Soil	Anthracene	52-115	19-133	40
8270-SIM	3541	Soil	Benz(a)anthracene	51-118	12-139	40
8270-SIM	3541	Soil	Benzo(a)pyrene	56-122	10-148	40
8270-SIM	3541	Soil	Benzo(b)fluoranthene	55-125	12-144	40
8270-SIM	3541	Soil	Benzo(e)pyrene	61-117	22-131	40
8270-SIM	3541	Soil	Benzo(g,h,i)perylene	49-125	10-148	40
8270-SIM	3541	Soil	Benzo(k)fluoranthene	55-124	11-145	40
8270-SIM	3541	Soil	Biphenyl	40-130	18-131	40
8270-SIM	3541	Soil	Bis(2-chloroethoxy)methane	70-130	70-130	40
8270-SIM	3541	Soil	Bis(2-chloroethyl) Ether	70-130	70-130	40
8270-SIM	3541	Soil	Bis(2-chloroisopropyl) Ether	70-130	70-130	40
8270-SIM	3541	Soil	Bis(2-ethylhexyl) Phthalate	70-130	70-130	40
8270-SIM	3541	Soil	Butyl Benzyl Phthalate	70-130	70-130	40
8270-SIM	3541	Soil	Carbazole	10-138	14-177	40
8270-SIM	3541	Soil	Chrysene	54-120	12-145	40
8270-SIM	3541	Soil	Di-n-butyl Phthalate	70-130	70-130	40
8270-SIM	3541	Soil	Di-n-octyl Phthalate	70-130	70-130	40
8270-SIM	3541	Soil	Dibenz(a,h)anthracene	37-135	12-143	40
8270-SIM	3541	Soil	Dibenzofuran	50-115	21-126	40
8270-SIM	3541	Soil	Dibenzothiophene	34-113	10-127	40
8270-SIM	3541	Soil	Diethyl Phthalate	70-130	70-130	40
8270-SIM	3541	Soil	Dimethyl Phthalate	70-130	70-130	40

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SEMIVOLATILE ORGANICS ANALYSES							
Method	Prep Method	Matrix	Analyte		LCS Accuracy (% Rec.)	Matrix Spike (% Rec.)	Precision (RPD)
8270-SIM	3541	Soil	Fluoranthene		55-121	10-149	40
8270-SIM	3541	Soil	Fluorene		52-112	22-125	40
8270-SIM	3541	Soil	Hexachlorobenzene		70-130	70-130	40
8270-SIM	3541	Soil	Hexachlorobutadiene		70-130	70-130	40
8270-SIM	3541	Soil	Hexachlorocyclopentadiene		70-130	70-130	40
8270-SIM	3541	Soil	Hexachloroethane		70-130	70-130	40
8270-SIM	3541	Soil	Indeno(1,2,3-cd)pyrene		42-133	10-151	40
8270-SIM	3541	Soil	Isophorone		70-130	70-130	40
8270-SIM	3541	Soil	N-Nitrosodi-n-propylamine		70-130	70-130	40
8270-SIM	3541	Soil	N-Nitrosodimethylamine		70-130	70-130	40
8270-SIM	3541	Soil	N-Nitrosodiphenylamine		70-130	70-130	40
8270-SIM	3541	Soil	Naphthalene		48-107	10-121	40
8270-SIM	3541	Soil	Nitrobenzene		70-130	70-130	40
8270-SIM	3541	Soil	Pentachlorophenol		10-126	10-144	40
8270-SIM	3541	Soil	Perylene		62-118	25-129	40
8270-SIM	3541	Soil	Phenanthrene		53-112	10-143	40
8270-SIM	3541	Soil	Phenol		70-130	70-130	40
8270-SIM	3541	Soil	Pyrene		47-129	10-150	40
8270-SIM	3541	Soil	2,4,6-Tribromophenol (Surr.)	10-181	NA	NA	NA
8270-SIM	3541	Soil	2-Fluorobiphenyl (Surr.)	23-127	NA	NA	NA
8270-SIM	3541	Soil	2-Fluorophenol (Surr.)	10-94	NA	NA	NA
8270-SIM	3541	Soil	Fluoranthene-d10 (Surr.)	10-136	NA	NA	NA
8270-SIM	3541	Soil	Fluorene-d10 (Surr.)	10-123	NA	NA	NA
8270-SIM	3541	Soil	Nitrobenzene-d5 (Surr.)	10-130	NA	NA	NA
8270-SIM	3541	Soil	Phenol-d6 (Surr.)	21-130	NA	NA	NA
8270-SIM	3541	Soil	Terphenyl-d14 (Surr.)	32-123	NA	NA	NA
8270-SIM	3510C/20C/35	Water	1,4-Dioxane		45-103	40-104	30
8270-SIM	3510C/20C/35	Water	1-Methylnaphthalene		28-135	27-154	30
8270-SIM	3510C/20C/35	Water	1-Methylphenanthrene		24-161	19-172	30
8270-SIM	3510C/20C/35	Water	2,3,4,5-Tetrachlorophenol		39-152	70-130	30
8270-SIM	3510C/20C/35	Water	2,3,4,6-Tetrachlorophenol		10-251	70-130	30
8270-SIM	3510C/20C/35	Water	2,3,4-Trichlorophenol		66-120	70-130	30
8270-SIM	3510C/20C/35	Water	2,3,5,6-Tetrachlorophenol		31-128	70-130	30
8270-SIM	3510C/20C/35	Water	2,3,5-Trichlorophenol		65-119	70-130	30
8270-SIM	3510C/20C/35	Water	2,3,5-Trimethylnaphthalene		23-155	34-149	30
8270-SIM	3510C/20C/35	Water	2,3,6-Trichlorophenol		63-122	70-130	30
8270-SIM	3510C/20C/35	Water	2,4,5-Trichlorophenol		53-135	70-130	30
8270-SIM	3510C/20C/35	Water	2,4,6-Trichlorophenol		54-132	70-130	30
8270-SIM	3510C/20C/35	Water	2,4-Dichlorophenol		70-130	70-130	30
8270-SIM	3510C/20C/35	Water	2,4-Dimethylphenol		70-130	70-130	30
8270-SIM	3510C/20C/35	Water	2,4-Dinitrophenol		70-130	70-130	30
8270-SIM	3510C/20C/35	Water	2,6-Dimethylnaphthalene		10-154	22-165	30
8270-SIM	3510C/20C/35	Water	2-Chlorophenol		70-130	70-130	30
8270-SIM	3510C/20C/35	Water	2-Methyl-4,6-dinitrophenol		70-130	70-130	30
8270-SIM	3510C/20C/35	Water	2-Methylnaphthalene		23-141	28-134	30
8270-SIM	3510C/20C/35	Water	2-Methylphenol		70-130	70-130	30
8270-SIM	3510C/20C/35	Water	2-Nitrophenol		70-130	70-130	30
8270-SIM	3510C/20C/35	Water	3,4,5-Trichlorophenol		66-125	70-130	30
8270-SIM	3510C/20C/35	Water	3-Methylcholanthrene		70-130	70-130	30
8270-SIM	3510C/20C/35	Water	4-Chloro-3-methylphenol		70-130	70-130	30
8270-SIM	3510C/20C/35	Water	4-Methylphenol		70-130	70-130	30
8270-SIM	3510C/20C/35	Water	4-Nitrophenol		70-130	70-130	30
8270-SIM	3510C/20C/35	Water	7,12-Dimethylbenz(a)anthracene		70-130	70-130	30
8270-SIM	3510C/20C/35	Water	Acenaphthene		41-135	33-135	30
8270-SIM	3510C/20C/35	Water	Acenaphthylene		40-138	28-143	30
8270-SIM	3510C/20C/35	Water	Anthracene		36-139	24-139	30
8270-SIM	3510C/20C/35	Water	Benz(a)anthracene		46-136	24-142	30
8270-SIM	3510C/20C/35	Water	Benzo(a)pyrene		43-138	10-153	30
8270-SIM	3510C/20C/35	Water	Benzo(b)fluoranthene		53-139	15-156	30
8270-SIM	3510C/20C/35	Water	Benzo(e)pyrene		28-160	16-152	30
8270-SIM	3510C/20C/35	Water	Benzo(g,h,i)perylene		42-146	19-150	30
8270-SIM	3510C/20C/35	Water	Benzo(k)fluoranthene		53-140	14-153	30
8270-SIM	3510C/20C/35	Water	Benzoic Acid		70-130	70-130	30

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SEMIVOLATILE ORGANICS ANALYSES							
Method	Prep Method	Matrix	Analyte		LCS Accuracy (% Rec.)	Matrix Spike (% Rec.)	Precision (RPD)
8270-SIM	3510C/20C/35	Water	Biphenyl		10-178	10-180	30
8270-SIM	3510C/20C/35	Water	Bis(2-ethylhexyl) Phthalate		10-255	70-130	30
8270-SIM	3510C/20C/35	Water	Carbazole		10-213	44-153	30
8270-SIM	3510C/20C/35	Water	Chrysene		51-134	23-144	30
8270-SIM	3510C/20C/35	Water	Dibenz(a,h)anthracene		37-148	12-162	30
8270-SIM	3510C/20C/35	Water	Dibenzofuran		10-183	30-150	30
8270-SIM	3510C/20C/35	Water	Dibenzothiophene		10-206	70-130	30
8270-SIM	3510C/20C/35	Water	Diphenyl Ether		58-130	70-130	30
8270-SIM	3510C/20C/35	Water	Fluoranthene		43-148	16-161	30
8270-SIM	3510C/20C/35	Water	Fluorene		43-139	31-151	30
8270-SIM	3510C/20C/35	Water	Indeno(1,2,3-cd)pyrene		40-146	10-167	30
8270-SIM	3510C/20C/35	Water	N-Nitrosodimethylamine		30-182	70-130	30
8270-SIM	3510C/20C/35	Water	Naphthalene		35-133	26-132	30
8270-SIM	3510C/20C/35	Water	Pentachlorophenol		10-151	10-203	30
8270-SIM	3510C/20C/35	Water	Perylene		17-161	70-130	30
8270-SIM	3510C/20C/35	Water	Phenanthrene		45-138	38-137	30
8270-SIM	3510C/20C/35	Water	Phenol		19-171	70-130	30
8270-SIM	3510C/20C/35	Water	Pyrene		37-154	18-155	30
8270-SIM	3510C/20C/35	Water	Quinoline		53-124	69-133	30
8270-SIM	3510C/20C/35	Water	1,4-Dioxine-d8 (Surr.)	40-124	NA	NA	NA
8270-SIM	3510C/20C/35	Water	2,4,6-Tribromophenol (Surr.)	10-189	NA	NA	NA
8270-SIM	3510C/20C/35	Water	2-Fluorobiphenyl (Surr.)	32-151	NA	NA	NA
8270-SIM	3510C/20C/35	Water	2-Fluorophenol (Surr.)	10-138	NA	NA	NA
8270-SIM	3510C/20C/35	Water	Fluoranthene-d10 (Surr.)	28-150	NA	NA	NA
8270-SIM	3510C/20C/35	Water	Fluorene-d10 (Surr.)	26-131	NA	NA	NA
8270-SIM	3510C/20C/35	Water	Nitrobenzene-d5 (Surr.)	36-174	NA	NA	NA
8270-SIM	3510C/20C/35	Water	Phenol-d6 (Surr.)	28-183	NA	NA	NA
8270-SIM	3510C/20C/35	Water	Terphenyl-d14 (Surr.)	32-157	NA	NA	NA
8270-SIM	3541	Tissue	1,2,4-Trichlorobenzene		41-114	59-109	40
8270-SIM	3541	Tissue	1,2-Dichlorobenzene		41-117	51-106	40
8270-SIM	3541	Tissue	1,3-Dichlorobenzene		41-112	43-104	40
8270-SIM	3541	Tissue	1,4-Dichlorobenzene		41-109	45-101	40
8270-SIM	3541	Tissue	1-Methylnaphthalene		10-174	46-104	40
8270-SIM	3541	Tissue	1-Methylphenanthrene		12-187	70-130	40
8270-SIM	3541	Tissue	2,3,5-Trimethylnaphthalene		10-184	70-130	40
8270-SIM	3541	Tissue	2,4,5-Trichlorophenol		25-159	81-132	40
8270-SIM	3541	Tissue	2,4,6-Trichlorophenol		30-154	72-137	40
8270-SIM	3541	Tissue	2,4-Dichlorophenol		50-124	73-140	40
8270-SIM	3541	Tissue	2,4-Dimethylphenol		10-107	56-145	40
8270-SIM	3541	Tissue	2,4-Dinitrophenol		40-166	10-197	40
8270-SIM	3541	Tissue	2,4-Dinitrotoluene		54-136	73-137	40
8270-SIM	3541	Tissue	2,6-Dimethylnaphthalene		10-186	70-130	40
8270-SIM	3541	Tissue	2,6-Dinitrotoluene		48-138	71-138	40
8270-SIM	3541	Tissue	2-Chloronaphthalene		39-133	37-137	40
8270-SIM	3541	Tissue	2-Chlorophenol		47-118	71-115	40
8270-SIM	3541	Tissue	2-Methyl-4,6-dinitrophenol		42-156	49-148	40
8270-SIM	3541	Tissue	2-Methylnaphthalene		12-159	39-116	40
8270-SIM	3541	Tissue	2-Methylphenol		45-113	66-123	40
8270-SIM	3541	Tissue	2-Nitroaniline		47-122	62-131	40
8270-SIM	3541	Tissue	2-Nitrophenol		46-125	52-163	40
8270-SIM	3541	Tissue	3,3'-Dichlorobenzidine		35-135	70-130	40
8270-SIM	3541	Tissue	3-Nitroaniline		50-121	10-122	40
8270-SIM	3541	Tissue	4-Bromophenyl Phenyl Ether		57-123	66-119	40
8270-SIM	3541	Tissue	4-Chloro-3-methylphenol		54-124	68-161	40
8270-SIM	3541	Tissue	4-Chloroaniline		39-95	10-104	40
8270-SIM	3541	Tissue	4-Chlorophenyl Phenyl Ether		58-115	63-123	40
8270-SIM	3541	Tissue	4-Methylphenol		45-115	60-134	40
8270-SIM	3541	Tissue	4-Nitroaniline		51-134	10-122	40
8270-SIM	3541	Tissue	4-Nitrophenol		48-139	49-161	40
8270-SIM	3541	Tissue	Acenaphthene		44-120	52-112	40
8270-SIM	3541	Tissue	Acenaphthylene		44-124	44-126	40
8270-SIM	3541	Tissue	Acetophenone		10-137	70-130	40
8270-SIM	3541	Tissue	Aniline		10-78	70-130	40

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SEMIVOLATILE ORGANICS ANALYSES							
Method	Prep Method	Matrix	Analyte		LCS Accuracy (% Rec.)	Matrix Spike (% Rec.)	Precision (RPD)
8270-SIM	3541	Tissue	Anthracene		47-128	51-121	40
8270-SIM	3541	Tissue	Atrazine		48-143	70-130	40
8270-SIM	3541	Tissue	Azobenzene		13-143	70-130	40
8270-SIM	3541	Tissue	Benz(a)anthracene		51-135	49-127	40
8270-SIM	3541	Tissue	Benzaldehyde		10-113	70-130	40
8270-SIM	3541	Tissue	Benzo(a)pyrene		49-144	50-126	40
8270-SIM	3541	Tissue	Benzo(b)fluoranthene		54-138	46-124	40
8270-SIM	3541	Tissue	Benzo(e)pyrene		23-184	70-130	40
8270-SIM	3541	Tissue	Benzo(g,h,i)perylene		45-138	46-124	40
8270-SIM	3541	Tissue	Benzo(k)fluoranthene		55-141	51-126	40
8270-SIM	3541	Tissue	Benzoic Acid		10-214	10-214	40
8270-SIM	3541	Tissue	Benzyl Alcohol		30-122	46-133	40
8270-SIM	3541	Tissue	Biphenyl		12-172	70-130	40
8270-SIM	3541	Tissue	Bis(2-chloroethoxy)methane		46-116	56-124	40
8270-SIM	3541	Tissue	Bis(2-chloroethyl) Ether		52-109	56-112	40
8270-SIM	3541	Tissue	Bis(2-chloroisopropyl) Ether		46-112	42-118	40
8270-SIM	3541	Tissue	Bis(2-ethylhexyl) Phthalate		39-174	53-172	40
8270-SIM	3541	Tissue	Butyl Benzyl Phthalate		56-145	39-171	40
8270-SIM	3541	Tissue	Caprolactam		31-156	70-130	40
8270-SIM	3541	Tissue	Carbazole		10-171	61-127	40
8270-SIM	3541	Tissue	Chrysene		59-132	59-123	40
8270-SIM	3541	Tissue	Di-n-butyl Phthalate		60-146	47-161	40
8270-SIM	3541	Tissue	Di-n-octyl Phthalate		49-156	35-184	40
8270-SIM	3541	Tissue	Dibenz(a,h)anthracene		36-155	42-144	40
8270-SIM	3541	Tissue	Dibenzofuran		11-168	57-115	40
8270-SIM	3541	Tissue	Dibenzothiophene		10-168	70-130	40
8270-SIM	3541	Tissue	Diethyl Phthalate		61-133	60-139	40
8270-SIM	3541	Tissue	Dimethyl Phthalate		60-120	68-124	40
8270-SIM	3541	Tissue	Fluoranthene		47-139	49-130	40
8270-SIM	3541	Tissue	Fluorene		49-120	53-120	40
8270-SIM	3541	Tissue	Hexachlorobenzene		59-124	62-121	40
8270-SIM	3541	Tissue	Hexachlorobutadiene		51-105	43-114	40
8270-SIM	3541	Tissue	Hexachlorocyclopentadiene		19-123	10-105	40
8270-SIM	3541	Tissue	Hexachloroethane		51-110	29-116	40
8270-SIM	3541	Tissue	Indeno(1,2,3-cd)pyrene		37-148	30-148	40
8270-SIM	3541	Tissue	Isophorone		54-127	61-147	40
8270-SIM	3541	Tissue	N-Nitrosodi-n-propylamine		42-132	48-139	40
8270-SIM	3541	Tissue	N-Nitrosodimethylamine		24-115	55-111	40
8270-SIM	3541	Tissue	N-Nitrosodiphenylamine		58-132	71-132	40
8270-SIM	3541	Tissue	Naphthalene		42-116	30-112	40
8270-SIM	3541	Tissue	Nitrobenzene		48-117	54-119	40
8270-SIM	3541	Tissue	Pentachlorophenol		27-160	10-181	40
8270-SIM	3541	Tissue	Perylene		19-189	70-130	40
8270-SIM	3541	Tissue	Phenanthrene		49-121	58-108	40
8270-SIM	3541	Tissue	Phenol		52-118	55-130	40
8270-SIM	3541	Tissue	Pyrene		52-129	58-110	40
8270-SIM	3541	Tissue	Pyridine		12-90	70-130	40
8270-SIM	3541	Tissue	2,4,6-Tribromophenol (Surr.)	47-152	NA	NA	NA
8270-SIM	3541	Tissue	2-Fluorobiphenyl (Surr.)	43-133	NA	NA	NA
8270-SIM	3541	Tissue	2-Fluorophenol (Surr.)	41-112	NA	NA	NA
8270-SIM	3541	Tissue	Fluoranthene-d10 (Surr.)	48-108	NA	NA	NA
8270-SIM	3541	Tissue	Fluorene-d10 (Surr.)	40-97	NA	NA	NA
8270-SIM	3541	Tissue	Nitrobenzene-d5 (Surr.)	35-128	NA	NA	NA
8270-SIM	3541	Tissue	Phenol-d6 (Surr.)	43-133	NA	NA	NA
8270-SIM	3541	Tissue	Terphenyl-d14 (Surr.)	49-137	NA	NA	NA
8310	3550B	Soil	2-Methylnaphthalene		10-170	70-130	40
8310	3550B	Soil	Acenaphthene		47-96	30-101	40
8310	3550B	Soil	Acenaphthylene		54-97	10-148	40
8310	3550B	Soil	Anthracene		50-93	29-119	40
8310	3550B	Soil	Benz(a)anthracene		70-103	14-146	40
8310	3550B	Soil	Benzo(a)pyrene		45-103	10-156	40

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SEMIVOLATILE ORGANICS ANALYSES							
Method	Prep Method	Matrix	Analyte		LCS Accuracy (% Rec.)	Matrix Spike (% Rec.)	Precision (RPD)
8310	3550B	Soil	Benzo(b)fluoranthene		75-105	10-157	40
8310	3550B	Soil	Benzo(g,h,i)perylene		71-109	10-173	40
8310	3550B	Soil	Benzo(k)fluoranthene		79-102	10-163	40
8310	3550B	Soil	Chrysene		75-103	10-193	40
8310	3550B	Soil	Dibenz(a,h)anthracene		61-123	10-178	40
8310	3550B	Soil	Fluoranthene		54-118	10-173	40
8310	3550B	Soil	Fluorene		48-96	20-124	40
8310	3550B	Soil	Indeno(1,2,3-cd)pyrene		73-106	10-173	40
8310	3550B	Soil	Naphthalene		42-98	10-127	40
8310	3550B	Soil	Phenanthrene		41-118	31-130	40
8310	3550B	Soil	Pyrene		43-129	10-153	40
8310	3550B	Soil	*p*-Terphenyl (Surr.)	42-139	NA	NA	NA
8310	3510C	Water	2-Methylnaphthalene/Dibenzofuran		65-100	70-130	30
8310	3510C	Water	Acenaphthene		70-100	54-105	30
8310	3510C	Water	Acenaphthylene		70-96	41-112	30
8310	3510C	Water	Anthracene		74-107	61-113	30
8310	3510C	Water	Benz(a)anthracene		77-109	62-119	30
8310	3510C	Water	Benzo(a)pyrene		73-106	61-116	30
8310	3510C	Water	Benzo(b)fluoranthene		77-109	48-135	30
8310	3510C	Water	Benzo(g,h,i)perylene		55-130	59-121	30
8310	3510C	Water	Benzo(k)fluoranthene		78-110	66-113	30
8310	3510C	Water	Chrysene		74-108	55-122	30
8310	3510C	Water	Dibenz(a,h)anthracene		52-129	54-130	30
8310	3510C	Water	Fluoranthene		74-104	47-135	30
8310	3510C	Water	Fluorene		70-100	36-132	30
8310	3510C	Water	Indeno(1,2,3-cd)pyrene		43-137	54-116	30
8310	3510C	Water	Naphthalene		65-101	53-109	30
8310	3510C	Water	Phenanthrene		74-106	58-116	30
8310	3510C	Water	Pyrene		69-117	34-150	30
8310	3510C	Water	p-Terphenyl (Surr.)	42-137	NA	NA	NA
8315	Method	Soil	Acetaldehyde		46-125	34-148	40
8315	Method	Soil	Formaldehyde		39-153	39-153	40
8315	Method	Water	Acetaldehyde		10-168	17-165	30
8315	Method	Water	Formaldehyde		39-153	39-153	30
8330	Method	Soil	1,3,5-Trinitrobenzene		75-116	64-122	11
8330	Method	Soil	1,3-Dinitrobenzene		73-119	65-124	11
8330	Method	Soil	2,4,6-Trinitrotoluene		74-118	67-122	12
8330	Method	Soil	2,4-Dinitrotoluene		72-121	62-129	12
8330	Method	Soil	2,6-Dinitrotoluene		69-115	64-121	13
8330	Method	Soil	2-Amino-4,6-dinitrotoluene		50-147	55-138	12
8330	Method	Soil	2-Nitrotoluene		73-116	63-123	11
8330	Method	Soil	3-Nitrotoluene		71-118	61-125	17
8330	Method	Soil	4-Amino-2,6-dinitrotoluene		53-142	60-129	12
8330	Method	Soil	4-Nitrotoluene		77-110	67-119	11
8330	Method	Soil	HMX		74-119	62-125	14
8330	Method	Soil	Nitrobenzene		70-119	62-124	12
8330	Method	Soil	RDX		69-121	57-127	13
8330	Method	Soil	Tetryl		37-150	14-156	29
8330	Method	Soil	1-Chloro-3-nitrobenzene (Surr.)	61-124	NA	NA	NA
8330	Method	Water	1,3,5-Trinitrobenzene		71-108	56-125	14
8330	Method	Water	1,3-Dinitrobenzene		72-110	51-140	11
8330	Method	Water	2,4,6-Trinitrotoluene		73-111	57-134	12
8330	Method	Water	2,4-Dinitrotoluene		72-113	53-143	13
8330	Method	Water	2,6-Dinitrotoluene		69-111	43-145	14
8330	Method	Water	2-Amino-4,6-dinitrotoluene		68-114	49-142	14
8330	Method	Water	2-Nitrotoluene		49-112	40-144	52
8330	Method	Water	3-Nitrotoluene		52-114	43-142	45
8330	Method	Water	4-Amino-2,6-dinitrotoluene		72-111	48-142	12
8330	Method	Water	4-Nitrotoluene		57-113	43-144	37
8330	Method	Water	HMX		66-109	47-128	13
8330	Method	Water	Nitrobenzene		53-115	42-145	44
8330	Method	Water	RDX		60-117	42-143	11
8330	Method	Water	Tetryl		55-134	49-138	14

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SEMIVOLATILE ORGANICS ANALYSES							
Method	Prep Method	Matrix	Analyte		LCS Accuracy (% Rec.)	Matrix Spike (% Rec.)	Precision (RPD)
8330	Method	Water	1-Chloro-3-nitrobenzene (Surr.)	55-112	NA	NA	NA
8330	Method	Tissue	1,3,5-Trinitrobenzene		70-130	70-130	40
8330	Method	Tissue	1,3-Dinitrobenzene		70-130	70-130	40
8330	Method	Tissue	2,4,6-Trinitrotoluene		70-130	70-130	40
8330	Method	Tissue	2,4-Dinitrotoluene		70-130	70-130	40
8330	Method	Tissue	2,6-Dinitrotoluene		70-130	70-130	40
8330	Method	Tissue	2-Amino-4,6-dinitrotoluene		70-130	70-130	40
8330	Method	Tissue	2-Nitrotoluene		70-130	70-130	40
8330	Method	Tissue	3-Nitrotoluene		70-130	70-130	40
8330	Method	Tissue	4-Amino-2,6-dinitrotoluene		70-130	70-130	40
8330	Method	Tissue	4-Nitrotoluene		70-130	70-130	40
8330	Method	Tissue	HMX		70-130	70-130	40
8330	Method	Tissue	Nitrobenzene		70-130	70-130	40
8330	Method	Tissue	RDX		70-130	70-130	40
8330	Method	Tissue	Tetryl		70-130	70-130	40
8330	Method	Tissue	1-Chloro-3-nitrobenzene (Surr.)	83-112	NA	NA	NA
8330M	Method	Soil	Picric Acid		70-130	70-130	40
8330M	Method	Soil	2,6-Dinitro-4-methylphenol (Surr.)	51-126	NA	NA	NA
8330M	Method	Water	Picric Acid		70-130	70-130	30
8330M	Method	Water	2,6-Dinitro-4-methylphenol (Surr.)	64-125	NA	NA	NA
8332	Method	Soil	Nitroglycerine		78-112	49-134	40
8332	Method	Soil	PETN		81-111	78-113	40
8332	Method	Soil	1-Chloro-3-nitrobenzene (Surr.)	79-105	NA	NA	NA
8332	Method	Water	Nitroglycerine		32-131	70-130	30
8332	Method	Water	PETN		44-127	70-130	30
8332	Method	Water	2,3-Dimethyl-2,3-dinitrobutane (Surr.)	48-109	NA	NA	NA
Organotin	Method	Soil	Dibutyltin		27-163	10-150	40
Organotin	Method	Soil	Monobutyltin		10-101	10-78	40
Organotin	Method	Soil	Tetrabutyltin		17-135	11-151	40
Organotin	Method	Soil	Tributyltin		26-131	10-146	40
Organotin	Method	Soil	Tripropyltin (Surr.)	20-121	NA	NA	NA
Organotin	Method	Water	Dibutyltin		39-127	34-127	30
Organotin	Method	Water	Monobutyltin		41-163	26-151	30
Organotin	Method	Water	Tetrabutyltin		32-112	10-99	30
Organotin	Method	Water	Tributyltin		51-119	50-120	30
Organotin	Method	Water	Tripropyltin (Surr.)	47-136	NA	NA	NA
Organotin	Method	Tissue	Dibutyltin		10-94	10-121	40
Organotin	Method	Tissue	Monobutyltin		10-115	10-113	40
Organotin	Method	Tissue	Tetrabutyltin		10-110	10-125	40
Organotin	Method	Tissue	Tributyltin		10-97	10-113	40
Organotin	Method	Tissue	Tripropyltin (Surr.)	10-114	NA	NA	NA
8015M	3550B	Soil	Diesel Range Petroleum Hydrocarbons		76-131	64-141	40
8015M	3550B	Soil	Residual Range Petroleum Hydrocarbons		66-136	49-144	40
8015M	3550B	Soil	Bromofluorobenzene (Surr.)	10-154	NA	NA	NA
8015M	3550B	Soil	o-Terphenyl (Surr.)	56-135	NA	NA	NA
8015M	3510C	Water	Diesel Range Petroleum Hydrocarbons		67-151	47-166	30
8015M	3510C	Water	Residual Range Petroleum Hydrocarbons		60-145	54-153	30
8015M	3510C	Water	Bromofluorobenzene (Surr.)	35-145	NA	NA	NA
8015M	3510C	Water	o-Terphenyl (Surr.)	39-152	NA	NA	NA
AK 102	Method	Soil	Diesel Range Petroleum Hydrocarbons		75-125	60-140	20
AK 102	Method	Soil	o-Terphenyl (Surr.)	50-150	60-120	NA	NA
AK 102	Method	Water	Diesel Range Petroleum Hydrocarbons		75-125	75-125	20
AK 102	Method	Water	o-Terphenyl (Surr.)	50-150	60-120	NA	NA
AK 103	Method	Soil	Residual Range Petroleum Hydrocarbons		60-120	60-140	20
AK 103	Method	Soil	n-Triacontane (Surr.)	50-150	60-120	NA	NA
NWTPH-Dx	Method	Soil	Diesel Range Petroleum Hydrocarbons		62-158	50-168	40
NWTPH-Dx	Method	Soil	o-Terphenyl (Surr.)	50-150	NA	NA	NA
NWTPH-Dx	Method	Water	Diesel Range Petroleum Hydrocarbons		53-161	40-164	30
NWTPH-Dx	Method	Water	o-Terphenyl (Surr.)	50-150	NA	NA	NA
NCASI 94.03	Method	Water	Methanol		70-132	49-141	30

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STANDARD OPERATING PROCEDURE

for

SAMPLE PREPARATION OF BIOLOGICAL TISSUE FOR METALS ANALYSIS BY GFAA, ICP-OES, AND ICP-MS

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SOP No.: MET-TDIG

Revision: 1

February 27, 2002

Approved by: _____ Supervisor — 2/27/02 Date

_____ QA Manager — 2-27-02 Date

_____ Laboratory Manager — 2/27/02 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: 3/29/04
Initials: _____ Date: 1/22/05
Initials: _____ Date: 01/26/06

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NUMBER: _____

Initials: _____ Date: _____

SAMPLE PREPARATION OF BIOLOGICAL TISSUE FOR METALS ANALYSIS BY GFAA, ICP-OES, AND ICP-MS

1. SCOPE AND APPLICATION

This procedure describes techniques used for sample preparation and acid digestion of biological tissue samples. This procedure is applicable to the analysis of biological tissue for heavy metals. The procedure provides a convenient and efficient digestion/dissolution technique which allows for the simultaneous or sequential analysis of the sample for metals. The digestates may be analyzed by graphite furnace atomic absorption (GFAA), Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES), or Inductively Coupled Plasma-Mass Spectrometry (ICP-MS). The procedure includes, but is not restricted to, the metals listed in Table 1.

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2. METHOD SUMMARY

A representative tissue sample is Lyophilized, blended, then sub-sampled for microwave or conventional oven digestion. Oxidation is brought about by the use of concentrated nitric acid in a Teflon closed vessel. The digestate is then analyzed for metallic constituents by GFAA, ICP-OES, or ICP-MS methods.

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3. INTERFERENCES

Refer to the determinative method for a discussion of interferences.

4. SAFETY

Nitric Acid is extremely corrosive. Care should be taken while working with this chemical. Personal protective equipment will include safety glass (with side shields), gloves and a lab coat. Follow normal precautions as per the CAS Safety Manual.

5. SAMPLE, COLLECTION, PRESERVATION AND STORAGE

Samples are typically collected in plastic containers. Sample may be refrigerated or frozen, depending on project requirements.

6. APPARATUS AND EQUIPMENT

6.1. Lyophilizing apparatus such as a LABCONCO Model 4.5 benchtop freeze dryer.

6.2. Microwave digestion apparatus including Teflon closed vessels fitted with rupture discs or equivalent pressure relief.

6.3. Conventional laboratory oven capable of precise temperature control at 105°C.

6.4. Analytical balance capable of weighing to 0.1 mg.

6.5. 50 mL graduated poly tubes.

7. STANDARDS AND REAGENTS

7.1. Reagent water - ASTM Type II water

7.2. Concentrated nitric acid.

7.3. 1,000 ppm stock metal standards for preparation of spike solution(s) to include analyte(s) of interest.

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8. 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.

9. PREVENTIVE MAINTENANCE

9.1. Routine cleaning of the sample handling and digestion apparatus is necessary. Refer to the SOP for Metals Laboratory Glassware Cleaning.

9.2. Record all maintenance and monitoring activities in a lab notebook.

10. PROCEDURE

10.1. Obtain a representative tissue sample that will yield ~ 300 mg of freeze-dried solids.

Note: This is designed to be a general guideline. Approximately 300 mg of dry sample is typically required to obtain the desired detection limits that often are necessary for tissue analysis.

10.2. Slice the sample into thin pieces prior to filling the drying vessel.

10.3. Cap the vessel and freeze the sample in a conventional freezer.

10.4. When the sample is frozen, remove from freezer and follow the manufacturer's instructions for operation of the freezer dryer.

10.5. Blend the dry solids to obtain a homogeneous sample. The sample may be stored dry until digestion.

10.6. Microwave digestion option

10.6.1. Transfer 300 mg of dried sample, weighed to the nearest 0.1 mg, to a 50 mL decomposition vessel.

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10.6.2. Add 4.5mL concentrated nitric acid to the vessel.

Note: The procedure may be continued immediately with greater chance of rupturing the safety seal on the decomposition vessel than if the sample is allowed to stand for several hours, or more, pre-digesting at room temperature. Pre-digesting can also be enhanced by use of heat lamps.

10.6.3. Follow the manufacturer's instructions for the use of the decomposition vessels.

10.6.4. Place the vessel in the carrousel and expose at 1/2 power for 5 minutes.

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10.6.5. Cool the vessel, open to relieve pressure and vent gases, then return to carrousel.

10.6.6. Note: perform this step in the hood.

10.6.7. 1 Expose at full power for 10 minutes.

10.6.8. Cool the vessel, open to relieve pressure and vent gases, then transfer to a volumetric container and dilute to 30mL. The sample is ready for analysis.

10.7. Conventional oven digestion option

10.7.1. Transfer 300 mg of dried sample, weighed to the nearest 0.1 mg, to a 50 mL decomposition vessel.

10.7.2. Add 4.5mL concentrated nitric acid to the vessel.

Note: The procedure may be continued immediately with greater chance of rupturing the safety seal on the decomposition vessel than if the sample is allowed to stand for several hours, or more, pre-digesting at room temperature. Pre-digesting can also be enhanced by use of heat lamps.

10.7.3. Follow the manufacturer's instructions for the use of the decomposition vessels. Place the vessel in a conventional oven at 105°C for a minimum of 24 hours.

10.7.4. Cool the vessel, open to relieve pressure and vent gases, then transfer to a volumetric container and dilute to 30mL. The sample is ready for analysis.

11. QA/QC REQUIREMENTS

11.1. If using the conventional oven option, monitor oven temperatures for each batch. Report all deficiencies to the Lab Manager; corrective action will be taken.

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11.2. Prepare one blank per digestion batch, or per 20 samples, or per SDG , whichever is more frequent. Analyze at least one preparation blank per group of samples digested.

11.3. Prepare duplicates at 5% frequency or one per batch, whichever is more frequent.

11.4. Prepare one laboratory control sample at 5% frequency or one per batch, whichever is more frequent.

11.5. Prepare spikes at 5% frequency or one per batch, whichever is more frequent. Spikes should be added directly to the dry sample. Spike solutions should be multi-element with analyte concentrations high enough to minimize volume added to sample. The acid matrix of the spiking solution should be 20-50% nitric acid, if possible.

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11.6. Analyze a standard reference material (SRM) at 5% frequency or one per batch, whichever is more frequent. SRM's should be representative of the tissue sample being analyzed.

12. REPORTING

12.1. Digestion data sheets, including weights and volumes used, are completed and a batch lot number is assigned and attached to the data sheet. The Manufacturer's lot number for the reagents used are added to the digestion data sheet. (See appendix A)

12.2. Spiking sheets are completed including all spike data and volumes of spiking solutions used. (See appendix A)

13. REFERENCES

Recommended Guidelines for Measuring Metals in Puget Sound Marine Water, Sediment, and Tissue Samples; April 1997.

Table 1

Selected Elements and Analysis Procedures for Tissue Samples

Element	GFAA	ICP-OES	ICP-MS
Aluminum		√	√
Antimony		√	√
Arsenic	√		√
Barium		√	√
Beryllium		√	√
Boron		√	
Cadmium	√	√	√
Calcium		√	
Chromium		√	√
Cobalt		√	√
Copper		√	√
Iron		√	
Lead	√	√	√
Lithium		√	
Magnesium		√	
Manganese		√	√
Molybdenum		√	√
Nickel		√	√
Potassium		√	
Phosphorus		√	
Selenium	√		
Silver		√	√
Sodium		√	
Strontium		√	
Thallium	√	√	√
Tin		√	√
Vanadium		√	
Zinc		√	√

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APPENDIX A

Benchsheets

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Service Request #: _____ Method: EPA METHOD 160.3 MODIFIED

Analysis For: Freeze Dried Solids

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

COLUMBIA ANALYTICAL SERVICES, INC.

Service Request #: _____

Analysis For: Freeze Dried Solids

Lab Code	Wet Weight (g)	Tare (g)	Tare + Dry Wt.(g)	Dry Weight (g)	% Total Solids

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Time In : _____ Time Out: _____

Comments:

$\bar{x}$ = RPD =

Analyst:	Date:
Reviewed By:	Date:

Columbia Analytical Services
Metals Tissue Digestion Sheet

Service Request Number(s) :

Method : Tissue

Analysis for : ICP ICP-MS GFAA
other: _____

Sample	Initial Weight (g)	freeze Dry	Wet	Final Volume (ml)	Matrix

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Time Digestion Started:_____**Oven Temp:**_____ **Time Digestion Ended:**_____
Lot # Acids Used: HNO3 _____ **Oven Temp:**_____

LCS: **Dorm-2, Dolt-3**

QCP CICV-1, MET1-40-B, _____ **mls. added**
QCP CICV-2, MET1-40-C, _____ **mls. added**
QCP CICV-3, MET1-40-D, _____**mls. Added**
SS6, MET1-46-C, _____**mls. Added**

SPIKE INFO
SS1-MET1-45-H,_____**mls added**
SS5-MET1-45-I,_____**mls added**
SS6-MET1-46-C,_____**mls added**
Additional spikes:_____
Comments:

Analyst _____ **Date** _____
Reviewer_____ **Date** _____

metdig.xls
11/11/2004

STANDARD OPERATING PROCEDURE

for

DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY (ICP)

SOP No.: MET-ICP

Revision: 19

December 20, 2007

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Approved by: _____ Supervisor _____ Date 12/20/07

_____ QA Manager _____ Date 12-20-07

_____ Laboratory Manager _____ Date 12/20/07

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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: _____
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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 and water digestates using EPA ICP methods 6010B, 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.

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 MRL for DoD projects is no less than 3x the MDL. 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 or AFCEE QAPP (as applicable), 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 a stock solution which is different from the stock used to prepare calibration standards and is 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. 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.

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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

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 MRL 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 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 Hydrofluoric acid may also be used in this procedure. 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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5.5 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.

5.6 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.

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 either stored in a 50 mL plastic centrifuge tube or in a 100 mL volumetric flask.

6.2 For Method 200.7 each time water samples are preserved after arrival at the lab, hold the samples for a minimum of 16 hours and verify the pH<2 prior to digestion.

6.3 Soil samples with greater than 60% solids 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 Digestates are brought to 3< pH<12 and disposed of through the sewer system 2 weeks after data is reviewed. Following analysis, digestates are stored until all results have been reviewed.

7 APPARATUS & EQUIPMENT

7.1 Inductively Coupled Plasma Atomic Emission Spectrometer

7.1.1 Thermo Jarrell Ash ICAP-61.

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7.1.2 Thermo Jarrell Ash IRIS.

7.2 Cross-Flow Nebulizer (TJA-61), Concentric nebulizer (IRIS)

7.3 Peristaltic Pump with Tubing Flow Rated at 1.0 cc/min

7.4 Computer system interfaced to the ICP. A compatible Windows-based data system is used to acquire, store, and perform calculations on raw data.

7.5 Argon

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8 STANDARDS, REAGENTS, & CONSUMABLE MATERIALS

8.1 All standards are prepared from NIST traceable or certified standards as per the SOP Std. Prep. Manufacturer's expiration dates are used to determine the viability of standards.

8.2 1000ppm or 10,000ppm Stock Standard Solutions (AA Grade) - Commercially available certified solutions.

8.3 Working Standardization Standards - These are prepared from a pre-mixed stock solution. Documentation for these standards is the same as above.

8.4 Standards Preparation

8.4.1 Calibration Standards

Calibration standards are prepared from commercially purchased single element 1000 ppm, 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 (IRIS and TJA61) vary based on the requirements of each instrument (e.g. the IRIS line switches for multiple elements and requires calibration at various levels while the TJA61 does not). The composition of the IRIS calibrations standards are outlined in Table 3. For the TJA61 three intermediate stock solutions are first produced. Theses are outlined in Table 4. From these stock solutions two final working calibration standards are prepared as outlined in Table 5.

8.4.2 Continuing Calibration Verification (CCV) Standards

CCV standards are analyzed at the midpoint of the calibration. For the IRIS these standards are produced by making a two-fold dilution of the 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.

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8.4.3 Initial Calibration Verification (ICV) Standards

The ICV working standards for both instruments are produced by direct dilution of three certified mixed stock solutions (QCP-CICV1, QCP-CICV2, and QCP-CICV3) purchased from Inorganic Ventures 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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8.4.4 Interference Check Solutions (ICSA & ICSAB)

The ICSA and ICSAB working standards for both instruments are produced by direct dilution of certified mixed stock solutions (CLPP-ICS-A and CLPP-ICS-B) purchased from Inorganic Ventures. 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.

8.5 QC Solutions – The solutions and materials used for the LCS and matrix spikes are described in the applicable digestion SOP.

8.6 Standard Log

8.6.1 The manufacturer, lot number, and expiration date are recorded in a standard logbook kept in the ICP lab. The operator who prepares the standard dates and initials 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.

8.6.2 The lot number and date opened for acids used are recorded in the standard log.

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. Water filters are changed 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 as needed.

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.

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9.2.2 The torch, nebulizer, and spray chamber are cleaned as necessary. These maintenance events are also documented in the maintenance logbook.

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. 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.

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.

11.2 Profile – If available, manual profile is suggested prior to standardization. Currently, manual profile is not available on the TJA61. Automatic profile may be performed if spectrum shifter position is less than 1.5. Specific directions for profile procedures are displayed on the computer screen by selecting F5-Profile from any analytical method.

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11.3 CuMn Ratio

11.3.1 The required ratio is determined by optimizing the instrument annually, or whenever a significant change in the operating conditions is experienced. A solution of 1 ppm Cu, Cd, and Mn, is aspirated into the nebulizer.

11.3.2 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.

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11.3.3 The CuMn ratio is checked daily and prior to any standardization. These data, as well as instrument parameters, are recorded in the instrument logbook.

11.3.4 Linear dynamic range (LDR) - 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 succeedingly higher standard concentrations of the analyte until the observed analyte concentration is no more than 10% 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. Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified 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.

11.4 Calibration/Standardization

11.4.1 IRIS Instrument

11.4.1.1 Plasma is ignited and instrument is allowed to warm up for at least 15 minutes.

11.4.1.2 Analyze the intensity check solution. The recoveries must be within ±20 % of the true value obtained when the torch and nebulizer were last cleaned.

11.4.1.3 The Cu/Mn ratio is taken from intensity check solution. Ratio is to remain within 20% of value when IECs were determined.

11.4.1.4 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.

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11.4.1.5 Standardize by running a Blank Standard and a High Standard for each element in the analytical method.

11.4.1.6 After the first CCB and before the ICS standards a CRI (MRL) standard is analyzed. The acceptance criteria are a detected result of ± 100%.

11.4.1.7 Control limits for other standards are as follows CCV=10, ICV=10%, CSA & ICSAB=20%.

11.4.2 61-E standardization Procedure

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11.4.2.1 Plasma is ignited and instrument is allowed to warm up for at least 30 minutes.

11.4.2.2 Instrument is profiled and Cu/Mn is analyzed. The ratio is to remain with in ±20% of the value when IECs were determined.

11.4.2.3 Standardize by running the same steps as for the IRIS in steps 11.4.1.4 – 11.4.1.6 above.

11.4.3 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 the result must be within ± 10% of the true value.

11.5 Analytical Run

11.5.1 Following standardization and ICV analysis, the remainder on the run is determined by what analytical method is being performed. These are listed below.

11.5.1.1 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.

11.5.1.2 Methods 200.7 and 6010B: ICB, CCV, CCB, MRL, ICSA, ICSAB, routine samples. The MRL is a low level standard with concentrations at the MRL. For AFCEE projects, the MRL standard concentrations will be equal to the project MRLs.

11.5.1.3 ICS solutions are to be run at the beginning of the day (sequence) and at least every 8 hours thereafter. Results for the spiked analytes in the ICSAB solution must agree with ±20% of the expected value.

11.5.2 Evaluate the control analyses using the following criteria:

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11.5.2.1 For methods 200.7 and 6010B, the following criteria apply:

- The ICV must verify within ± 5% of the true values for method 200.7 and ± 10% of the true values for method 6010B.

  - 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 MRL is a low level standard with concentrations at the MRL. Refer to project-specific criteria. For AFCEE projects, the MRL standard concentrations will be equal to the project MRLs.

- The ICSA and ICSAB criteria is ± 20%.

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11.5.2.2 For CLP, refer to SOW ILM04.0 for acceptance criteria.

11.5.2.3 If any of the ICV, ICB, CCV, CCB, MRL, ICSA, or ICSAB fail the criteria listed, re-analyze. All of these parameters must meet criteria prior to analysis.

11.5.3 At a minimum, a continuing calibration verification (CCV) and continuing calibration blank (CCB) pair are analyzed after standardization and after every 10 samples.

11.5.3.1 The CCV immediately following standardization must verify within ± 10% of the true values (± 5% for method 200.7) with a relative standard deviation of <3% from 4 replicate integrations. Calculate %RSD as follows:

$$\%RSD = \frac{StdDev_{CCV}}{Average_{CCV}} x\,100$$

where: $StdDev_{ccv}$ = Standard deviation of the 4 replicate integrations
$Average_{ccv}$ = Average of the 4 replicate CCV integrations

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11.5.3.2 Additional CCVs analyzed in the sample analysis sequence must verify within ±10% of the expected value.

11.5.3.3 The RSD for individual burns of the CCV must be ≤ 5% RPD.

11.5.3.4 CCBs are evaluated as in section 11.5.2.1.

11.5.4 The analytical run sequence is performed as follows:

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11.5.4.1 Select the correct method.

11.5.4.2 Aspirate Standard 0 (Blank) into nebulizer.

11.5.4.3 Allow to equilibrate for 1-2 minutes.

11.5.4.4 Follow directions on computer screen to perform standardization.

11.5.4.5 Operator will sign and date the first page of standardization.

11.5.4.6 Run samples in order given in section 11.5.1 and/or as specified by project requirements.

11.5.4.7 The analyst must allow adequate rinse time (typically 1 minute) between samples, as indicated by monitoring the blank value between samples. Actual time may vary depending on the nature of the particular samples.

11.5.4.8 If using the IRIS ICP, run the IRIS intensity check standard daily. Values are in intensity counts, and QC table values are set each time the nebulizer is changed. Acceptance range is ± 20%.

12 QA/QC REQUIREMENTS

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 of for each analyte must meet LCS criteria and the RSD< 30%.

12.2 Method Detection Limits

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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 starting in Section 11 to analyze the samples. Refer to the CAS SOP for The Determination of Method Detection Limits.

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.

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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 Each sample preparation batch must have a method blank associated with it. The method blank result should be < MRL. Contamination of the method blank 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 sample.

12.3.2 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, the control limits are derived from lab data and are listed in Table 2. 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.

12.3.3 A Duplicate sample is digested one per batch, or per 20 samples (i.e. 5%) for 6010B analysis, or per 10 samples (i.e. 10%) for 200.7 analysis. The default

criteria is <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. Criteria are subject to change as statistical criteria are later generated. The RPD criteria are project-specific for soil samples. If not, redigest the sample batch. Determine if the sample is non-homogenous and redigest if it is homogenous.

12.3.4 A Matrix Spike sample is digested one per batch, or per 20 samples (i.e. 5%) for 6010B analysis, or per 10 samples (i.e. 10%) for 200.7 analysis. The matrix spike recovery will be calculated while the job is in progress. Where specified by project requirements, a matrix spike duplicate may be required. Matrix spike recovery criteria for method 200.7 is 75-125% for both water and soil samples. For 6010B, the control limits are derived from lab data and are listed in Table 2. 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). Check for possible matrix interference by analyzing a serial dilution (5x). Serial dilution should be ± 10% of sample. 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.

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12.3.5 The software will generate a Q flag for data that doesn't fall within the required limits. Corrective action will be taken and demonstration of acceptability will be documented.

12.3.6 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. If the analyte concentration is too low to permit a dilution test, then a post-spike may be performed.

12.3.7 Additional QC measures include control charting and compiling of QC data for generation of control limits.

12.4 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 made available to the ICP operator.

12.5 Semi-annually, instrument interferences are calculated for all interferences as per ILM04.0 (Section E, paragraph 11) and Method 6010B (Section 3.0). 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 by the supervisor. Copies of this data are kept on file and made available.

12.6 CLP analyses are performed as per the QA/QC guidelines in the most current CLP SOW.

13 DATA REDUCTION, REVIEW, AND REPORTING

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 IRIS and Table 9 for the TJA61.

Aqueous samples are reported in µg/L:

$$\mu g / L\ (Sample) = C^{*}\ x\ Digestion\ Dilution\ Factor\ x\ Post\ Digestion\ Dilution\ Factor$$

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Solid samples are reported in mg/Kg:

$$mg / Kg\ (Sample) = C^{*}\ x\ Post\ Digestion\ Dilution\ Factor\ x\ \frac{Digestion\ Vol.\ (ml)}{Sample\ wt.\ (g)}\ x\ \frac{1mg}{1000g}\ x\ \frac{1L}{1000ml}\ x\ \frac{1000g}{1Kg}$$

C*= Concentration of analyte as measured at the instrument in mg/L (in digestate).

13.1.1 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.

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$$Hardness, mg\ equivalent CaCO_3/L = 2.497[Ca, mg/L] + 4.118[Mg, mg/L]$$

13.1.2 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.

13.2 Data Review and Reporting

13.2.1 It is the analysts 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 14. 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.

13.2.2 Tier III or Higher Deliverable

Data is reviewed by the ICP operator. A disk is generated during the course of the analytical run. The package including standards, QC sheet and run log is prepared and

included with the raw data in the work order file. The data is then processed through the MARRS software.

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 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

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This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional available method performance data.

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

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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 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 acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 5-9 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.

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 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.

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18.3 Training and proficiency is documented in accordance with the SOP ADM-TRANDOC.

19 REFERENCES

19.1 USEPA, Contract Laboratory Program, SOW #ILM04.0

19.2 Thermo Jarrell Ash ICAP61 Manual

19.3 USEPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Update III, Method 6010B, Revision 2, December 1996.

19.4 USEPA, Methods for Determination of Metals in Environmental Samples, Supplement I, EPA/600/R-94/111, Method 200.7, Revision 4.4, May 1994.

19.5 *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	DoD MRL	Standard MRL	MDL	Units	Method
Aluminum	Water	CLP	90	**50**	**30**	ug/L	6010 / 200.7
Antimony	Water	CLP	90	**50**	**30**	ug/L	6010 / 200.7
Arsenic	Water	CLP	100	**100**	**20**	ug/L	6010 / 200.7
Barium	Water	CLP	5	**5**	**0.7**	ug/L	6010 / 200.7
Beryllium	Water	CLP	5	**5**	**0.4**	ug/L	6010 / 200.7
Boron	Water	CLP	50	**50**	**9**	ug/L	6010 / 200.7
Cadmium	Water	CLP	9	**5**	**3**	ug/L	6010 / 200.7
Calcium	Water	CLP	90	**50**	**30**	ug/L	6010 / 200.7
Chromium	Water	CLP	9	**5**	**3**	ug/L	6010 / 200.7
Cobalt	Water	CLP	10	**10**	**3**	ug/L	6010 / 200.7
Copper	Water	CLP	21	**10**	7	ug/L	6010 / 200.7
Iron	Water	CLP	20	**20**	**3**	ug/L	6010 / 200.7
Lead	Water	CLP	60	**50**	**20**	ug/L	6010 / 200.7
Lithium	Water	CLP	20	**10**	**6**	ug/L	6010 / 200.7
Magnesium	Water	CLP	20	**20**	**0.7**	ug/L	6010 / 200.7
Manganese	Water	CLP	5	**5**	**0.6**	ug/L	6010 / 200.7
Molybdenum	Water	CLP	18	**10**	**6**	ug/L	6010 / 200.7
Nickel	Water	CLP	60	**20**	**20**	ug/L	6010 / 200.7
Phosphorus	Water	CLP	600	**200**	**200**	ug/L	6010 / 200.7
Potassium	Water	CLP	2700	**2000**	**900**	ug/L	6010 / 200.7
Selenium	Water	CLP	120	**100**	**40**	ug/L	6010 / 200.7
Silicon	Water	CLP	400	**400**	**40**	ug/L	6010 / 200.7
Silver	Water	CLP	18	**10**	**6**	ug/L	6010 / 200.7
Sodium	Water	CLP	150	**100**	**50**	ug/L	6010 / 200.7
Strontium	Water	CLP	10	**10**	**0.5**	ug/L	6010 / 200.7
Thallium	Water	CLP	200	**200**	**50**	ug/L	6010 / 200.7
Tin	Water	CLP	60	**50**	**20**	ug/L	6010 / 200.7
Titanium	Water	CLP	10	**10**	**3**	ug/L	6010 / 200.7
Vanadium	Water	CLP	15	**10**	**5**	ug/L	6010 / 200.7
Zinc	Water	CLP	21	**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	DoD MRL	Standard MRL	MDL	Units	Method
Aluminum	Water	3010A	150	**50**	**50**	ug/L	6010
Antimony	Water	3010A	60	**50**	**20**	ug/L	6010
Arsenic	Water	3010A	100	**100**	**30**	ug/L	6010
Barium	Water	3010A	6	**5**	**2**	ug/L	6010
Beryllium	Water	3010A	5	**5**	**0.2**	ug/L	6010
Boron	Water	3010A	90	**50**	**30**	ug/L	6010
Cadmium	Water	3010A	9	**5**	**3**	ug/L	6010
Calcium	Water	3010A	50	**50**	**10**	ug/L	6010
Chromium	Water	3010A	6	**5**	**2**	ug/L	6010
Cobalt	Water	3010A	12	**10**	**4**	ug/L	6010
Copper	Water	3010A	18	**10**	**6**	ug/L	6010
Iron	Water	3010A	20	**20**	**3**	ug/L	6010
Lead	Water	3010A	90	**50**	**30**	ug/L	6010
Lithium	Water	3010A	24	**10**	**8**	ug/L	6010
Magnesium	Water	3010A	20	**20**	**0.5**	ug/L	6010
Manganese	Water	3010A	6	**5**	**2**	ug/L	6010
Molybdenum	Water	3010A	12	**10**	**4**	ug/L	6010
Nickel	Water	3010A	60	**20**	**20**	ug/L	6010
Phosphorus	Water	3010A	270	**200**	**90**	ug/L	6010
Potassium	Water	3010A	3000	**2000**	**1000**	ug/L	6010
Selenium	Water	3010A	120	**100**	**40**	ug/L	6010
Silicon	Water	3010A	600	**400**	**200**	ug/L	6010
Silver	Water	3010A	27	**10**	**9**	ug/L	6010
Sodium	Water	3010A	180	**100**	**60**	ug/L	6010
Strontium	Water	3010A	10	**10**	**0.5**	ug/L	6010
Thallium	Water	3010A	200	**200**	**60**	ug/L	6010
Tin	Water	3010A	60	**50**	**20**	ug/L	6010
Titanium	Water	3010A	10	**10**	**2**	ug/L	6010
Vanadium	Water	3010A	12	**10**	**4**	ug/L	6010
Zinc	Water	3010A	10	**10**	**3**	ug/L	6010

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TABLE 1 continued

Selected Elements, Method Reporting Limits, and Method Detection Limits

Element	Matrix	Digestion	DoD MRL	Standard MRL	MDL	Units	Method
Aluminum	Soil	3050B	21	**10**	7	mg/Kg	6010 / 200.7
Antimony	Soil	3050B	24	**10**	**8**	mg/Kg	6010 / 200.7
Arsenic	Soil	3050B	20	**20**	**5**	mg/Kg	6010 / 200.7
Barium	Soil	3050B	2	**2**	**0.2**	mg/Kg	6010 / 200.7
Beryllium	Soil	3050B	1	**1**	**0.03**	mg/Kg	6010 / 200.7
Boron	Soil	3050B	10	**10**	**2**	mg/Kg	6010 / 200.7
Cadmium	Soil	3050B	3	**1**	**1**	mg/Kg	6010 / 200.7
Calcium	Soil	3050B	10	**10**	**2**	mg/Kg	6010 / 200.7
Chromium	Soil	3050B	2	**2**	**0.7**	mg/Kg	6010 / 200.7
Cobalt	Soil	3050B	2	**2**	**0.6**	mg/Kg	6010 / 200.7
Copper	Soil	3050B	2	**2**	**0.7**	mg/Kg	6010 / 200.7
Iron	Soil	3050B	4	**4**	**0.6**	mg/Kg	6010 / 200.7
Lead	Soil	3050B	21	**20**	7	mg/Kg	6010 / 200.7
Magnesium	Soil	3050B	6	**4**	**2**	mg/Kg	6010 / 200.7
Manganese	Soil	3050B	2	**2**	**0.07**	mg/Kg	6010 / 200.7
Molybdenum	Soil	3050B	3	**2**	**0.9**	mg/Kg	6010 / 200.7
Nickel	Soil	3050B	9	**4**	**3**	mg/Kg	6010 / 200.7
Phosphorus	Soil	3050B	60	**40**	**20**	mg/Kg	6010 / 200.7
Potassium	Soil	3050B	600	**200**	**200**	mg/Kg	6010 / 200.7
Selenium	Soil	3050B	40	**40**	**6**	mg/Kg	6010 / 200.7
Silver	Soil	3050B	6	**2**	**2**	mg/Kg	6010 / 200.7
Sodium	Soil	3050B	60	**20**	**20**	mg/Kg	6010 / 200.7
Strontium	Soil	3050B	6	**2**	**2**	mg/Kg	6010 / 200.7
Thallium	Soil	3050B	60	**40**	**20**	mg/Kg	6010 / 200.7
Tin	Soil	3050B	15	**10**	**5**	mg/Kg	6010 / 200.7
Titanium	Soil	3050B	2	**2**	**0.5**	mg/Kg	6010 / 200.7
Vanadium	Soil	3050B	6	**2**	**2**	mg/Kg	6010 / 200.7
Zinc	Soil	3050B	3	**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	DoD MRL (1)	Standard MRL (1)	MDL (1)	Units	Method
Aluminum	Tissue	PSEP	18	**10**	**6**	mg/Kg	6010
Antimony	Tissue	PSEP	12	**5**	**3**	mg/Kg	6010
Barium	Tissue	PSEP	1	**1**	**0.2**	mg/Kg	6010
Beryllium	Tissue	PSEP	1	**1**	**0.02**	mg/Kg	6010
Boron	Tissue	PSEP	5	**5**	**0.6**	mg/Kg	6010
Cadmium	Tissue	PSEP	0.9	**0.5**	**0.3**	mg/Kg	6010
Calcium	Tissue	PSEP	60	**40**	**20**	mg/Kg	6010
Chromium	Tissue	PSEP	1.8	**1**	**0.6**	mg/Kg	6010
Cobalt	Tissue	PSEP	1.6	**1**	**0.4**	mg/Kg	6010
Copper	Tissue	PSEP	1.5	**1**	**0.5**	mg/Kg	6010
Iron	Tissue	PSEP	12	**4**	**4**	mg/Kg	6010
Lead	Tissue	PSEP	12	**5**	**3**	mg/Kg	6010
Magnesium	Tissue	PSEP	12	**4**	**3**	mg/Kg	6010
Manganese	Tissue	PSEP	0.6	**0.5**	**0.2**	mg/Kg	6010
Molybdenum	Tissue	PSEP	2.1	**1**	**0.7**	mg/Kg	6010
Nickel	Tissue	PSEP	3	**2**	**1**	mg/Kg	6010
Phosphorus	Tissue	PSEP	30	**20**	**10**	mg/Kg	6010
Potassium	Tissue	PSEP	600	**200**	**200**	mg/Kg	6010
Sodium	Tissue	PSEP	24	**10**	**8**	mg/Kg	6010
Strontium	Tissue	PSEP	1	**1**	**0.2**	mg/Kg	6010
Tin	Tissue	PSEP	6	**5**	**2**	mg/Kg	6010
Vanadium	Tissue	PSEP	1.6	**1**	**0.4**	mg/Kg	6010
Zinc	Tissue	PSEP	1.6	**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 Matrial: ERA PrioityPollutnT/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

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[1] Dry Weight Basis
[2] List limits are for "Blank Spike" analysis. SRM samples have separate limits.

TABLE 3
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
Arsenic	(1)	100	10	1000	1.0
Beryllium	(1)	100	10	1000	1.0
Beryllium	(1)	100	10	1000	1.0
Boron	(1)	100	10	1000	1.0
Cadmium	(1)	100	10	1000	1.0
Calcium	(1)	100	10	1000	1.0
Chromium	(1)	100	10	1000	1.0
Coblat	(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
Magnesium	(1)	100	10	1000	1.0
Magnesium	Elemental Stock	1000	4	1000	4.0
Manganese	(1)	100	10	1000	1.0
Molybdenum	(1)	100	10	1000	1.0
Nickel	(1)	100	10	1000	1.0
Selenium	(1)	100	10	1000	1.0
Silicon	(1)	100	10	1000	1.0
Silver	(1)	100	10	1000	1.0
Tin	Elemental Stock	1000	1	1000	1.0
Thallium	(1)	100	10	1000	1.0
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) Inorganic Ventures Mixed Standard, QCS-26

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TABLE 3 - 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
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	1000	50	1000	50
Phosphorus	Elemental Stock	10000	0.5	1000	5.0
Potassium	Elemental Stock	10000	2	1000	20
Sodium	Elemental Stock	10000	2	1000	20
Strontium	Elemental Stock	1000	5	1000	5.0
HCl	-	-	50	1000	5%
HNO3	-	-	10	1000	1%

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TABLE 4
TJA61 Intermediate Calibration Stock Solutions

STD2A Intermediate Stock

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Antimony	Elemental Stock	1000	50	500	100
Barium	Elemental Stock	1000	5	500	10
Cadmium	Elemental Stock	1000	5	500	10
Chromium	Elemental Stock	1000	50	500	100
Nickel	Elemental Stock	1000	50	500	100
Potassium	Elemental Stock	10000	25	500	500
Sodium	Elemental Stock	10000	25	500	500
Hydrochloric Acid	-	-	25	500	5%
Nitric Acid	-	-	5	500	1%

STD2B Intermediate Stock

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Boron	Elemental Stock	1000	50	500	100
Lithium	Elemental Stock	1000	25	500	50
Phosphorus	Elemental Stock	10000	25	500	500
Silicon	Elemental Stock	1000	50	500	100
Strontium	Elemental Stock	1000	5	500	10
Thallium	Elemental Stock	1000	50	500	100
Tin	Elemental Stock	1000	50	500	100
Hydrochloric Acid	-	-	5	500	5%
Nitric Acid	-	-	25	500	1%

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TABLE 4 - continued

STD3 Intermediate Stock

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	Elemental Stock	10000	5	500	100
Arsenic	Elemental Stock	1000	20	500	40
Beryllium	Elemental Stock	1000	5	500	10
Calcium	Elemental Stock	10000	5	500	100
Cobalt	Elemental Stock	1000	5	500	10
Copper	Elemental Stock	1000	5	500	10
Iron	Elemental Stock	10000	5	500	100
Lead	Elemental Stock	1000	50	500	100
Magnesium	Elemental Stock	10000	5	500	100
Manganese	Elemental Stock	1000	5	500	10
Molybdenum	Elemental Stock	1000	5	500	10
Selenium	Elemental Stock	1000	20	500	40
Titanium	Elemental Stock	1000	5	500	10
Vanadium	Elemental Stock	1000	5	500	10
Zinc	Elemental Stock	1000	25	500	50
Hydrochloric Acid	EMD	-	25	500	5%
Nitric Acid	Baker	-	5	500	1%

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TABLE 5
TJA61 Working Calibration Solutions

STD2 WORKING FOR TJA-61

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Antimony	STD2A STOCK	100	50	500	10
Barium	STD2A STOCK	10	50	500	1
Boron	STD2B STOCK	100	50	500	10
Cadmium	STD2A STOCK	10	50	500	1
Chromium	STD2A STOCK	100	50	500	10
Lithium	STD2B STOCK	50	50	500	5
Nickel	STD2A STOCK	100	50	500	10
Posphorus	STD2B STOCK	500	50	500	50
Potassium	STD2A STOCK	500	50	500	50
Silicon	STD2B STOCK	100	50	500	10
Sodium	STD2A STOCK	500	50	500	50
Strontium	STD2B STOCK	10	50	500	1
Thallium	STD2B STOCK	100	50	500	10
Tin	STD2B STOCK	100	50	500	10
Hydrochloric Acid	-	-	5	500	5%
Nitric Acid	-	-	25	500	1%

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STD3 WORKING FOR TJA-61

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	STD3STOCK	100	50	500	10
Arsenic	STD3STOCK	40	50	500	4
Beryllium	STD3STOCK	10	50	500	1
Calcium	STD3STOCK	100	50	500	10
Cobalt	STD3STOCK	10	50	500	1
Copper	STD3STOCK	10	50	500	1
Iron	STD3STOCK	100	50	500	10
Lead	STD3STOCK	100	50	500	10
Magnesium	STD3STOCK	100	50	500	10
Manganese	STD3STOCK	10	50	500	1
Molybdenum	STD3STOCK	10	50	500	1
Selenium	STD3STOCK	40	50	500	4
Titanium	STD3STOCK	10	50	500	1
Vanadium	STD3STOCK	10	50	500	1
Zinc	STD3STOCK	50	50	500	5
Hydrochloric Acid	-	-	25	500	5%
Nitric Acid	-	-	5	500	1%

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
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
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%

ICV2 Solution (for IRIS only)

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Calcium	Elemental Stock	1000	5	1000	5
Iron	Elemental Stock	1000	25	1000	25
Magnesium	Elemental Stock	1000	25	1000	25
Hydrochloric Acid	-		50	1000	5%
Nitric Acid	-		10	1000	1%

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TABLE 6 - continued

ICV3 Solution

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Boron	Elemental Stock	1000	5	500	10
Lithium	Elemental Stock	1000	2.5	500	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	0.05
Nitric Acid	-		5	500	0.01

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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
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	318.1	(High Line)
Chromium	267.7	
Coblat	228.6	
Copper	324.7	
Iron	259.9	
Iron	271.4	(High Line)
Lead	220.3	
Lithium	670.7	
Magnesium	285.2	
Magnesium	202.5	(High Line)
Manganese	257.6	
Molybdenum	202.0	
Nickel	231.6	
Phosphorus	178.2	
Potassium	766.4	
Selenium	196.0	
Silicon	251.6	
Silver	328.0	
Sodium	589.5	
Strontium	346.4	
Thallium	190.8	
Tin	189.9	
Titanium	323.4	
Vanadium	310.2	
Zinc	206.2	

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TABLE 9
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	
Nickel	231.6	
Phosphorus	214.9	
Potassium	766.5	
Selenium	196	
Silicon	288.1	
Sodium	588.9	
Strontium	421.5	
Thallium	190.8	
Tin	189.9	
Titanium	334.9	
Vanadium	292.4	
Zinc	213.8	
Titanium	323.4	
Vanadium	310.2	
Zinc	206.2	

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STANDARD OPERATING PROCEDURE

PERCENT LIPIDS IN TISSUE
SOC-LIPID
Revision 1
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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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: _____

PERCENT LIPIDS IN TISSUE

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine the lipid content in biological tissue. The procedures used are based on the those described in the Puget Sound Protocols, Bligh & Dyer, and the EPA Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories.

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1.2. This procedure is typically applied to marine tissues. In some cases, the procedure can be used to determine lipids in other biological tissue.

2. METHOD SUMMARY

2.1. A homogenized portion of the tissue sample is extracted with organic solvent. The extract is evaporated using moderate heat and the lipid weight is determined. Percent lipids is calculated from the weight measured.

2.2. The extraction may either be performed independently of the extraction done for other organics analyses or a portion of the extract from another extraction may be used for the percent lipids determination

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3. DEFINITION

Lipids - Naturally occurring compounds that are soluble in such organic solvents as methylene chloride, hydrocarbons, chloroform, benzene, and alcohols. This includes a diverse range of compounds, like fatty acids and their derivatives, carotenoids, terpenes, steroids, cholesterols, and bile acids

4. INTERFERENCES

4.1. Since the procedure is used to provide an estimation of lipid content based on non-volatile extracted material, interferences from co-extracted components that are not considered lipids may occur.

4.2. Due to the nature of the procedure, interferences attributed to laboratory procedures are not typically encountered.

5. SAFETY

Personal protective equipment will include safety glasses (with side shields), gloves, and a lab coat. Follow normal precautions as per the CAS Safety Manual.

6. SAMPLE HANDLING AND PRESERVATION

Tissue samples should stored at -20 ± 4°C until analysis. When frozen, samples should be analyzed within 1 year of sampling. Project-specific protocols may apply to sample storage and holding time requirements.

7. APPARATUS

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7.1. Evaporating pans: Aluminum weighing pans.

7.2. VOA vials, 40mL, precleaned.

7.3. Tissuemizer.

7.4. Drying oven, capable of maintaining a temperature of 70°C.

7.5. Hotplate, capable of maintaining a temperature of 70°C.

7.6. Analytical balance, capable of weighing to 0.1mg.

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8. REAGENTS

8.1. Sodium sulfate (granular, anhydrous), Na2SO4. Purify by heating at 400°C for 4 hours in a shallow tray or crucible, or by precleaning the sodium sulfate with methylene chloride.

8.2. Extraction solvents:

8.2.1. Methylene chloride

8.2.2. Chloroform/Methanol (1:1)

9. PREVENTIVE MAINTENANCE

9.1. 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.

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

Note: If samples are suspected to be high in lipids (i.e. whole fish), the % lipids must be done before samples are run on GPC to prevent overloading of GPC column. The maximum amount of lipid that can be loaded on the GPC column is 2 g. If lipid is over 2g see the extraction supervisor for assistance.

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11.1. PSEP Option:

11.1.1. Homogenize the tissue sample using the CAS *SOP for Tissue Sample Preparation*. If using a portion of extract from an analytical extraction, use 2 ml of extract from the 10 ml intermediate volume before GPC cleanup and skip to section 11.1.6.

11.1.2. Weigh 2 grams of tissue into a VOA vial. Dry the sample with sodium sulfate.

11.1.3. Add 10mL methylene chloride.

11.1.4. Using a tissuemizer, homogenize the tissue/solvent mixture until sample is thoroughly mixed.

11.1.5. Vortex and centrifuge at 100xG for 10 minutes.

11.1.6. Heat sample pans in oven at 70°C for one hour. Remove from oven and weigh as soon as pans are no longer warm (less than 5 minutes). Weigh the pans to the nearest 0.0001 gram.

11.1.7. Pipet 2mL of the extract into a DCM-rinsed glass syringe with a new filter. Filter the extract into a pan and evaporate to dryness. Using a hot plate with temperature <70°C. Place pans in oven at 70°C for one hour.

11.1.8. Remove from oven and weigh as soon as pans are no longer warm (less than 5 minutes). Weigh the pans to the nearest 0.0001 gram.

11.1.9. Calculate percent (%) lipids and GPC loading.

11.2. Bligh & Dyer Option:

11.2.1. Prepare pans: Heat aluminum sample pans in oven at 70° C for a minimum of one hour. Cool pans in a desiccator for a minimum of 30 minutes. Weigh pans to the nearest 0.0001 gram.

11.2.2. Homogenize tissue in accordance with CAS *SOP for Tissue Sample Preparation.*

11.2.3. Weigh the appropriate amount of tissue into a scintillation vial for samples ≤ 3 grams, into a 40 mL VOA vial for samples 3 - 6 grams, or into a 250 mL beaker for large sample 6 - 30 grams.

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11.2.4. Add the appropriate amount of 1:1 Chloroform/Methanol (v/v):

≤ 2 grams, add 10 mL
> 2 - 5 grams, add 25 mL
5 - 10 grams, add 50 mL
10 - 30 grams, add 100 mL

11.2.5. Using a tissumizer, homogenize the sample/solvent mixture for approximately 1 minute or until sample is thoroughly dessimated. Rinse the tissuemizer with solvent. Collect the rinsate with the sample extract. Centrifuge the sample extract at 1000xG for 10 minutes. Transfer extract to a clean VOA vial, leaving the tissue behind. Rinse the tissue portion by adding 1-2 mL of solvent to the tissue. Vortex, centrifuge, and combine with the extract in the VOA vial. Examine the extract to verify that a single phase is present. High water content can result in phase separation. In these cases, add solvent in 1 mL increments until a single phase is achieved.

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11.2.6. **Low Volume Extractions**: Back extract by adding 7 mL of sodium sulfate/DI water (25g sulfate/1000mL H2O) to the extract). Vortex extract and centrifuge at 1000xG for 10 minutes. Lipids will be retained in bottom chloroform layer. Pipette the top water layer off the bottom chloroform solvent layer and discard. If colloidal proteinaceous material is observed, add 0.5 mL of sodium sulfate/DI water, vortex, and centrifuge for another 10 minutes. Discard the top water layer.

11.2.7. **High Volume Extractions**: Transfer the extract with rinses into a 250 mL separatory funnel. Back extract 2 times with 100 mL sodium sulfate/DI water, discarding the top water layer each time (lipids will be retained in the bottom layer). Filter the bottom layer through muffled anhydrous sodium sulfate into a KD. Rinse the funnel with 10 - 20 mL of methylene chloride. Concentrate to

approximately 5 mL on the S-evap, keeping the temperature ≤ 80°C. The extract may need to be centrifuged or filtered at this point.

11.2.8. Concentrate the extract to approximately 2mL using nitrogen.

11.2.9. Place pans on a hot plate at a setting of 3. Filter the extracts through 3mL syringes into the pans and evaporate to dryness. DO NOT LET EXTRACTS BOIL. Heat sample pans in oven at 70° C for a minimum of one hour. Cool pans in a desiccator for a minimum of 30 minutes. Weigh pans to the nearest 0.0001 gram and record the weight.

12. QA/QC REQUIREMENTS

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12.1. QC Samples Required

12.1.1. Analyze one duplicate and triplicate per 20 samples.

12.1.2. Analyze one blank, an empty aluminum pan, per twenty samples.

12.2. Acceptance Criteria

12.2.1. RPD for duplicates should be $<20\%$.

12.2.2. Method blank weight should be less than the absolute value of 0.5mg.

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12.3. Calculations

12.3.1. Calculate percent (%) lipids

PSEP Option:

$$\%\,Lipids = \frac{\left[\left(Wt_{dish+lipid(g)} - Wt_{dish(g)}\right) - Blank\right] \times 5}{Sample\ Wt.(g)} \times 100$$

Bligh & Dyer Option:

$$\%\,Lipids = \frac{\left[\left(Wt_{dish+lipid(g)} - Wt_{dish(g)}\right) - Blank\right]}{Sample\ Wt.(g)} \times 100$$

12.3.2. Calculate number of (g) loaded on GPC (5 ml):

$$\frac{\%\ Lipid}{200} \times Sample\ Wt.(g)$$

12.4. Reporting

12.4.1. Fill out the Excel report for % lipids found at R:\SVG\Forms\lipids.

12.4.2. Samples are weighed to the nearest 0.1mg and report to the nearest 0.1%.

12.4.3. Report results using no more than 3 significant figures.

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12.5. Data review

12.5.1. 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. All data will be initialed, dated and attached to required data quality worksheet.

12.5.2. 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:\SVG\WIP.

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12.5.3. 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.

14. METHOD PERFORMANCE

Refer to the reference methods for additional method performance data available. 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.

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.

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 laboratory Safety Manual, Sections 6 and 7.

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17. REFERENCES

17.1. USEPA Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Volume 1 - Fish Sampling and Analysis, Third Edition, November, 2000.

17.2. Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound, January 1996 and subsequent chapter revisions.

17.3. Bligh & Dyer, Canadian J. Biochemistry and Physiology, Aug. 1959

17.4. CAS *SOP for Tissue Sample Preparation*.

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Attachment C

Laboratory Quality Assurance Manuals

Cover Page:

Quality Assurance Manual

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	10/29/07
Quality Assurance Manager - Nasreen K. DeRubeis	Date
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Purpose and Scope

The purpose of this Laboratory Quality Assurance Manual (LQAM) is to describe the implementation of the Quality System at TestAmerica Pittsburgh laboratory. The LQAM is written within the guidelines of the TestAmerica Quality Management Plan (QMP), which applies to all TestAmerica laboratories. The organization of this QAM is based on the "EPA Requirements for Quality Management Plans" (EPA QA/R-2, August 1994). This QAM outlines specific policies, organization, responsibilities, and activities required to assure high quality laboratory services. The QAM also fulfills the requirements of our clients, government agencies, and NELAC to document the laboratory Quality System.

This LQAM contains references to other essential TestAmerica quality documents. The company-wide QMP, TestAmerica Pittsburgh QAM, and referenced policies and SOPs are interrelated. Together they provide an integrated quality foundation that meets the objectives of the TestAmerica Quality Assurance Policy, as stated in Section 1.2.

The requirements set forth in this document are applicable to all employees at the TestAmerica Pittsburgh laboratory. The policies and practices described here are presented as minimum guidelines only. Based on good scientific judgment, more rigorous requirements may be applied by laboratory employees. Specific requirements delineated in project plans may supersede general quality requirements described in this manual.

Vista
Analytical Laboratory

QUALITY MANUAL

Revision 8

Effective Date: March 2007

William J. Luksemburg
President

Rose M. Harrelson
Quality Assurance Manager

Martha M. Maier
Laboratory Director

James M. Hedin
Director of Instrumentation 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.

Quality Assurance Manual

Second Edition

July 2007

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1.0 Introduction

Nautilus Environmental, LLC (Nautilus) operates two facilities for conducting marine, freshwater, and terrestrial bioassays on a variety of sample types including municipal and industrial effluents, storm water runoff, receiving waters, chemical products, sediments, and soils. We also perform hazardous waste characterization tests for liquid and solid samples. We are accredited by the States of California and Washington. Each state conducts routine audits of the laboratory facilities, procedures, and Quality Assurance (QA) methods and documentation. Documentation of accreditation status is maintained on file and is available to clients for review. In addition, we participate in the annual federal Discharge Monitoring Report (DMR) QA Study, testing blind samples using EPA procedures, and are audited by select clients.

This manual describes the QA program and its associated quality control (QC) procedures that are implemented in the laboratories. Our QA program is designed: 1) to ensure that all tests are performed in accordance with applicable guidelines and regulations; and 2) to increase test performance and precision. The program is broken down into several systems, which are monitored by the QA Officer. These systems include: 1) Management of Change (MOC); 2) Corrective Action Records (CAR); 3) Equipment and Facility Maintenance; 4) Data Generation and Handling (Review, Analysis, Reporting, and Storage); 5) Technical Staff Training; 6) Laboratory Standard Operating Procedure (SOP) Development; and 7) Reference Manual Development.

QC problems leading to poor condition of test animals, inadequate test procedures, or other symptoms may necessitate repeat testing, or lead to incorrect over or under estimations of toxicity, or improperly identified causative toxicants. QC practices address all activities that affect the final quality of the toxicity test data including: sample/material handling, water quality and test conditions, source and condition of test organisms, condition of equipment, instrument calibration and maintenance, measurement of test precision, record keeping, problem identification and corrective actions, data evaluation, and personnel training.

All data generated by the laboratory is monitored for completeness and accuracy at the end of each day and at the end of each individual test period. The detail of record keeping and the general and statistical evaluation of test data are closely monitored to ensure generation of the highest quality data.

1.1 Overview

1.1.1 Proper and Thorough Documentation

Laboratory staff members make a concerted effort to document all aspects of operation. Any permanent changes made in the laboratory are recorded using the MOC forms (see Section 3.1). Any mistakes made in the laboratory are properly documented and corrected using CARs (see Section 3.2).

Documentation of calibrations, instrument adjustments or repairs, dilution/culture water quality measurements, organism culture information, sample or test material source, characterization, receipt, use and disposal, chemical reagent purity, receipt and disposal, etc. are made on a real-time basis. These records are retained in bound books in a secure designated data storage area.

All toxicity test data are reviewed by the technical staff on a daily basis throughout each test period. The level of detail recorded at Nautilus meets or exceeds protocol requirements. At test termination, the Data Manager or Lab Supervisor reviews all test data for completeness and accuracy to ensure the daily reviews are being properly conducted.

1.1.2 Facilities and Equipment

Facilities and equipment are designed around test requirements. Separate heating and cooling systems maintain testing chambers and culture rooms at the desired temperature (±1°C), which is monitored continuously in one location, and intermittently in multiple locations in each environmental chamber. All equipment is maintained within manufacturer's requirements to ensure measurements are as accurate and precise as possible.

Calibration of instruments is performed routinely using NIST-traceable standards as applicable. All critical instrumentation is duplicated to prevent data collection problems associated with instrument failure.

All test chambers and other materials contacting samples are constructed of polypropylene, borosilicate glass, Teflon, or grade 316 stainless steel and are subjected to rigorous cleaning in accordance with EPA procedures using detergent, acid (ACS grade), acetone (ACS grade) and deionized water before use.

1.1.3 Laboratory Water Quality

Water quality of both fresh and saline dilution and culture waters is ensured by several means. Standard reconstituted dilution/culture water is made using deionized water as the base water. Filtered natural seawater is an additional source of saline water used in the laboratories. The quality of reagent grade chemicals or commercial salts used to prepare synthetic fresh or saltwater, respectively, is evaluated using appropriate reference toxicant tests with a number of species (e.g. *Ceriodaphnia dubia, Mysidopsis bahia,* etc.). In addition, both base freshwater and natural seawater, are analyzed annually for organic priority pollutants and trace metals.

1.1.4 Sample Materials

All sample materials tested in the bioassay laboratory are collected, handled, and transported using proper chain-of-custody procedures to ensure sample integrity. Sample containers are pre-cleaned and certified. Sample receipt is documented in a laboratory logbook, and the sample is assigned a tracking number. The final data path details sample-related activities from collection time to disposal time.

1.1.5 Test Organism Quality

Test organisms are used only if they appear healthy, are actively feeding, are the correct species and life stage, and exhibit low mortality (<10%) during culture and acclimation. The quality of all foods is evaluated before use. Culture water quality, animal source, incubation and hatch dates, acclimation conditions and other relevant information is documented for all test organisms cultured in the laboratory. The health of test organisms is routinely evaluated using reference toxicants, the results of which are graphed on laboratory control charts for periodic review by the QA Officer.

1.1.6 Reference Toxicant Testing

Regardless of the organism source (e.g. cultured in-house or purchased from a supplier), a reference toxicant test (i.e. positive control) must be conducted at least once per month for each test method used during that month. If a test method is used only once per month, or less frequently, then a reference toxicant test should be conducted concurrent to sample toxicity testing. If the test organisms were obtained from an outside supplier, regardless of the in-house testing frequency a concurrent

reference toxicant test is required if that supplier cannot provide valid control chart data using the same test method (including reference toxicant type) as the testing laboratory.

Reference toxicant tests are used to evaluate the overall health and sensitivity of the test organisms and to demonstrate the continuing ability of the laboratory to successfully and consistently perform the tests. Control charts are generated for each test species and reference toxicant combination. Charts are reviewed on a monthly basis by the QA Officer to ensure intra-laboratory variability does not exceed EPA benchmarks for each test method. If variability exceeds recommended limits, the QA Officer may conduct investigations and initiate corrective actions.

1.1.7 Testing Procedures

Laboratory SOPs are adapted from appropriate protocols and reference documents (e.g. EPA, ASTM, WDOE). Any deviations from protocol requirements are properly documented and explained. Side-by-side experimental results are kept on file to support protocol changes made by the laboratory. The bioassay laboratory staff is properly trained in all procedures, and their reviews of reference documents and laboratory SOPs are documented (see Section 3.5).

1.1.8 Analytical Laboratories

Analytical laboratories are selected based on reputation, and thoroughness of their QA Plan. Concerted efforts are made to ensure maximum communication and cooperation for every project that requires supporting analytical chemistry data. All analytical reports are reviewed in detail by the project manager upon receipt, and any discrepancies are discussed with the analytical laboratory.

1.1.9 Health and Safety

Nautilus Health & Safety regulations are enforced in the laboratory at all times. In addition, the laboratory has customized emergency and chemical hygiene plans tailored to site-specific needs of the facilities. Annual training is conducted on all policies and procedures. Monthly safety meetings and inspections are also conducted by the laboratory Safety, Health and Environment Coordinator to ensure compliance with and understanding of safety issues and rules. All training, meetings, and

inspections are documented on a real-time basis.

2.0 Laboratory Organization

Position Title	Responsibilities
Managing Director	▪ Has final fiscal responsibility for both laboratories and overall group. ▪ Sets marketing and business development goals for the operation. ▪ Works with laboratory managers to meet goals in a timely manner.
Lab Manager	▪ Has fiscal responsibility for his/her laboratory. ▪ Directs technical advancement. ▪ Dictates new market direction. ▪ Supervises project management, may manage select projects. ▪ Engages in marketing and client correspondence. ▪ Prepares project proposals, and specification sheets. ▪ Supervises Research & Development studies. ▪ Supervises special project report preparation. ▪ Reports directly to the Managing Director.
Operations Manager	▪ Sets test schedule, coordinates sampling efforts with clients. ▪ Oversees lab operations with respect to ordering supplies and test organisms, scheduling facility maintenance, and coordinating all deliveries made to the laboratory. ▪ Reports directly to the Lab Manager.
Senior Scientist	▪ Prepares project proposals. ▪ Engages in marketing and client correspondence for special projects. ▪ Advises Lab Managers on research & development, and technical advancement. ▪ Reports directly to the Managing Director.

Lab Supervisor

- Supervises day-to-day technical operation of the laboratory.
- Conducts/supervises technical staff training.
- Sets staff schedule and level.
- Manages select projects, and assigns project management responsibility to the technical staff as appropriate.
- Prepares special project reports.
- Conducts R&D studies under the supervision of the Lab Manager.
- Reports directly to the Lab Manager.

Project Manager

- Conducts analysis and review of test data to ensure acceptability and protocol compliance.
- Prepares project reports.
- Reports directly to the Lab Manager.

QA Officer

- Oversees the QA program, including conduct of system audits.
- Updates the QA Manual, the Employee Quick Reference Manual, Laboratory SOPs, logbooks, and datasheets.
- Maintains reference toxicant control charts and data archives.
- Conducts R&D studies under the supervision of the Lab Manager.
- Reports directly to the Managing Director, and indirectly to the Lab Managers.

Lab Technicians

- Conduct bioassay testing.
- Monitor and record data.
- Culture and care for laboratory test organisms.
- Perform routine lab maintenance.
- Report directly to Lab Supervisor.

Safety Coordinator

- Coordinates safety training of laboratory staff.
- Ensures safety policy compliance among laboratory staff.
- Oversees chemical hygiene, as well as hazardous waste generation, handling and disposal.
- Conducts routine inspections of laboratory conditions and safety-related equipment.

Field Technicians

- Collect, preserve, and deliver samples in accordance with proper collection techniques and chain-of-custody procedures.
- Report to Lab Manager or Project Manager as project dictates.

Lab Aides

- Prepare glassware for use in testing under EPA guidelines.
- Assist with general lab maintenance and cleaning.
- Report to Lab Supervisor.

3.0 QA Systems

The QA program is broken down into several key components. These system components are maintained by lab personnel based on qualifications and experience. The program as a whole is monitored by the QA Officer, who reviews each component on a regular schedule, and the entire program annually. At the end of a program audit, the QA Officer compiles a summary report and submits that report to the QA Program Development Group. This group consists of the Managing Director, Lab Manager, Lab Supervisor, and a Senior Scientist. The report is discussed, and system changes are recommended. The QA Officer implements the changes to improve the program. Each system is described in detail below.

3.1 Management of Change

Permanent changes that will affect data quality in the laboratory are documented using the Management of Change (MOC) system. The system is broken down into four categories: 1) Changes to test methods, procedures, processes, and experimental designs directly related to SOP updates; 2) changes to specifications directly related to equipment or control limits; 3) changes to personnel or laboratory operations not

directly related to technical work; and 4) changes to documentation including issuance of updated SOPs, QAM, Employee QRM, datasheets, training forms, CARs, etc. The Lab Supervisor discusses and reviews MOC forms with the technical staff at tailgate meetings. Each staff member must sign the form acknowledging his/her understanding of the updated information. MOC forms are reviewed by the QA Officer on a monthly basis to ensure changes have been properly implemented, and that forms are being properly completed, signed, and filed.

3.2 Corrective Actions

In the event there is a problem with any data or lab operation (e.g., erroneous instrument calibration, test temperature out of range) it is the Lab Technician's responsibility to inform the Lab Supervisor and/or the QA Officer immediately. At this time, a Corrective Action Record (CAR) will be completed by the Lab Technician, and approved by the Lab Supervisor or QA Officer. Together, the staff will identify an appropriate course of action to remedy the problem. These CARs are reviewed on a monthly basis by the QA Officer to ensure they are being appropriately utilized, and also to identify recurring problems or trends. The QA Officer will discuss trends and ways to correct them with the Lab Supervisor, and the Lab Manager, if necessary. If appropriate, a copy of the CAR will be included with the affected client data file.

3.3 Facility and Equipment Maintenance

All pertinent lab-monitoring records are kept in bound logbooks and maintained securely for a minimum of five years. Records of field collections, organism receipt, shipping, culture and holding, chemical receipt, chemical stock preparation, laboratory water quality, instrument calibration, oven and refrigerator temperatures, and environmental chamber temperatures are maintained. Depending on frequency of monitoring, logbooks are reviewed for completeness either daily or weekly by a Lab Technician, as designated by the Lab Supervisor.

All lab instruments and equipment have regular service schedules according to specific manufacturer requirements. Service files are maintained for each piece of equipment including all invoices, and certifications, as appropriate. A master list of laboratory equipment is maintained with current certifications, and service dates. The service schedules are tracked on an annual calendar. The QA Officer reviews the equipment maintenance system semi-annually to ensure that the Lab Technician in charge of the

program is maintaining files, and servicing equipment in an appropriate and timely manner.

3.4 Data Generation and Handling

Test data for each bioassay are recorded on datasheets designed specifically for individual test types. All information on raw datasheets is recorded in black ink. Errors are crossed through with a single line and initialed by the Lab Technician. Supplemental information is footnoted on each page. A Lab Technician checks Datasheets for completeness at the end of each day. The Lab Supervisor assigns data review duties on a monthly basis. The Lab Supervisor, and the Project Manager conduct additional spot-checks to ensure Lab Technicians are properly reviewing test data.

EPA methodology test results are analyzed using CETIS, a software program developed by Tidepool Scientific Software. CETIS is a comprehensive toxicity data analysis and database software package that is used for reporting toxicity test data as part of permit monitoring activities. CETIS performs all appropriate statistical analyses and records the results in a format approved by EPA Regions IX and X as well as the States of California and Washington. Graphpad Prism version 4.0, SigmaStat version 2.03, and Excel 2000 are routinely used programs for a variety of additional statistical and graphical analyses. Following the entry of data into a computer file, the original data form is filed in the project file with the chain-of custody forms and a full narrative report. All information is archived for a minimum of five years or as project-specific contract terms dictate.

Requirements for test acceptability vary according to each protocol and are detailed in the associated SOP. Data are evaluated against the acceptability and statistical (e.g. appropriate analysis selection) criteria associated with each protocol. Reference toxicant control charts are maintained for each test protocol and species combination and are used in the evaluation of water quality, animal health, and procedural implementation.

3.5 Training Program

The Lab Supervisor maintains a training file for each Nautilus Lab Technician. Upon hire, the technician is trained immediately in health & safety policies/procedures, and introduced to the QA program. QA program training includes review of the QA manual, the Employee Quick Reference Manual, and introduction to the MOC and CAR systems. Each new employee must sign a form acknowledging his/her review and understanding of these systems.

Technical training begins immediately. Solely the Lab Supervisor and Senior Lab Technicians conduct training. Dates and trainer information are recorded in the employee file for each procedure certified. Certification includes first shadowing the trainer, and then performing the test proficiently on their own under the scrutiny of the trainer. The Lab Supervisor conducts random spot checks of each technician quarterly to ensure their continued adherence to and understanding of the procedures. All reviews are documented and acknowledged by the person being observed.

The Lab Manager also conducts semi-annual audits of the training process itself to ensure that both the Lab Supervisor and Senior Lab Technicians train employees consistently with one another, and with the laboratory SOPs. All reviews are documented, and acknowledged by the trainer and trainee.

3.6 Reference Document Development

It is the responsibility of the QA Officer to review day-to-day variations in testing procedures. These variations include comparisons of results between projects, variability in batches of organisms from suppliers and between suppliers, variability in chemical/physical properties of holding and acclimation in test water, and consistency of employee performance. In the event of a problem with any laboratory procedure, the QA Officer, Lab Supervisor, and Lab Manager will deliberate the problem, and discuss the potential solutions. Experiments will be conducted by the QA Officer to ensure the feasibility of any new procedure. Upon development of an improved method that is in accordance with test protocol requirements, a new SOP will be written, reviewed, and implemented. In accordance with these principles, the QA Officer reviews the laboratory SOPs, QA manual, Employee Quick Reference Manuals,

logbooks, and datasheets annually. All of these items are updated annually, at minimum, or as necessary to improve data quality.

4.0 Facilities and Equipment

4.1 Laboratory Locations

California
5550 Morehouse Drive, Suite 150
San Diego, California 92121

Washington
5009 Pacific Highway East, Suite 2
Tacoma, Washington 98424

4.2 Temperature-Controlled Environmental Chambers

Samples are stored in coldrooms maintained at 4°C, until needed for testing. Testing is conducted in temperature and light-controlled environmental chambers. Modifiable environmental chambers are set at 12, 15, 20, or 25°C and maintained within ± 1°C of the target temperature. A Supco CR-87 temperature recorder continuously monitors the temperature at one location inside each environmental chamber. The charts generated by each recorder are exchanged weekly and kept on file at the laboratory or at a secure offsite storage location indefinitely. The temperature of each controlled chamber is verified with a thermometer using a surrogate temperature blank comparable in volume to test conditions. These measurements are recorded daily in a laboratory logbook. For other project-specific temperatures, water baths equipped with either a heater or a chiller unit, as appropriate, is maintained as alternate temperature control systems. Fluorescent lighting provides illumination in all testing areas. Specific wavelengths are used for individual tests as dictated by the protocol and laboratory SOP. Photoperiod is controlled with a timing device. All environmental chamber temperatures are verified annually using a NIST-certified thermometer.

4.3 Instrumentation

In-house instrumentation is adequate to measure appropriate physical and chemical variables for both marine and freshwater bioassays. All calibration measurements are recorded in the laboratory logbooks maintained for each meter or instrument.

4.3.1 San Diego Laboratory

Salinity

Salinity measurements are taken with an Orion 115 hand-held temperature compensated meter. The digital meter has accuracy after adjustment (via cell constant) of ± 0.5% max in the range 0.0 to 80.0 parts per thousand (ppt) and a temperature measurement range of –5.0°C to 105°C. The instrument is calibrated daily to seawater.

Conductivity

Conductivity is measured using an Orion 130 meter. The digital meter has an accuracy ± 0.5 percent of measurement value ± 1 digit at operating temperature -10°C to +55° C. The temperature measurement range is –5.0°C to 99.9°C. The instrument is calibrated daily to known standards.

pH

Measurements of pH are taken using an Orion 250A, or 250A+ meter with a temperature compensated electrode, which is calibrated daily using standard pH buffer solutions. Both models measure a pH in the range of 0 to 14 with relative accuracy of ±0.02 and a temperature measurement range of –5.0°C to 105°C.

Dissolved Oxygen

Dissolved oxygen (DO) measurements are made with a YSI 55 meter. The DO measurement range is 0-20 mg/L ± 0.3 with a temperature measurement range of –5.0°C to 45°C. The meter is calibrated daily to air saturated with deionized water.

Ammonia

Routine analysis of ammonia is performed using a Hach DR2000 spectrophotometer following the Hach ammonia salicylate colorimetric method 8038. This method is equivalent to USEPA method 350.2. The meter is calibrated to a known standard before each use as suggested by the manufacturer.

Alkalinity and Hardness

Alkalinity and hardness are both measured using the Hach digital titrator model 16900. The Hach methods for measuring alkalinity and hardness are 8203 and 8213, respectively.

Chlorine

Total and free chlorine residual measurements are made using a Hach DR700 Colorimeter and accompanying reagent packets. Total chlorine measurements are performed following Hach method 8167, free chlorine using method 8021. Both methods are equivalent to the USEPA method 330.5.

Light

Measurements of light intensity are performed using a Dickson DLM2 photometer. The meter is sent to the manufacturer for annual calibration.

Weights and Volumes

Materials used in reference toxicant testing are weighed to the nearest 0.01 mg using a Mettler AE240 balance. The balance is calibrated prior to use and professionally serviced annually using weights traceable to NIST standards. Liquid measurements are made using borosilicate plastic or glass pipettes and graduated cylinders.

4.3.1 Tacoma Laboratory

Salinity

Salinity is measured using an Orion 130A meter. The digital meter has an accuracy ± 0.5 percent of measurement value ± 1 digit at operating temperature -10°C to +55°C. The temperature measurement range is –5.0°C to 99.9°C. The instrument is calibrated daily to seawater.

Conductivity

Conductivity is measured using an Orion 130A meter. The digital meter has an accuracy ± 0.5 percent of measurement value ± 1 digit at operating temperature -10° C to +55°C. The temperature measurement range is –5.0°C to 99.9°C. The instrument is calibrated daily to known standards.

pH

Measurements of pH are taken using an Orion 320 meter with a temperature compensated electrode, which is calibrated daily using standard pH buffer solutions. This model measures pH in the range of 0 – 14 ± 0.02 (at room temp.) and measures temperatures from 0.0 to 100°C.

Dissolved Oxygen

Dissolved oxygen (DO) measurements are made with YSI 55 and YSI 58 meters. The DO measurement range for both models is 0-20 mg/L ± 0.3 with a temperature measurement range of –5.0°C to 45°C. The meters are calibrated daily to air saturated with deionized water.

Ammonia

Routine analysis of ammonia is performed using a Hach DR2000 spectrophotometer following the Hach ammonia salicylate colorimetric method 8038. This method is equivalent to USEPA method 350.2. The meter is calibrated to a known standard before each use as suggested by the manufacturer.

Alkalinity and Hardness

Alkalinity is measured by titration using 0.02 N Sulfuric acid as the titrant and 1:1 v:v methyl red and bromocresal green indicator. Hardness is measured by hand titration using 0.01 M EDTA solution as the titrant and calmagite hardness indicator.

Chlorine

Total and free chlorine residual measurements are made using a Hach DR700 Colorimeter and accompanying reagent packets. Total chlorine measurements are performed following Hach method 8167, free chlorine using method 8021. Both methods are equivalent to the USEPA method 330.5.

Light

Measurements of light intensity are performed using a Dickson DLM2 photometer. The meter is sent to the manufacturer for annual calibration.

Weights and Volumes

Materials used in reference toxicant testing are weighed to the nearest 0.01 mg using a Mettler AE240 balance. The balance is calibrated prior to use and professionally serviced annually using weights traceable to NIST standards. Liquid measurements are made using borosilicate plastic or glass pipettes and graduated cylinders.

5.0 Reference and Laboratory Waters

Laboratory waters (freshwaters and seawater) are analyzed annually for organochlorine and organophosphorus pesticides, metals, bacterial properties, ammonia, total organic carbon, chemical and biological oxygen demand, total suspended solids, and tributyltin (seawater only). All determinations are performed by a contracted analytical laboratory using appropriate chemical techniques and are maintained on permanent file.

Water quality is also evaluated on a performance basis for every test conducted in the laboratory. A control sample is run with each test conducted in the lab to ensure the quality of the water used for dilution. The decision to repeat a test is made on a case-

by-case basis by the Lab Manager and includes consideration of control failure and reference toxicant performance as indicated by laboratory control charts.

5.1 San Diego Laboratory

5.1.1 Seawater

The laboratory's seawater system provides filtered, temperature-regulated seawater for testing and animal culture and holding. Natural seawater is collected from the Pacific Ocean and is high-pressure sand filtered at the Scripps Institution of Oceanography in La Jolla, California before transport to the lab by truck. In the lab, seawater is stored in two 2200-gallon storage tanks. The seawater is continuously cycled through an in-line 20-µm fiber filter system and a chiller unit prior to being delivered to sample preparation and testing areas.

Seawater is piped throughout the lab via PVC supply lines at a rate of 10-20 gallons per minute; thus the majority of the water is recycled back into the storage tanks. The seawater system is a once-through system in that all water released from the supply lines into test or culture areas is discharged into the sewer system. The stored water is cycled through the filter and chiller system approximately 10-15 times per day.

Synthetic seawater is made using Forty Fathoms or Marine Mix artificial salt and deionized water. Synthetic seawater is made at least 24 hours prior to use and discarded if not used within 7 days. Synthetic seawater is stored in 20-L lined buckets or large nalgene containers, and supplied with continuous aeration through fiberglass air stones.

Each week, salinity, alkalinity, and total chlorine are measured and recorded in a bound logbook for each type of seawater used for testing.

5.1.2 Freshwater

The laboratory generates high-purity deionized water using a system set up and maintained by U.S. Filter. Water is fed through a system consisting of a pre-filter, an activated carbon tank, two mixed bed resin tanks, and a post-filter. This water is used for synthetic seawater preparation, dilution of natural seawater, and as a final rinse for all glassware.

Nanopure water is used as the base for 20 percent dilute mineral water (DMW), and reagent preparations. It is obtained by piping deionized water through an additional series of filters.

Another type of high quality, laboratory-grade freshwater is obtained from a Culligan water treatment system. It employs a series of carbon filters and water softeners regularly maintained by the manufacturer. This water is supplied by PVC pipe throughout the lab.

The alkalinity, hardness, and total chlorine of Culligan water are measured and recorded in a bound logbook prior to use. DMW alkalinity and hardness are measured and recorded daily because DMW is prepared and used on a daily basis for organism culture.

5.2 Tacoma Laboratory

5.2.1 Seawater

The laboratory obtains natural seawater (for testing echinoderms and bivalves) from the Point Defiance Zoo and Aquarium in Tacoma, Washington, which collects and filters seawater from Puget Sound. Synthetic seawater is made using Marine Mix or Forty Fathoms artificial salt and to deionized water. Both natural and synthetic seawater are stored in clean 20-L cubitainers and large nalgene containers. Synthetic seawater is made at least 24-hours prior to use and discarded if not used within 7 days.

5.2.2 Freshwater

The laboratory generates high-purity deionized water using a system built and maintained by U.S. Filter. Water is fed through a system consisting of a pre-filter, an activated carbon tank, two mixed bed resin tanks, and a post-filter.

The laboratory dechlorinates water using a system set up and regularly maintained by U.S. Filter. Water is fed through a system consisting of a pre-filter, two activated carbon tanks, and a post-filter.

Moderately Hard Synthetic Water (MHSW) is made according to EPA specifications using high quality chemicals and deionized water. MHSW is aerated in 200-L carboys at room temperature. It is discarded if not used within 14 days.

Alkalinity and hardness are measured and recorded for each batch of MHSW prepared for testing or culturing (every 1-4 days). Total chlorine, alkalinity, and hardness are monitored monthly in dechlorinated water.

6.0 Sample Materials

Nautilus works with clients to facilitate sample collection, shipment, and proper handling. Proper chain-of-custody procedures are followed at all times and all tests are initiated within the allowable holding time except when specified to conduct testing out of holding by our client.

6.1 Collection

6.1.1 Effluent and Receiving Water Samples

Sampling method may be dictated by NPDES permit language. Effluent and receiving water collection may be performed by a grab or a composite sampler. Grab samples are good monitors of instantaneous toxicity and are used when trying to monitor effluent variation over a 24-hour period. Samples are tested immediately, reducing the possibility of changes in toxicity due to storage. Composite samples are preferred over grab samples in certain situations because the likelihood of obtaining a representative effluent sample is far greater when sampling over time. Standard EPA procedure is to take composite samples over a 24-hour period with collections at 1/2-hour intervals (48 collections per day). For a 7-day chronic bioassay, at least three 24-hour composites are used for a single test. Samples are tested as soon after collection as possible to reduce the loss of volatile toxic components. The maximum allowable holding time for virtually all samples is 36 hours to initiate a test and 72 hours thereafter. Samples from clients with site-specific holding times due to geography or process issues are handled on a case-by-case basis.

6.1.2 Sediment Samples

Sediments are collected, handled, and preserved according to the procedures in the EPA/Corps of Engineers Implementation Manual 1977, and site-specific recommendations made by the EPA and Corps of Engineers for individual projects. Test material may be collected using a wide variety of methods including but not limited to vibracores, boxcores, Van Veen grabs, pipe dredges, and diver cores. Cores collected for dredged material evaluations are taken to project depth plus a 2-foot overage allowance or to the depth of refusal (the depth at which the vibracore can no longer proceed). Reference sediment is collected at an appropriate and pre-determined uncontaminated site that is selected based upon its similarity to the test sites in grain size and organic carbon content. Control material is collected at the site of test animal collection. Control testing is conducted to determine the health of the test organisms and verify that holding and testing procedures do not affect test animal health. Prior to testing, all test material, reference, and control sediments are held in properly labeled containers and maintained at 4°C until prepared for testing. For most tests, sediments are sieved through a 0.5 or 1.0 mm mesh screen prior to testing to remove resident animals and debris. Testing is initiated within 14 days of sample collection unless otherwise dictated by the sampling and analysis plan.

6.1.3 Title 22, WDOE 80-12 - Hazardous Waste Characterization

Samples for hazardous waste characterization are collected in accordance with Title 22 Regulations in California and the WDOE 80-12 regulations in Washington. Procedures vary according to the specific matrix, type of site monitored and project-specific permit or order language. Please refer to the SOP's for procedural details.

6.2 Sample Receipt and Handling

6.2.1 Chain of Custody

Field Procedures

Proper chain-of-custody forms are maintained by lab personnel for all field-collected samples. Chain-of-custody forms include sample type, sampler's name, date and time of collection, location of the collection, quantity of material, type of collection container, date of delivery, and type of test to be performed. If the samples received

are collected by a Nautilus field technician, that technician is responsible for completing the chain-of-custody forms. In most cases, sample containers are provided to clients or personnel from other Nautilus departments who collect the sample according to our instructions and EPA procedures. In those cases, chain-of-custody forms accompany the sample containers, and the client, internal or external, is responsible for completing the forms, which are received, checked against items received, and counter-signed upon receipt at the laboratory.

Laboratory Procedures

Samples are received at the laboratory with a chain-of-custody form including sample identification, collection date and time, sample type, number and type of containers, analyses required, the name of the project manager and collector, and the relinquishing signature. When transferring sample possession, the transferor and transferee sign, date, and record the time as well as any pertinent information about the sample (e.g., warm sample, container leaking, unusual turn-around time). The form allows for transfer of a group of samples at one time. Samples are handled by the minimum number of people possible. Upon receipt, the sample is logged into a laboratory book and assigned a unique identification number. Physical and chemical parameters including temperature, alkalinity, hardness, free and total chlorine residuals, pH, dissolved oxygen, conductivity or salinity, and a notation of any required adjustments are recorded. The samples are stored at 4°C until used in testing, at which time they are adjusted to the appropriate test temperature. Samples are discarded only when the test report is complete or the sample holding time has expired. All samples are disposed of in accordance with state, federal, and local regulations.

All samples received for hazardous waste characterization are logged in a separate sample receipt logbook and stored in a locked refrigerator at 4°C until the test is initiated. Samples characterized as hazardous are either returned to the client or disposed of under applicable hazardous waste requirements.

6.2.2 Sample Adjustments

Salinity Adjustments

In most cases, marine tests are conducted between 28-36 ppt salinity. If the salinity of samples collected for marine testing is less than 28 ppt and the concentration series for testing includes treatments higher than 10 percent sample, the salinity is increased by adding hypersaline brine or artificial sea salts. Brine of salinity between 70 and 90 ppt is used in the Nautilus labs; brine of salinity 90 or above is diluted to the acceptable range due to potential toxic effects. For site or situation specific testing requiring lower salinities, dilution water salinity may be lowered using deionized water. Salinity manipulations are made within the bounds of test organism tolerance.

Chlorine Adjustments

Chlorine residual is occasionally present in treated wastewater discharges and is often removed to avoid confounding toxicity when free chlorine levels exceed 0.1 mg/L. This procedure is allowed under EPA protocols; however, variances exist for specific NPDES permits and monitoring programs. Project-specific requirements for dechlorination procedures are provided in writing on weekly and daily checklists by the project manager. Chlorine is removed by adding sodium thiosulfate in 0.1 g increments until the free chlorine level falls below this concentration. Internal testing of sodium thiosulfate has been conducted at concentrations well above laboratory application rates to ensure no potential for deleterious effects due to the addition of this compound.

pH Adjustments

When required, adjustment of pH is performed by carefully adding 0.1 or 1.0 M HCl or NaOH. The amount and type of chemical used as well as initial and final pH values are documented.

Effluent Samples: If the pH of a sample upon receipt is greater than 9.0 or less than 6.0, it must be adjusted for routine testing procedures. When pH is outside this range in effluent samples, the pH is adjusted to match that of the receiving water body. If pH for the receiving water is unknown, the pH shall be adjusted to 7.0 for freshwater samples or receiving environments and 8.0 for marine samples or receiving

environments. When a sample requires pH adjustment, a second pH-adjusted control must accompany the bioassay test. The pH-adjusted test shall also be performed concurrent to an un-adjusted effluent sample.

Hazardous Waste: If the pH of samples prepared for hazardous waste characterization is greater than 8.5 or less than 5.5, it must be adjusted to fall just within this range. A second pH-adjusted control must accompany the bioassay test when a sample requires pH adjustment.

7.0 Test Organism Culture and Holding

7.1 Procurement

Test organisms used in bioassays are collected from sources known to be generally free of pollutants, purchased from reputable suppliers, or obtained from laboratory stocks. If organisms are purchased, the vendors are screened by reputation, depth of knowledge concerning the organism of choice, evidence of their QA program, and their ability to consistently deliver healthy test organisms. Test organisms are appropriately acclimated to laboratory conditions and held at test temperature and salinity prior to use.

7.2 Health Assessment

Test organisms are evaluated on a performance basis for every test conducted in the laboratory. A test control and full reference toxicant test are run concurrent to each effluent test or batches of organisms, as appropriate to evaluate the health of the test organisms.

7.3 Documentation

Records of time and place of animal collection, feeding information, and data on water quality in the holding aquaria are maintained in a bound logbook and the records retained indefinitely.

8.0 Reference Toxicant Testing

Reference toxicant testing is conducted to evaluate the health of the organisms used in testing and to ensure that testing procedures do not cause adverse effects to the health of the organism. Nautilus conducts a reference toxicant test in association with each set of client bioassays (concurrent or based on organism batch), and prepares control charts for each reference toxicant and organism combination using point estimate data. The control chart displays the mean and outer limits at ± 2 standard deviations. Tests exceeding these warning levels are examined on a case-by-case basis. For tests with specific requirements, acceptance criteria and reference toxicant data will be used for test validation. Criteria for each test are detailed in each SOP.

The laboratory waters specified in Section 5.0 are used as the dilution water for reference toxicant testing. Dilution ranges for reference toxicant concentrations are based on written protocols, historical data, and are subject to change by the laboratory to better estimate effects when not protocol-specified.

9.0 Testing Procedures

Nautilus employs procedures derived from the following documents. Any modified methods are specified in internal SOPs.

9.1 Water-only Exposures

American Public Health Association. Most Recent. Standard Methods for the Examination of Water and Wastewater. Washington, DC.

ASTM. 1993. Conducting Static Acute Toxicity Tests with Larvae of Four Bivalve Mollusks. ASTM Designation E 724 - 80.

California Department of Fish and Game. 1987. Static Acute Bioassay Procedures for Hazardous Waste Samples.

Dinnel, P. A., J. M. Link, and Q. S. Stober, 1987. Improved methodology for a sea urchin sperm cell bioassay for marine waters. Arch. Environ. Contam. Toxicol. 16:23-32.

Marine Bioassay Project, State of California Water Resources Control Board. 1989. Experimental Evaluation of Effluent Toxicity Testing Protocols with Giant Kelp, Mysids, Red Abalone, and Topsmelt. (Division of Water Quality Report No. 89-5WQ).

Marine Bioassay Project, State of California Water Resources Control Board. 1996. Experimental Evaluation of Effluent Toxicity Testing Protocols with Giant Kelp, Mysids, Red Abalone, and Topsmelt. (Division of Water Quality Report No. 96-1WQ).

US Army Corps of Engineers. 1981. Procedures for Handling and Chemical Analysis of Sediment and Water Samples. Environmental Laboratory, Waterways Experimental Station, Vicksburg, Mississippi (Technical Report EPA/CE-81-1).

US EPA. 1970. Handbook for Analytical Quality Control in Water and Wastewater Laboratories. Environmental and Support Laboratory, Cincinnati, Ohio, (EPA-600/4-79-019 March 1970).

US EPA. 1978. Quality Assurance Guidelines for Biological Testing. Environmental Monitoring and Support Laboratory, Las Vegas, NV (EPA-600/4-78-043).

US EPA. 1985. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms. Environmental and Support Laboratory, Cincinnati, Ohio, (EPA-600/4-85-019).

US EPA. 1988. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio (EPA/600/4-87-028).

US EPA. 1989. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. Environmental and Support Laboratory, Cincinnati, Ohio (EPA/600/4-89/001).

US EPA. 1991a. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms. Environmental and Support Laboratory, Cincinnati, Ohio, (EPA-600/4-90-027).

US EPA. 1991b. Methods for Aquatic Toxicity Identification Evaluation - Phase I Toxicity Characterization Procedures, 2nd Edition, (EPA/600/6-91/003).

US EPA. 1993a. Methods for Aquatic Toxicity Identification Evaluations - Phase II Toxicity Identification Procedures for Samples Exhibiting Acute and Chronic Toxicity. (EPA/600/R-92/080).

US EPA. 1993b. Methods for Aquatic Toxicity Identification Evaluations - Phase III Toxicity Conformation Procedures for Samples Exhibiting Acute and Chronic Toxicity (EPA/600/R-92/081).

US EPA. 1993c. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms. Environmental and Support Laboratory, Cincinnati, Ohio (EPA-600/4-90-027F).

US EPA. 1994. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. Environmental and Support Laboratory, Cincinnati, Ohio (EPA/600/4-91-002).

US EPA. 1995. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to West Coast Marine and Estuarine Organisms. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio (EPA/600/R-95/136).

US EPA. 1996a. Ecological Effects Test Guidelines. OPPTS 850.1000 Special Considerations for Conducting Aquatic Laboratory Studies. EPA 712-C-96-113, Public Draft.

US EPA. 1996b. Ecological Effects Test Guidelines. OPPTS 850.1010 Aquatic Invertebrate Acute Toxicity Test, Freshwater Daphnids. EPA 712-C-96-114, Public Draft.

US EPA. 1996c. Ecological Effects Test Guidelines. OPPTS 850.1075 Fish Acute Toxicity Test, Freshwater and Marine. EPA 712-C-96-118, Public Draft.

US EPA. 1996d. Marine Toxicity Identification Evaluation (TIE) – Phase I Guidance Document (EPA/600/R-96/054).

US EPA. 2002a. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. Fifth Edition. United States Environmental Protection Agency Office of Water, Washington DC (EPA-821-R-02-012).

US EPA. 2002b. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. Fourth Edition. United States Environmental Protection Agency Office of Water, Washington DC (EPA-821-R-02-013).

US EPA. 2002c. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. Third Edition. United States Environmental Protection Agency Office of Water, Washington DC (EPA-821-R-02-014).

Washington State Department of Ecology. 1981. Biological Testing Methods. Hazardous Waste Section (DOE 80-12).

Washington State Department of Ecology. 1998. Laboratory Guidance and Whole Effluent Toxicity Test Review Criteria. Water Quality Program (Publication No. WQ-R-95-80).

9.2 Sediment (Whole and Suspended-Particulate Phase) Exposures

ASTM. 1990. Standard Test Method for Particle-Size Analysis of Soils. American Society for Testing and Materials, ASTM Designation D 422-63 (Reapproved 1990).

ASTM. 1994. Standard Guide for Conducting Sediment Toxicity Tests with Freshwater Invertebrates. American Society for Testing and Materials, ASTM Designation E 1383-94.

ASTM. 1997. Standard Guide for Conducting Laboratory Soil Toxicity or Bioaccumulation Tests with the Lumbricid Earthworm *Eisenia fetida*. American Society for Testing and Materials, ASTM Designation E 1676-97.

ASTM. 1998. Standard Guide for Conducting Static Acute Toxicity Tests Starting With Embryos of Four Species of Saltwater Bivalve Molluscs. American Society for Testing and Materials, ASTM Designation E 724-98.

ASTM. 1999. Standard Guide for Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods. American Society for Testing and Materials, ASTM Designation E 1367-99.

ASTM. 2000a. Standard Guide for Determination of Bioaccumulation of Sediment-Associated Contaminants by Benthic Invertebrates. American Society for Testing and Materials, ASTM Designation E 1688-00a.

ASTM. 2000b. Standard Guide for Measuring the Toxicity of Sediment-Associated Contaminants with Freshwater Invertebrates. American Society for Testing and Materials, ASTM Designation E 1706-00.

ASTM. 2000c. Standard Guide for Conducting Sediment Toxicity Tests with Polychaetous Annelids. American Society for Testing and Materials, ASTM Designation E 1611-00.

ASTM. 2002d. Standard Guide for Designing Biological Tests with Sediments. American Society for Testing and Materials, ASTM Designation E 1525-02.

Dillon, T.M., D.W. Moore and A.B. Gibson. 1993. Development of a chronic sublethal bioassay for evaluating contaminated sediment with the marine polychaete worm Nereis (*Neanthes*) *arenaceodentata*. Environ. Tox. And Chem. Vol 12: 589-605.

Plumb, R.H., Jr. 1981. Procedure for handling and chemical analysis of sediment and water samples. Tech. Rep. EPA/CE-81-1 prepared by Great Lakes Laboratory, State University College at Buffalo, Buffalo, NY, for the U.S. Environmental Protection Agency/ U.S. Army Corps of Engineers Technical Committee on Criteria for Dredged and Fill Material. Published by the U.S. Army Engineer Waterways Experiment Station, Vicksburg, MS.

Puget Sound Estuary Program (PSEP). 1991. Recommended Guidelines for Conducting Laboratory Bioassays on Puget Sound Sediments. Interim Final. U.S. Environmental Protection Agency Region 10, Office of Puget Sound, Seattle, Washington.

Puget Sound Dredged Disposal Analysis (PSSDA) Program. 2000. Dredged Material Evaluation and Disposal Procedures. A Users Manual for the PSSDA Program. U.S. Army Corps of Engineers, Seattle District, U.S. Environmental Protection Agency Region 10, Office of Puget Sound, Seattle, WA, Washington Department of Natural Resources, Washjington Department of Ecology.

Swartz, R. C., W. A. DeBen, J. K. P. Jones, J. O. Lamberson, and F. A. Cole, 1985. Phoxocephalid Amphipod Bioassay of Marine Sediment Toxicity. Aquatic Toxicology and Hazard Assessment. 7th Symposium. ASTM STP854. pp. 284-307.

US Army Corps of Engineers. 1998a. Dredge Material Evaluation Framework, Lower Columbia River Management Area. US Army Corps of Engineers, Northwest Division, US

EPA Region 10, Washington State Department of Ecology, Oregon Department of Environmental Quality, Washington State Department of Natural Resources.

US Army Corps of Engineers. 1998b. Great Lakes Dredged Material Testing & Evaluation Manual. US Environmental Protection Agency (USEPA) Regions 2, 3, and 5, the USEPA Great Lakes National Program Office, and U.S. Army Corps of Engineers (USACE) Great Lakes & Ohio River Division.

US Army Corps of Engineers. 2003. Evaluation of Dredged Material Proposed for Disposal at Island, Nearshore, or Upland Confined Disposal Facilities - Testing Manual. US Army Corps of Engineers. ERDC/EL TR-03-01.

US EPA. 1991. Evaluation of Dredged Material Proposed for Ocean Disposal (Green Book Testing Manual), US EPA Office of Water, Department of the Army US Army Corps of Engineers. EPA 503/8-91/001.

US EPA. 1994. Methods for Assessing the Toxicity of Sediment-associated Contaminants to Estuarine and Marine Amphipods. US EPA Office of Research and Development, Washington DC. EPA 600/R-94/025.

US EPA. 1998. Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. - Testing Manual (The Inland Testing Manual). US EPA Office of Water, Department of the Army U.S. Army Corps of Engineers. EPA 823-B-98-004.

US EPA. 2000. Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates. EPA/600/R-99/064

Washington State Department of Ecology. 1996. Earthworm Bioassay Protocol for Soil Toxicity Screening. WDOE Publication Number 96-327.

9.2.1 Aquatic Protocols Applied to Sediments

ASTM. 1994. Standard Guide for Conducting Bioconcentration Tests with Fishes and Saltwater Bivalve Mollusks. ASTM Designation E 1022-94.

ASTM. 1996. Standard Guide for Conducting Acute Toxicity Tests on Test Materials with Fishes, Macroinvertebrates, and Amphibians. ASTM Designation E 729-96.

ASTM. 1997. Standard Guide for Conducting *Daphnia magna* Life-Cycle Toxicity Tests. ASTM Designation E 1193-97.

ASTM. 1998a. Standard Guide for Conducting Early Life-Stage Toxicity Tests with Fishes. ASTM Designation E 1241-98.

ASTM. 1998b. Standard Guide for Conducting Acute Toxicity Tests with Echinoid Embryos. ASTM Designation E 1563-98.

ASTM. 1998c. Standard Guide for Conducting Acute Toxicity Test with the Rotifer Brachionus. ASTM Designation E 1440-91 (Reapproved 1998).

Dinnel, P. A., J. M. Link, and Q. S. Stober, 1987. Improved methodology for a sea urchin sperm cell bioassay for marine waters. Arch. Environ. Contam. Toxicol. 16:23-32.

Marine Bioassay Project, State of California Water Resources Control Board. 1996. Experimental Evaluation of Effluent Toxicity Testing Protocols with Giant Kelp, Mysids, Red Abalone, and Topsmelt. (Division of Water Quality Report No. 96-1WQ).

US EPA. 1993. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms. Fourth Edition. United States Environmental Protection Agency, Environmental and Support Laboratory, Cincinnati, Ohio (EPA-600/4-90-027F).

US EPA. 1995. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to West Coast Marine and Estuarine Organisms. United States Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio (EPA/600/R-95/136).

US EPA. 1994. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. Third Edition. United States Environmental Protection Agency, Environmental and Support Laboratory, Cincinnati, Ohio (EPA/600/4-91-002). .

US EPA. 2002a. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. Fifth Edition. United States Environmental Protection Agency Office of Water, Washington DC (EPA-821-R-02-012).

US EPA. 2002b. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. Fourth Edition. United States Environmental Protection Agency Office of Water, Washington DC (EPA-821-R-02-013).

US EPA. 2002c. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. Third Edition. United States Environmental Protection Agency Office of Water, Washington DC (EPA-821-R-02-014).

10.0 Analytical Laboratory QA/QC

Laboratory QA/QC activities provide information needed to assess potential laboratory contamination, analytical precision and accuracy, and representativeness. Field-collected QA/QC samples such as blanks, and duplicates are used to certify values obtained through analytical analyses. Blanks help verify that the equipment and sample containers are not contaminated, and the sampling techniques used are non-contaminating. Sample duplicates allow evaluation of sampling error introduced by both field sampling and laboratory analyses. Duplicate samples are sent “blind” to the laboratories. Duplicate analyses results are evaluated by calculating the relative percent difference (RPD) between the two sets of results. This serves as a measure of the reproducibility (precision) of the sample results.

The QA/QC requirements presented in the following sections are intended to provide a common foundation for the analytical laboratory's protocols. It should be noted that the QA/QC requirements specified represent the minimum requirements for any given analytical method. Additional requirements that are method-specific should always be followed, as long as the minimum requirements presented in this document have been met.

Two criteria must be met for any analytical methods used:

1) Sufficient sensitivity must be obtained to achieve the required reporting objectives for any target analytes. The confidence of these reporting requirements is estimated by assessing the analytical variation resulting from repeated analyses of spiked samples close to these levels (sensitivity criteria).

2) Analyses of certified reference materials (CRMs) must yield values within the specified range of the certified values. Due to the inherent variability in analyses near the method detection limit (MDL), control limit criteria for relative accuracy only apply to analytes having CRM true values that are >10 times the MDL established by the laboratory (accuracy criteria).

The laboratory must review their performance and make corrections if QA/QC criteria are not met. Evaluation of performance is continuously evaluated based on participation in inter-laboratory comparison exercises (round-robins), analysis of CRMs or Laboratory Control Standards (LCSs), inter-calibration samples, spiked samples, sample duplicates, and laboratory reagent blanks. Laboratory personnel should review the results for all QA/QC samples immediately following the analysis of each sample batch. These results then should be used to determine whether or not control limit criteria have been met. If corrective actions must be taken, they must be completed before processing subsequent sample batches.

10.1 Sample Tracking and Handling

All samples are kept properly chilled and are transferred to the analytical laboratory within holding times to achieve the highest quality data possible. To ensure proper tracking and handling of the samples, documentation must accompany the samples from the initial collection to the final extractions and analysis. Minimum documentation includes:

1) Sampling Analysis Request Form

2) Chain-of-Custody Form(s)

These forms (or equivalents) will be used to track and handle samples.

It is imperative to assuring quality data results that the analytical laboratory provide written confirmation of each and all analytical tests to be conducted, their respective detection limits, analytical methods, and costs before analyses are allowed to be conducted.

10.2 Initial documentation of method detection limits

Prior to the analysis of samples, the laboratory will calculate method detection limits (MDLs) for each analyte and establish an initial calibration curve for all analytes.

The MDL will be used to demonstrate the capability of a laboratory to reach the sensitivity required to measure a specific constituent and demonstrate acceptable precision. The MDL represents a quantitative estimate of low-level response detected at the maximum sensitivity of a method. The Code of Federal Regulations (40 CFR Part 136) gives the following definition: "the MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte." The calculated MDL is a function of method precision at low analyte concentrations.

The procedures specified in 40 CFR Part 136 (Federal Register, Oct. 28, 1984) are used to calculate nominal MDLs for each analytical method employed. Briefly, at least seven replicates of each representative matrix should be spiked at a concentration between one and five times the estimated detection limit (except for certain trace

metals; see below for details), or at the reporting level (RL, see below) as a default. The amount of sample (i.e., weight of sediment) used in calculating the MDL should match, as closely as possible, the amount of sample typically used. The mean and standard deviation of the replicates are used to compute the MDL by multiplying the standard deviation by the Student t value for the 99% confidence interval (for n = 7, t = 3.143).

10.3 Reporting levels

Reporting levels (RLs) are used to report concentrations of target analytes. The RL is the lowest quantitative value, which can be justified and reported in terms of calibration reliability. Values below the RL, but above the nominal MDL are reported when detected, but must be flagged or annotated using the footnote supplied for data reporting.

10.3.1 Performance criteria at the RL

The initial performance demonstration of precision near the RL can be derived from the MDL determination or separate analysis. The standard deviation of at least 7 replicates of clean matrix spiked at or near the RL should be < 0.35 times the RL. In order for test performance to be estimated for ongoing organics analyses, each sample batch should include at least one spike at or near the RL.

10.4 Initial calibration

Equipment should be calibrated prior to the analysis of each sample batch, after each major equipment disruption, and whenever on-going continuing calibration checks do not meet recommended control limit criteria.

10.4.1 Organics

Calibration range must be established for each constituent from a minimum of five analytical standards of increasing concentration. The calibration range should be well characterized and must be established prior to the analysis of samples. Only data that results from quantification within the demonstrated working calibration range may be reported by the laboratory without annotation (i.e., quantification based on extrapolation outside the calibration range is not acceptable). Samples with measured concentrations outside the calibration range should be diluted as appropriate, and

reanalyzed. For results below the lowest calibration point (RL), samples may be further concentrated, or results "flagged" (annotated) as MDL. The latter is acceptable only if: (1) sample extraction/concentration steps were sufficient to meet the target analyte RL goals of the study, or (2) matrix problems have required sample dilution.

10.4.2 Trace metals

ICP-AES and ICP-MS instruments are calibrated with a calibration blank and a minimum of one calibration standard.

10.5 Calibration verification

An initial calibration verification standard is analyzed at the beginning of each analysis following the calibration procedure to check the accuracy of the calibration. Standards are made from a source different from the source that is used for the calibration standards. The initial calibration is near the mid-range of the calibration and must be within ±10% of the true value when analyzed. ICP-AES and ICP-MS also require a second initial calibration standard of a substantially different concentration than the first initial calibration standard; the second initial calibration standard must also be within ±10% of the true value when analyzed.

For continuing trace metal measurements, the continuing calibration verification (CCV) verifies that the instrument stays in calibration throughout the analysis. The CCV is prepared in the same acid matrix as the calibration standard. It is analyzed after every ten samples and at the end of the run. The CCV can come from any source that is near the mid-range of the calibration.

For trace organics measurements using full scan GC/MS, instrument tuning needs to be performed by analyzing 50 ng of decafluorotriphenylphosphine (DFTPP) prior to use of the instrument. The fragmentation profiles from this analysis have to be within the EPA-recommended criteria (see USEPA SW-846). The initial instrument calibration performed to establish calibration ranges for specific analytes is checked through the analysis of calibration verification standards (i.e., calibration standard solutions) prior to analysis of each batch of samples. Calibration verification standard solutions used for the calibration checks should contain all the analytes of interest at concentrations at or near the mid-level of a multi-point calibration range.

If the control limit for analysis of the calibration verification standard is not met, the analyst(s) should identify and eliminate the source(s) causing the failure and perform another calibration verification. If problems persist, preventive maintenance or corrective actions must be performed. If the calibration verification criteria cannot be met, a new initial calibration must be performed. No sample analysis should begin until satisfactory calibration verification is achieved.

Calibration blanks (pure matrix used to prepare calibration standard solutions) shall be measured prior to analysis of samples to ensure that the instrument is free of contamination. Concentrations of all target analytes obtained from analysis of the calibration blanks should be below MDLs.

10.5.1 Method blanks

Method blanks (also called procedural blanks) are used to assess laboratory contamination during all stages of sample preparation and analysis. For both organic and inorganic analyses, one laboratory reagent blank should be run in every sample batch. The method blank should be processed through the entire analytical procedure in a manner identical to the samples. A reagent blank concentration equal to or greater than three times the MDL for one or more of the analytes of interest requires definitive corrective action to identify and eliminate the source(s) of contamination before proceeding with sample analysis. For trace organics, if the method blank contains any analyte with a measured concentration greater than the RL, all samples should be re-analyzed within the batch if that analyte is detected in samples. Concentrations lower than the RL should be reported, but not used to correct concentrations in the field samples.

10.5.2 Sample duplicates

Analysis of sample duplicates is used to assess the precision of an analytical method in quantifying target analytes and not required for all methods. The relative percent difference (RPD) between the sample and sample duplicate results is calculated as follows:

$$RPD = \frac{(C1 - C2)}{(C1 + C2)/2} \times 100$$

Where: C1 = the larger of the duplicate results for a given analyte, and C2 = the smaller of the duplicate results for a given analyte.

The data from this process are typically used to establish a statistical range with which the precision of subsequent analyses can be assessed.

10.5.3 Matrix spikes and matrix spike duplicates

A laboratory spiked sample matrix (commonly called a matrix spike or MS) and a laboratory spiked sample matrix duplicate (commonly called a matrix spike duplicate or MSD) will be used both to evaluate the effect of the sample matrix on the recovery of the compound(s) of interest and to provide an estimate of analytical precision. A minimum of one MS should be analyzed for 5 percent of samples. The matrix spike solution should contain all the analytes of interest. The final spiked concentration of each analyte in the sample should be between 10 and 100 times the MDL for that analyte, as previously calculated by the laboratory (unless the unspiked sample contains more than this amount in which case 1 to 5 times the pre-existing concentration in the sample).

Recovery data for the fortified compounds ultimately are intended to provide a basis for determining the prevalence of matrix effects in the samples analyzed during the project. Low matrix spike recoveries may be a result of matrix interference and further instrument response checks may not be warranted, especially if the low recovery occurs in both the MS and MSD, and the other QC samples in the batch indicate that the analysis was "in control". An explanation for low percent recovery values for MS/MSD results should be given in the cover letter accompanying the data package. Corrective actions taken and verification of acceptable instrument response must be included.

Analysis of the MS/MSD also is useful for assessing laboratory precision. The RPD between the MS and MSD results should be within the control limits for at least one result per batch. If results for any analytes do not meet the control limit criteria, calculations and instruments should be checked. A repeat analysis may be required to confirm the results.

10.5.4 Certified reference materials

CRMs generally are the most useful QC samples for assessing the accuracy of a given analysis (i.e., closeness of a measurement to the "true" value). CRMs can be used to assess accuracy because they have "certified" concentrations of the analytes of interest, as determined through replicate analyses by a reputable certifying organization using two independent measurement techniques for verification. In addition, the certifying organization may provide "non-certified" or "informational" values for other analytes of interest. Such values are determined using a single measurement technique, which may introduce unrecognized bias. Therefore, non-certified values must be used with caution in evaluating the performance of a laboratory using a method which differs from the one used by the certifying organization.

As an alternative, an LCS may be used in place of a CRM. An LCS is similar to a CRM in that it is a homogeneous matrix that closely matches the samples being analyzed. Although concentrations of the analytes of interest in these materials are not certified, they can be used to assess the precision (i.e., consistency) of a single laboratory, as well as to determine the degree of comparability among different laboratories. In practice, LCSs may be preferred for routine (i.e., day to day) analysis because CRMs are relatively expensive. However, CRMs still must be analyzed at quarterly intervals to provide a check on accuracy.

11.0 Health and Safety

In both field and laboratory operations, careful preparation, cleanliness, and orderly methodologies contribute to the reliability of the data and safety of the personnel. Nautilus Health & Safety policies are strictly enforced at all times. The Lab also maintains current Chemical Hygiene and Emergency Action Plans. Training on these policies occurs annually. The Safety Coordinator holds safety meetings monthly. These meetings act as reminders for specific aspects of safe work practices.

Monthly inspections are conducted by the Safety Coordinator to ensure and document that all safety equipment is in proper working order, and that staff members are properly utilizing the personal protective equipment provided. The San Diego Laboratory also undergoes annual safety inspections conducted by the California

Department of Environmental Health, and bi-annual inspections conducted by the City of San Diego.

Most laboratory personnel maintain First Aid and CPR certifications. The Lab Manager, Data Manager, Lab Supervisor, and QA Officer maintain current OSHA 40-hour HAZWOPER certification.

12.0 Waste Disposal

All samples and wastes are disposed of in accordance with local, state, and federal regulations. Any containers used for laboratory acids, solvents, effluents and other laboratory chemicals are disposed of according to established guidelines (ASTM, 1987).

Most water samples are discharged into the sewer system as permitted under the City of San Diego and City of Tacoma Water Utilities Department Industrial Waste Programs.

Under California Title 22 and WDOE 80-12, if a sample is characterized as hazardous by toxicity testing, both the remaining sample and its associated dilution water are returned to the client for disposal or packaged for hazardous disposal with a qualified firm through our laboratory. If previous arrangements have been made for Nautilus to dispose of the material, the waste is disposed of in accordance with state, federal, and local regulations.

Sediments from the continental US that are classified as non-hazardous based on chemistry and toxicity are disposed of by the local trash service. Sediment samples that exhibit elevated chemistry and toxicity, and sediments from outside the continental US are properly disposed of by a licensed hazardous waste disposal firm.

13.0 Glossary of Terms

Accelerated Testing -- Some NPDES permits require the client to perform additional repeated testing in a short timeframe when a permit limit or monitoring requirement has been exceeded. If the material continues to exhibit toxicity, the applicable regulatory agency may require a TIE or TRE.

Acute toxicity test -- A method used to determine the concentration of a substance that produces a toxic effect on a specified percentage of test organisms in a short

period of time (e.g., 96 hours). As a general guideline, lethality is the measure of acute toxicity. Acute toxicity is usually defined as TUa (see Toxicity Unit) = $100/LC_{50}$. Note that acute means short-term, not necessarily mortality.

Ambient toxicity -- Toxicity occurring in a receiving body.

American Society of Testing and Materials (ASTM) -- Publisher of some test methods used in the laboratory.

Bioassay -- A test used to evaluate the relative potency of a chemical by comparing its effect on a living organism with the effect of a standard preparation on the same type of organism. The term "bioassay" is commonly, though not technically correct, used interchangeably with the term "toxicity test."

Biological Oxygen Demand (BOD) -- A measure of the use of oxygen by organisms in a sample.

Chain-of-Custody Form (COC) – A legal tracing record documenting the time of sample collection, the number of samples collected, the possession of the samples during all phases of collection and transport, and the person responsible for holding the sample until testing occurs.

Chronic toxicity -- An adverse effect that is manifested following a long-term (by duration or life stage) exposure. A chronic effect can be lethality, growth, reduced reproduction, etc. Note that chronic means long in duration or significant in terms of the life stage that has been exposed.

Chronic toxicity test -- A method used to determine the concentration of a substance in water that produces an adverse effect on a test organism over an extended period of time or during a significant component of the lifespan. Both discrete and continuous endpoints are evaluated, such as mortality and growth, respectively.

Coefficient of Variation (CV) -- The standard statistical measurement of the relative variation of a distribution or set of data, defined as the standard deviation divided by the mean. Coefficient of variation is a measurement of precision within and among laboratories.

Confidence Limits or Interval (CL or CI) -- The limits or interval within which, at

some specified level of probability, the true value of a result lies. Typically LC_{50} values are reported with 95% confidence limits.

Control -- An exposure of test organisms to a known clean sample material (without toxicant), conducted concurrent to test exposures of all matrices to ensure animal health, procedural integrity, and facilitate interpretation.

Critical life stage -- The period of time in an organism's life span in which it is the most susceptible to adverse effects caused by exposure to toxicants, usually during early development (e.g., egg, embryo, larva). Chronic toxicity tests are often run on critical life stages to replace long duration, life cycle tests since toxic effects often occur during the critical life stage.

Death -- Defined as the lack of movement or reaction even after gentle prodding.

Definitive bioassay -- A bioassay designed to establish a precise concentration at which a particular endpoint occurs. Exposures for these tests are usually longer than screening or range-finding assays and usually incorporate multiple replicates and exposure concentrations.

Discharge Monitoring Report (DMR) -- US EPA's annual program of blind sample testing that evaluates the methods and data quality of all laboratories that perform NPDES permit testing.

Dilution allowance or dilution credit -- Allowance given to account for initial dilution of effluent into receiving waters. Becomes an important factor when calculating proper test concentrations for toxicity testing. See Zone of Initial Dilution.

Dilution water -- The laboratory waters to which the test substance is added (diluted) and in which the organisms undergo exposure.

Dissolved Oxygen (DO) – concentration of oxygen that is dissolved in a sample.

Dose Response Curve -- A mathematical representation of the response of test organisms to increasing concentrations of a toxicant/effluent.

Effect Concentration (EC) -- A point estimate (statistically or graphically derived) of the toxicant concentration that would cause a population to show an adverse discrete

effect, such as death or lack of fertilization, in a given time period (for example, 96-hour EC_{50}).

EC_{50} -- The estimated concentration of a substance that adversely effects 50 percent of a test population during continuous exposure over a specified period of time.

Environmental Laboratory Accreditation Program (ELAP) – A State of California program under the Department of Health Services that monitors and accredits environmental testing laboratories.

Exposure time -- Length of time a test organism is exposed to a test material.

False Negative -- Statistical term meaning that the test data produced a non-toxic result even though the sample is actually toxic.

False Positive -- Statistical term meaning that the test data produced a toxic result even though the sample is not toxic.

Flow-through tests -- Refers to the continuous or very frequent passage of fresh test solution through a test chamber with no recycling. Because of the large volume (often 400 L/day) of effluent normally required for flow-through tests, it is generally considered too costly and impractical to conduct these tests off-site.

Good Laboratory Practices (GLP) – The standards in accordance with which our Quality Assurance program and systems were developed.

Hazardous Waste Operations and Emergency Response (HAZWOPER) -- A training program designed to prepare laboratory and field staff for handling, storage, and transportation of hazardous materials.

Hazardous Waste Characterization Test -- A test to determine whether or not a particular material exceeds state toxicity guidelines and is therefore classified as hazardous waste for purposes of disposal. Other matrices, including corrosivity and flammability, must also be evaluated for compliance.

Hypothesis Testing -- A technique that determines which, if any, concentrations are statistically different from the control. Endpoints routinely determined from hypothesis testing are NOECs and LOECs.

Inhibition Concentration (IC) -- A point estimate (statistically or graphically derived) of the toxicant concentration that would cause a given percent reduction in a continuous biological measurement such as fecundity or growth. For example, an IC_{25} would be the estimated concentration of toxicant that would cause a 25% reduction in mean young produced or in growth.

Lethal Concentration (LC) -- Toxicant concentration producing death of test organism. For example, a 96-hour LC_{50} would be the test concentration at which 50% of exposed organisms die within 96 hours of exposure.

LC_{50} -- Concentration of a substance at which 50 percent of exposed organisms die within a specific time interval. Also referred to as the median lethal concentration (MLC).

Lowest Observed Effect Concentration (LOEC) -- The lowest toxicant concentration of an effluent or a toxicant in a chronic bioassay that causes an adverse effect statistically different from the control. Also referred to as the LOEL (Lowest Observed Effect Level).

Major Discharger -- Any permit(ee) with a design flow of 1.0 MGD (million gallons per day) or greater (municipal). Any permit(ee) which scores 80 or greater on the major/minor permit classification scale (industrial).

Material Safety Datasheets (MSDS) -- Designed to inform all laboratory personnel that handle chemicals and products of the risks associated with each chemical. These datasheets also give complete lists of the physical properties of a chemical, what personal protective equipment to use, as well as what to do in cases of spills or emergencies.

Maximum Acceptable Toxicant Concentration (MATC) -- Toxicant concentration that may be present in a receiving water body without causing significant harm to productivity or other uses. MATC is determined by long-term tests of either partial life cycle with sensitive life stages or a full life cycle of the test organism.

Method Control – Control treatment adjusted to reflect a specific method. Organisms are exposed to ensure that any deleterious effects are assignable to sample material (e.g., pH control to account for effects of pH adjustments).

Million Gallons per Day (MGD) – Million gallons per day of discharge.

Minimum Significant Difference (MSD) -- This is the magnitude of difference from the control where the null hypothesis (the sample is not toxic) is rejected in a statistical test comparing a treatment (sample concentrations) and a control. MSD is based on the number of replicates, control performance, and the power of the test.

Mixing Zone -- An allocated impact area in a water body where numeric water quality criteria can be exceeded as long as acutely toxic conditions are prevented. Also referred to as the ZID (Zone of Initial Dilution).

Neonate -- Recently hatched cladoceran (daphnids, e.g., *Ceriodaphnia dubia, Daphnia magna, Daphnia pulex*).

No Observed Effect Concentration (NOEC) -- The highest exposure concentration tested of an effluent or toxicant that does not cause adverse effect statistically different from the control. The NOEC must be a specific exposure concentration and not an estimate between concentrations (See Point Estimate Techniques).

No Observed Effect Level (NOEL) -- The highest measured continuous concentration of a sample or other toxicant that causes no observed effect on a test organism. The NOEL must be one of the concentrations tested.

The National Pollutant Discharge Elimination System (NPDES) – The system prescribed by Section 402 of the Clean Water Act (the basics for permitting point source discharges).

Point Estimate Techniques -- Statistical and graphical techniques, such as Probit Analysis, Interpolation Method, or Trimmed Spearman Karber Method, which are used to determine the sample concentration at which adverse effects occurred. For example, the LC_{50} is the concentration at which 50% mortality occurred. These regression methods do not require that a specific endpoint concentration be among tested concentrations as in hypothesis testing.

Point source -- A discrete conveyance such as a pipe, ditch, etc. contributing pollutants to the environment.

Pollutant -- A contaminant introduced into receiving water, which is subject to

technology-based, or water-quality based effluent limitations in the permit.

Publicly Owned Treatment Works (POTW) – Municipal wastewater treatment operations that may operate with primary, secondary, or tertiary treatment.

Quality Assurance (QA) -- Overall program, system, or management that assures quality work.

Quality Control (QC) -- The actual steps or procedures taken to assure quality data (double-checking data entry, etc.).

Reference Toxicant -- A chemical used to evaluate the consistency of response of a given species of test organisms over time. It is assumed that any change in sensitivity to the reference substance may indicate the existence of some similar change in degree of sensitivity to other chemicals/effluents whose toxicity is to be characterized. A greater than expected toxic response may indicate poor organism quality or problems with test procedures.

Regional Water Quality Control Board (RWQCB) – California agencies authorized by SWRCB to be responsible for issuing and enforcing discharge permits for point sources as well as general watershed monitoring and listing water bodies in their region for impairment. There are 9 regional boards in California.

Renewal test -- A test without continuous flow of solution, but with occasional renewal of a prescribed percentage of test solutions at some periodicity. (e.g. 24 hours)

Replicate -- Two or more duplicate tests, samples, organisms, concentrations, or exposure chambers. Note: Pseudo-replication means that one composited sample, or one batch of organisms is divided into multiple replicates to increase sample size for statistical power. Field replication occurs when multiple samples are collected from a single field site and tested separately to evaluate spatial variability.

Response -- The measured biological effect of exposure to the material tested. In acute toxicity tests this is usually death. In biostimulation tests this is usually biomass increase. In chronic toxicity tests this can be reduction in reproduction, fertilization, normal development, growth, and others, as well as death.

Screening test (also range-finding test) -- An abbreviated toxicity test with four or five widely spaced toxicant concentrations. Some Regional and State effluent biomonitoring programs stipulate its use. If lethality is observed in the screening test, a definitive test may be required to further characterize response.

Society of Environmental Toxicology and Chemistry (SETAC) -- Local and national professional organization encompassing the fields of aquatic and terrestrial toxicology.

Standard Operating Procedure (SOP) – Detailed descriptions of internal procedures.

State Water Resources Control Board (SWRCB) – California entity responsible for implementation of federal and state policies.

Static tests -- Toxicity tests with aquatic organisms in which no test solution is introduced following test initiation. Solutions remain unchanged throughout the duration of the test.

Static-renewal test -- A test method in which the test organisms are exposed to a fresh solution of the same concentration of sample approximately every 24 hours or other prescribed interval, either by transferring the test organisms from one test chamber to another, or by replacing all or a portion of the effluent solution in the test chambers.

Sublethal – non-lethal endpoints such as reproduction, growth, or fecundity.

Test Acceptability Criteria – For toxicity test results to be acceptable for compliance, the data must meet the minimum specific criteria as defined in the test method protocol.

Total Dissolved Solids (TDS) – The total amount of minerals, salts, metals, cations or anions dissolved in water. This includes anything present in water other than the pure water molecule (H_2O) and suspended solids.

Total Maximum Daily Load (TMDL) -- A TMDL is a calculation of the maximum amount of a pollutant that a water body can receive and still meet water quality objectives.

Total Organic Carbon (TOC) – Organic matter content is typically measured as total

organic carbon and dissolved organic carbon, which plays a major role in aquatic systems. It affects biogeochemical processes, nutrient cycling, biological availability, chemical transport and interactions. It also has direct implications in the planning of wastewater treatment and drinking water treatment.

Total Suspended Solids (TSS) --Suspended solids are any particles/substances that are neither dissolved nor settled in the water.

Toxic Substances Control Act (TSCA) -- The Toxic Substances Control Act was enacted to provide information about all chemicals and to control the production of new chemicals that might present an unreasonable risk of injury to health or the environment.

Toxicity -- Adverse effect to a test organism caused by constituents, single or in combinations. Toxicity is a result of concentration and time, modified by variables such as temperature, chemical form, and availability.

Toxicity test -- A measure of the toxicity of a chemical or other material using living organisms by determining the degree of response (survival, reproduction, growth, etc.) of an exposed organism to the whole sample.

Toxic Units (TUs) -- A measure of toxicity as determined by the acute toxicity units or chronic toxicity units. Higher TUs indicate greater toxicity. This measure is indirectly proportional to hypothesis and point estimate results and is a commonly used regulatory tool.

Toxic Unit Acute (TUa) – A federally mandated mathematical conversion of LC_{50} into a unitless relatable value. TUa = $100/LC_{50}$. Other definitions are state-specific; the California Marine equation = [log(100 - % Survival)]/1.7.

Toxic Unit Chronic (TUc) -- A mathematical conversion of an NOEC or NOEL into a unitless relatable value. TUc = 100/NOEC.

Toxicity Identification Evaluation (TIE) -- A set of procedures used to identify the specific constituent(s) responsible for sample toxicity. TIEs are a component of a TRE.

Toxicity Reduction Evaluation (TRE) -- A site-specific study conducted in a stepwise process designed to identify the causative agents of effluent toxicity, isolate the

sources of toxicity, evaluate the effectiveness of toxicity control options, and then confirm the reduction in effluent toxicity. TREs may include TIE testing as part of this process.

U.S. Environmental Protection Agency (US EPA) -- Federal agency responsible for developing and enforcing regulations that implement environmental laws enacted by Congress. EPA delegates to states and tribes the responsibility for issuing permits and for monitoring and enforcing compliance.

Washington Department of Ecology -- Washington State agency responsible for implementation of federal and state environmental policies.

Whole Effluent Toxicity (WET) -- The total toxic effect of an effluent measured directly with a toxicity test.

Zone of Initial Dilution (ZID) -- An allocated impact area or mixing zone, in a water body where numeric water quality criteria are modified to reflect a dilution factor.

QUALITY ASSURANCE MANUAL

Columbia Analytical Services, Inc.

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January 12, 2007

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DOCUMENT CONTROL

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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.

It is a policy at CAS 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. 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. The Quality System is established to meet the requirements of the National Environmental Laboratory Accreditation Conference (NELAC).

CAS 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.

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.

CAS is a network of laboratories. In addition to the Kelso, WA facility, to which this manual is applicable, CAS 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; and *EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5,* USEPA, 2001.

4.0 PROGRAM DESCRIPTION

The purpose of the QA program at CAS 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 CAS:

> "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."

In support of this mission, our QA program addresses all aspects of laboratory operations, including laboratory organization and personnel, standard operating procedures, sample management, sample and quality control data, calibration practices, standards traceability data, equipment maintenance records, method proficiency data (such as method detection limit studies and control charts), document control/storage and staff training records.

4.1 Facilities and Equipment

CAS features over 25,000 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, CAS 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
- Water Chemistry & General Chemistry Laboratories
- Semi-volatile Organics Sample Preparation Laboratories (3)
- Gas Chromatography/High Performance Liquid Chromatography Laboratory

- Gas Chromatography/Mass Spectrometry Laboratory
- Petroleum Hydrocarbon Laboratory
- Semi-volatile Organics Drinking Water Laboratory
- 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 System
- 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.

4.2 Technical Elements of the Quality Assurance Program

The Quality Assurance Program provides a platform on which technical operations are based. The program provides laboratory organization, procedures, and policies by which the laboratory operates. The necessary certifications and approvals administered by external agencies are maintained. 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, 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.

4.3 Operational Assessments

There are a number of methods used to assess the laboratory and its daily operations. In addition to the routine quality control (QC) measurements to measure quality, 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. On-time performance, report quality, training, and Quality Assurance are a few of the items that are used to assess performance from an external perspective. A frequent, routine assessment must also be made of the laboratory's facilities and resources in anticipation of accepting an additional or increased workload.

CAS 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.

4.4 Document Control

Procedures for control and maintenance of documents are described in the *SOP for Document Control (ADM-DOC_CTRL).* The procedures described in the SOP include distribution, tracking, filing, and copyrighting of CAS controlled documents. The requirements of the SOP apply to all standards preparation logbooks, instrument maintenance logbooks, run logbooks, standard operating procedures (SOPs), quality assurance manuals (QAMs), quality assurance project plans (QAPPs), Environmental Health & Safety (EHS) manuals, and other controlled CAS 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.

CAS 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).*

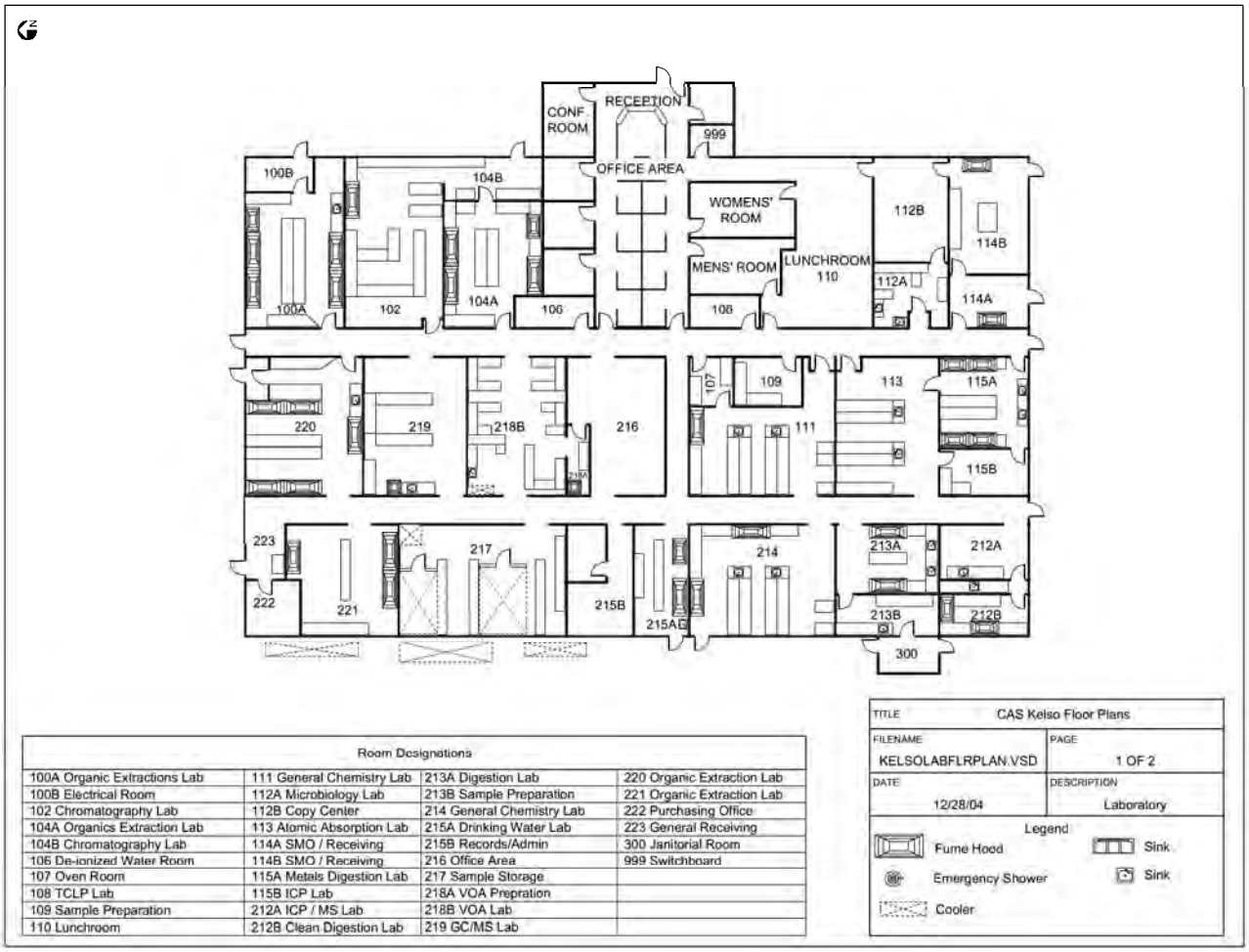
4.5 Subcontracting

Analytical services are subcontracted when CAS/Kelso needs to balance workload or when the requested analyses are not performed by CAS/Kelso. Subcontracting is only done with the knowledge and approval of the client. Subcontracting to another CAS 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 Quality Assurance Director is responsible for qualifying and oversight of subcontract laboratories.

4.6 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. The procedures for purchasing and procurement are described in the *SOP for Purchasing through CAS Purchasing Agent (SOP ADM-PUR).* Also, refer to section 10.4 for a discussion of reference materials.

Figure 4-1
CAS/Kelso Laboratory Floor Plan



5.0 PROFESSIONAL CONDUCT AND ETHICAL PRACTICES

One of the most important aspects of the success of CAS is the emphasis placed on the integrity of the data provided and services performed. To promote product quality, employees are required to comply with certain standards of conduct and ethical practices. The following examples of CAS 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 CAS 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 CAS *Confidentiality and Conflicts of Interest Employee Agreement* and the CAS *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.

CAS 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 CAS Employee Handbook. This includes the CAS Ombudsman Program, the CAS 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 CAS laboratory network additional capacity is typically available for subcontracting, if necessary.

6.0 ORGANIZATION AND RESPONSIBILITIES

The CAS/Kelso staff, consisting of approximately 110 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.

CAS is committed to providing an environment that encourages excellence. Everyone within CAS 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 CAS/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 CAS 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 CAS laboratories. The CQO is responsible for ensuring that annual internal audits are performed at each CAS laboratory; maintaining a data base of information about state certifications and accreditation programs; writing laboratory-wide SOPs; maintaining a data base of CAS-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 QA Director 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 CAS' 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 CAS laboratory and administrative staff to ensure that client-specific needs are understood, and that the services CAS 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.	28	Laboratory Director
Lee Wolf, B.S.	21	Quality Assurance Manager
Lynda Huckestein, B.S.	18	Client Services Manager Sample Management Office Manager
Jeff Coronado, B.S.	17	Inorganics Department Manager
Todd Poyfair, B.S.	15	Gas Chromatography and Petroleum Hydrocarbons Department Manager
Jeff Grindstaff, B.S.	18	Volatiles and Semivolatiles GC/MS Department Manager
Jim Smith, B.S.	19	Organics Drinking Water Department Manager
Eileen Arnold, B.A.	25	Environmental Health and Safety Officer
Paul Gowan, B.A.	20	Technical Information Specialist
Gary Ward, M.S.	31	CAS Information Technology Director CAS Chief Quality Officer
Steve Vincent, B.S.	31	CAS 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. CAS 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

CAS 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 configuration management plan are policies for controlling the software version that is in use in the laboratory.

7.2 IT Support

The local CAS 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, CAS corporate IT provides support for network-wide systems. CAS 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

CAS has various systems in place to address specific data management needs. The CAS Laboratory Information Management System (LIMS) is used to manage sample information and invoicing. Access is controlled by password. This system is used to establish and define sample identification, analysis specifications, and provide a means of sample tracking. This system is used during sample login to generate the internal Service Request. The Service

Request provides a summary of client information, sample information, required analyses, work instructions, deliverable requirements and other necessary information provided on the chain of custody. The LIMS also is the basis for valuable sample tracking mechanisms used throughout the laboratory. Laboratory analysts generate responsibility reports from the LIMS and perform internal chain of custody via the LIMS.

Where possible, instrument data acquired locally is immediately moved to a server (Microsoft Windows2003® domain) dedicated to this function. 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. Another server is dedicated to 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

CAS 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-CAS 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. CAS 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. CAS 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

CAS 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, IVA, IVB 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 (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 container, preservation and holding time information 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.

CAS routinely provides sample containers with appropriate preservatives for our clients. The containers are purchased as precleaned to a level 1 status, and conform to the requirements for analytical samples established by the USEPA. Certificates of analysis for the sampling 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 CAS 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 CAS. 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. Containers exhibiting an odor or abnormality after the second decontamination process are promptly and properly discarded. CAS keeps client-specific shipping requirements on file and utilizes major transportation carriers to guarantee that sample shipping requirements (same-day, overnight, etc.) are met. CAS also provides courier service that makes regularly scheduled trips to the Greater Portland, Oregon Metropolitan area.

When CAS 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 for maintaining the chain of custody of the sample(s) (SOP *for Chain of Custody for Sample Transfer between Laboratories* [SOP ADM-COC]), proper packaging and shipment, specification of proper methodology, etc. Blue or gel ice is the only temperature preservative used by CAS, unless otherwise specified by the client or receiving laboratory.

8.2 Sample Receipt and Handling

Standard Operating Procedures 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. Complete documentation of all sample storage is maintained in order to preserve the integrity of the samples.

Once samples are delivered to the CAS 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 kept refrigerated until they undergo analysis, unless otherwise specified. CAS stores samples in various refrigerators or freezers, depending on the type of analysis and the matrix of the sample. CAS has five walk-in refrigerators which house the majority of sample containers received at the laboratory. In addition to the walk-in refrigerators, there are four additional refrigerators, including dedicated refrigerated storage of VOC samples. These refrigerators are segregated by matrix type (soil or water) and method of analysis. The dedicated storage areas for VOC samples are monitored using storage blanks, as described in the *SOP for VOA Storage Blanks (VOC-BLAN)*. CAS also has six sub-zero freezers capable of storing samples at -20° C; these are primarily used for tissue and sediment samples requiring specialized storage conditions. The temperature of each sample storage unit used at CAS 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.

CAS adheres to the method-prescribed or project-specified holding times for all analyses. In order to comply with holding time requirements, the sampling date and time are entered into the LIMS system at the time of sample receipt and login. Each analyst then monitors 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. In order to report adherence to holding time requirements, 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 that holding time compliance can be demonstrated based on the analysis time. 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 retained 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 CAS.

Facility security and access is important in maintaining the integrity of samples received at CAS/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 CAS facility is equipped with an alarm system and CAS 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) to be 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 LIMS generated 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[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Bacterial Tests				
Coliform, Colilert	W, DW	P, Bottle or Bag	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Coliform, Fecal and Total	W, DW	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Fecal Streptococci	W	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Inorganic Tests				
Acidity	W	P,G	Cool, 4°C	14 days[EPA]
Alkalinity	W, DW	P,G	Cool, 4°C	14 days[EPA]
Ammonia	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Biochemical Oxygen Demand (BOD)	W	P,G	Cool, 4°C	48 hours
Bromate	W, DW	P,G	50mg/L EDA, cool to 4°C	28 days
Bromide	W, DW	P,G	None Required	28 days
Chemical Oxygen Demand (COD)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Chloride – Method 300.0	W, DW	P,G	None Required	28 days
Chloride – Method 9056	W	P,G	Cool, 4°C	Analyze immediately
Chlorine, Total Residual	W, DW	P,G	None Required	24 hours
Chlorite	W, DW	P,G	50mg/L EDA, cool to 4°C	14 days
Chlorophyll-A	W	G Amber	Cool, 4°C	Analyze immediately
Chromium VI – Method 7196A	W	P,G	Cool, 4°C	24 hours
Color	W, DW	P,G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	W, DW	P,G	Cool, 4°C, NaOH to pH>12, plus 0.6 g Ascorbic Acid	14 days
Cyanide, Weak Acid Dissociable	W	P,G	Cool, 4°C, NaOH to pH >12	14 days
Ferrous Iron	W, DW	G Amber	Cool, 4°C	24 hours
Fluoride – Method 300.0	W, DW	P,G	None Required	28 days
Fluoride – Method 9056	W	P,G	Cool, 4°C	Analyze immediately
Hardness	W, DW	P,G	HNO_3 to pH<2	6 months
Hydrogen Ion (pH)	W, DW	P,G	None Required	Analyze immediately
Kjeldahl and Organic Nitrogen	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrate – Method 300.0	W, DW	P,G	Cool, 4°C	48 hours
Nitrate – Method 353.2	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	48 hours
Nitrate – Method 9056	W	P,G	Cool, 4°C	Analyze immediately
Nitrate-Nitrite – Method 353.2	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrite – Method 300.0	W, DW	P,G	Cool, 4°C	48 hours

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Nitrite – Method 353.2	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	48 hours
Nitrite – Method 9056	W	P,G	Cool, 4°C	Analyze immediately
Orthophosphate – Method 365.3	W, DW	P,G	Cool, 4°C	Analyze immediately
Oxygen, Dissolved (Probe)	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	W, DW	P,G	Protect from temp. extremes	28 days
Phenolics, Total	W	G Only	Cool, 4°C, H_2SO_4 to pH<2	28 days
Phosphorus, Elemental	W	G Only	Cool, 4°C	48 hours
Phosphorus, Total	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Residue, Total	W	P,G	Cool, 4°C	7 days
Residue, Filterable (TDS)	W	P,G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	W	P,G	Cool, 4°C	7 days
Residue, Settleable	W	P,G	Cool, 4°C	48 hours
Residue, Volatile	W	P,G	Cool, 4°C	7 days
Silica	W	P Only	Cool, 4°C	28 days
Specific Conductance	W, DW	P,G	Cool, 4°C	28 days
Sulfate – Method 300.0	W, DW	P,G	Cool, 4°C	28 days
Sulfate – Method 9056	W	P,G	Cool, 4°C	Analyze immediately
Sulfide	W	P,G	Cool, 4°C, Add Zinc Acetate plus Sodium Hydroxide to pH>9	7 days
Sulfite	W	P,G	None Required	24 hours
Surfactants (MBAS)	W	P,G	Cool, 4°C	48 hours
Tannin and Lignin	W	P,G	Cool, 4°C	28 days
Temperature	W	P,G	None Required	Analyze immediately
Turbidity	W, DW	P,G	Cool, 4°C	48 hours
Metals				
Metals, except CrVI and Mercury	W, DW	P,G	HNO_3 to pH<2	6 months
	S	G, Teflon-Lined Cap	Cool, 4°C	6 months
Chromium VI – Method 7195	W	P,G	Cool, 4°C	24 hours
Mercury	W	P,G	HNO_3 to pH<2	28 days
	S	P,G	Cool, 4°C	28 days

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
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 (TOC)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Halogens, Total (TOX)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH<2, No headspace	28 days
Organic Halogens, Adsorbable (AOX)	W	G, Teflon-Lined Cap	Cool, 4°C, HNO_3 to pH<2	6 months
Petroleum Hydrocarbons, Total	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
Volatile Organics				
Petroleum Hydrocarbons, Volatile (Gasoline-Range Organics)	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	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.

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE)	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	W	G, Teflon-Lined Septum Cap	Adjust pH to 4-5, Cool, 4°C, No Headspace	14 days
EDB and DBCP	W,S	G, Teflon-Lined Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	28 days
Semivolatile Organics				
Petroleum Hydrocarbons, Extractable (Diesel-Range Organics)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction
Alcohols and Glycols	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Phenols	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Phthalate Esters	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Nitrosamines	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	W,S	G, Teflon-Lined Cap	Cool, 4°C	7 days until extraction;[f] 40 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Nitroaromatics and Cyclic Ketones	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction
Polynuclear Aromatic Hydrocarbons	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7 days until extraction;[f] 40 days after extraction
Haloethers	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Hydrocarbons	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Organophosphorus Pesticides	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Nitrogen- and Phosphorus-Containing Pesticides	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Herbicides	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7 days until extraction;[f] 40 days after extraction
Chlorinated Phenolics	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	W	G, Teflon-Lined Cap	NaOH to pH $\geq$10, Cool, 4°C[g]	30 days until extraction; 30 days after extraction
Drinking Water Organics				
Purgeable Organics	DW	G, Teflon-Lined Septum Cap,	Ascorbic Acid, HCl to pH$\leq$2, Cool, 4°C, No Headspace	14 days
EDB, DBCP, and TCP	DW	G, Teflon-Lined Septum Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	14 days
Carbamates, Carbamoyloximes	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	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	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH$\leq$ 2; Cool, 4°C	14 days until extraction; 30 days after extraction

Table 8-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Diquat and Paraquat	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	DW	G, Amber, Teflon-Lined Cap	Cool, 4°C	7 days until extraction; 14 days after extraction
Glyphosate	DW	G, Amber, Teflon-Lined Cap	100 mg/L $Na_2S_2O_3$, Cool, 4°C	14 days
Haloacetic Acids	DW	G, Amber, Teflon-Lined Cap	100 mg/L NH_4Cl, Cool, 4°C	14 days until extraction; 7 days after extraction
Semivolatile Organics	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH$\leq$ 2; Cool, 4°C	14 days until extraction; 30 days after extraction
Toxicity Characteristic Leaching Procedure (TCLP)				
Mercury	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	HW	P,G	Sample: Cool, 4°C TCLP extract: HNO_3 to pH<2	180 days until extraction; 180 days after extraction
Volatile Organics	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
Semivolatile Organics	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	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	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

a See Section 18.0 for sources of holding time information.
b DW = Drinking Water, W = Water; S = Soil or Sediment; HW = Hazardous Waste
c P = Polyethylene; G = Glass
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 INC.
An Employee-Owned Company

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
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 (FID) ☐ 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, NO2, NO3, BOD, TSS, TDS (circle)	NH3-N, COD, Total-P, TKN, TOC, DOC (circle) NO2+NO3	TOX 9020 ☐ AOX 1650 ☐ 506 ☐	REMARKS

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

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

*INDICATE STATE HYDROCARBON PROCEDURE: AK CA WI NORHTWEST 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 04/02

Figure 8-2

Columbia Analytical Services Inc.
Cooler Receipt and Preservation Form

Project/Client_____Work Order K060_____

Cooler received on _____ and opened on _____by_____

1.	Were custody seals on outside of coolers?	Y	N
	If yes, how many and where?_____		
2.	Were seals intact and signature & date correct?	Y	N
3.	Is the shipper's airbill available and filed? If no, record airbill number:_____	Y	N
4.	COC# _____ _____ _____ _____		
	Temperature of cooler(s) upon receipt: _____ _____ _____ _____		
	Temperature Blank: _____ _____ _____ _____		
5.	Were custody papers properly filled out (ink, signed, etc.)?	Y	N
6.	Type of packing material present_____		
7.	Did all bottles arrive in good condition (unbroken)?	Y	N
8.	Were all bottle labels complete (i.e analysis, preservation, etc.)?	Y	N
9.	Did all bottle labels and tags agree with custody papers?	Y	N
10.	Were the correct types of bottles used for the tests indicated?	Y	N
11.	Were all of the preserved bottles received at the lab with the appropriate pH?	Y	N
12.	Were VOA vials checked for absence of air bubbles, and if present, noted below?	Y	N
13.	Did the bottles originate from CAS/K or a branch laboratory?	Y	N
14.	Are CWA Microbiology samples received with >1/2 the 24hr. hold time remaining from collection?	Y	N
15.	Was C12/Res negative?	Y	N

Explain any discrepancies:_____
_____
_____
_____
_____
_____

RESOLUTION:_____

Samples that required preservation or received out of temperature:

Sample ID	Reagent	Volume	Lot Number	Bottle Type	Rec'd out of Temperature	Initials

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

CAS employs methods and analytical procedures from a variety of 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 CAS is described in SOPs specific to each method. A list of SOPs and NELAC-accredited methods are given in Appendix E. Further details are described below.

9.1 Standard Operating Procedures (SOPs) and Laboratory Notebooks.

CAS 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 Assurance 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 *Making Entries into Logbooks and onto Benchsheets* SOP (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 SOP 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 departure 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

CAS 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 CAS 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 (a.k.a. Laboratory Fortified Blank)

      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

CAS 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 sensitivity (provide lower detection limits), selectivity (minimize interferences while maintaining sensitivity), and overall data quality for low concentration applications. Examples are reductive precipitation metals analysis, specialized GC/MS analyses, and ultra-low level pesticides and PCBs.

9.6 Sample Cleanup

CAS commonly employs several cleanup procedures to minimize known common interferences prior to analysis. EPA methods 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 CAS 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. Records are maintained to provide traceability of reference materials.

Any item of the equipment which has been subjected to overloading or mishandling, or has been shown by verification or otherwise to be defective; is taken out of service until it has been repaired. The equipment is placed back in service only after verifying by calibration that the equipment performs satisfactorily. An evaluation of the effect of this defect on previous calibrations or tests is made and documented appropriately.

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. For NELAP accredited methods, the concentration of calibration verification standards are varied within the calibration range periodically.

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 of these thermometers is checked annually against a National Institute of Standards and Technology (NIST) certified thermometer. The NIST thermometer is recertified by a professional metrology organization on an annual basis.

10.2 Analytical Balances

Analytical balances are serviced on a semi-annual basis by a professional metrology organization. New certificates of calibration for each balance are issued to the laboratory on a semi-annual basis. 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 within 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 weights are recertified using NIST traceable standards by a professional metrology organization on an annual basis.

As needed, the balances are recalibrated using the manufacturers recommended operating procedures. The procedure for performing these measurements and use of acceptance criteria is described in the SOP ADM-SEMC.

10.3 Water Purification System

The water purification system is designed to produce deionized water with specific resistivity no less than 18 megohms-cm at 25°C, meeting specifications for Type I water, as described in *Standard Methods for the Examination of Water and Wastewater (SM1080)*. The system is monitored continuously for conductivity and resistivity with an on-line meter, which is recorded daily in a bound record book. The accuracy of the meter is verified annually. Deionizers are rotated and replaced on a regular schedule, and are monitored by an indicator light on the unit. The status of the system is monitored and recorded daily in a bound record book following a written SOP. Activated carbon filters are also in series with the demineralizers to produce "organic-free" water. Water for microbiology is checked at a point downstream of the purification system at a tap in the laboratory, and the monitoring documented.

10.4 Source and Preparation of Standard Reference Materials

All analytical measurements generated at CAS 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. 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.

Consumable reference materials routinely purchased by the laboratories (e.g., analytical standards) are purchased from nationally recognized, reputable vendors. All vendors have fulfilled the requirements for ISO 9001 certification and/or are accredited by A_2LA. CAS 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 examples 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. Compounds selected as system performance check compounds (SPCCs) must show a method-specified response factor in order for the calibration to be considered valid. Calibration check compounds (CCCs) must also meet method specifications for percent difference from the multipoint calibration. 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 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 CAS include total phenolics, phosphates, surfactants and tannin-lignin.

10.11 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 CAS acceptance limits are used to evaluate the calibration curve prior to sample analysis.

10.12 Ion Chromatographs

Calibration of the ion chromatograph (IC) involves generating a 5-point calibration curve. 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 bromide. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method.

10.13 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.

10.14 Ion-selective electrode

The method-prescribed number of standards (typically two for Standard Methods and EPA wastewater methods) 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.

10.15 Pipets

The calibration of pipets and autopipettors used to make critical-volume measurements is verified following the *SOP for Checking Pipet Calibration*. 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.

10.16 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 Services Quality Assurance (QA) Program is to ensure the accuracy, precision and comparability of all analytical results. Prior to using a procedure for the analysis of field samples, acceptable method performance is established by performing demonstration of capability analyses and performance characteristics are established by performing method detection limit studies and assessing accuracy and precision according to the reference method. CAS 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 methodology or are statistically derived based on the laboratory's actual historical data obtained from the various QC measurements for each analytical method. The Quality Control objectives are defined below.

11.1 Quality Control Objectives

11.1.2 Demonstration of Capability - Where required by mandatory test method, regulation, or accreditation protocols, a demonstration of capability (DOC) is made prior to using any test 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 reference material or quality control sample is obtained. 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 or MDL study 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). CAS 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. The current acceptance limits for accuracy and precision are available from the laboratory and on the accompanying CD-ROM. 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. CAS 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.

11.1.8 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 CAS objective is to generate data of known quality with the highest level of comparability, completeness, and usability to the client. 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 specified data qualifiers where necessary.

11.2 Method Detection Limits and Method Reporting Limits

Method Detection Limits (MDL) for analytical methods routinely performed at CAS/Kelso are determined annually, thus may change slightly from year to year. The MDLs are determined by following the *SOP for the Determination of Method Detection Limits,* which is based on the procedure in 40 CFR Part 136, Appendix B. As required by NELAC and DoD protocols, the validity of derived MDLs is verified using MDL 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. the lower limit of quantitation). Therefore, analyses are calibrated to the MRL, or lower. To take into account day-to-day fluctuations in instrument sensitivity, analyst performance, and other factors, the MRL is established at three times the MDL (or greater). The 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. In addition, a number of other quality control processes that may impact analytical results are also described below.

11.3.1 Method Blank (a.k.a. Laboratory Reagent Blank)

The method blank is either analyte-free water or analyte-free soil (when available), subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand, or an acceptable substitute may be used instead. The method blank is analyzed to demonstrate that the analytical system itself is not contaminated with the analyte(s) being measured. The method blank results should be below the Method Reporting Limit (MRL) or, if required for DoD projects, below half of the 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.

11.3.9 Laboratory Control Samples (a.k.a. Laboratory Fortified Blanks)

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.

11.3.10 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.

11.3.11 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:

$$\textit{Relative Percent Difference (RPD)} = (S1 - S2) \times 100 \div S_{ave}$$

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.

11.3.12 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.

11.3.13 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.

11.3.13 Control Charting

The generation of control charts is routinely performed at CAS. 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).

11.3.14 Glassware Washing

Glassware washing and maintenance play a crucial role in the daily operation of a laboratory. The glassware used at CAS undergoes a rigorous cleansing procedure prior to every usage. A number of SOPs have been generated that outline the various procedures used at CAS; 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

CAS 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 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

The integrity of the data generated is assessed through the evaluation of the results of the calibrations and analysis of 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. Details are provided in applicable SOPs. The numerical criteria for evaluation of these QC samples are listed within each method-specific Standard Operating Procedure. 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 NELAC Quality Systems requirements. Additional requirements exist for certain programs, such as projects under the DoD QSM protocols, AFCEE QAPP 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. For DoD projects, the concentration of these two consecutive must be varied as required by the DoD QSM, Version 3.

- 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) it is verified that the result is within the calibration range. If not, the sample is diluted and analyzed to bring the result into calibration range. For sample and sample duplicate 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 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. The MRL must meet project requirements.

- 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. It is verified that the result is within the calibration range. If not, the sample is diluted and analyzed to bring the result into calibration range. For GC and HPLC tests, results from confirmation analysis are evaluated to confirm positive results and to determine the reported value. If obvious matrix interferences are present, additional cleanup of the sample using appropriate procedures may be necessary and the sample is reanalyzed. 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. The MRL must meet project requirements.

- Surrogate Results (Organic) – Following sample analysis and calculations the percent recovery of each surrogate is compared to specified control limits. If recoveries are acceptable and other sample evaluation is complete, 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. The source of the problem is identified and, depending on the source of the problem, the LCS and the associated batch is reanalyzed or re-prepared and reanalyzed.

- 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. CAS 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 CAS-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 CAS client.

12.5 Documentation

CAS 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 computer-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.

12.5.2 Documentation of Batch-related QC and Calibration Data

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, CAS provides several levels of analytical reports. Basic 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 HTRW specified data package and reporting requirements
- AFCEE – Air Force Center for Environmental Excellence project-specific reporting

When requested, CAS provides Electronic Data Deliverables (EDDs) in the format specified by client need or project specification. 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 CAS 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 CAS/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 CAS/Kelso are conducted regularly by various regulatory agencies and clients. Table 13-1 summarizes some of the major programs in which CAS/Kelso participates. Programs and certifications are added as required. Additionally, internal system audits of CAS/Kelso are conducted regularly by 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 30 days. 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 30 day 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 and files, 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

CAS/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. CAS 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 CAS Quality Assurance Director. For any results outside acceptance criteria, the analysis data is reviewed to identify a possible cause for the deficiency, and corrective action is taken and documented.

Table 13-1
Current CAS Performance and System Audit Programs

Federal and National Programs

- Naval Facilities Engineering Service Center
  Validated Laboratory for NFESC Parameters
- U.S. Air Force, Air Force Center for Environmental Excellence (AFCEE)
  Approved Laboratory for AFCEE Projects
- U.S. Army Corps of Engineers - MRD, HTRW Center of Expertise
  Validated Laboratory for HTRW parameters
- National Environmental Laboratory Accreditation Conference (NELAC) Accredited

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 NELAC Accreditation No. E87412
- 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. LA070008
- 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
- 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

Table 13-1 (continued)

State and Local Programs (continued)

- 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 Pennsylvania Department of Environmental Protection
  Registered Environmental Laboratory
- 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 CAS (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 CAS contain extensive information about the instruments used at the laboratory.

An initial demonstration of analytical control is required on every instrument used at CAS 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).

Equipment records also include a copy of the manufacturer's manual(s) and dates and results of calibrations.

Preventive maintenance procedures, frequencies, etc. are available for each instrument used at CAS. 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

To the extent possible, samples shall be reported only if all quality control measures are acceptable. 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 may be notified in order to ascertain if contact with the client is necessary.

Problems with analysis, as well as the corresponding corrective actions taken, are documented on Nonconformity and Corrective Action Reports (See Figure 15-1) following the requirements in the *SOP for Nonconformity and Corrective Action Documentation* (SOP No. ADM - NCAR). This form is utilized 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.

Corrective action due to a performance audit or a check sample problem is initiated by the Quality Assurance Manager; the affected laboratory supervisors and managers are promptly informed of performance audit results requiring corrective action.

Figure 15-1

Nonconformity and Corrective Action Report

NONCONFORMITY

N&CA Report No. _____

PROCEDURE (SOP or METHOD): EVENT DATE: _____

EVENT: ☐ Missed Holding Time ☐ QC Failure ☐ Lab Error (spilled sample, spiking error, etc.)
☐ Method Blank Contamination ☐ Login Error ☐ Project Management Error
☐ Equipment Failure ☐ Unacceptable PT Sample Result ☐ Other (describe):

SAMPLES / PROJECTS / CUSTOMERS / SYSTEMS AFFECTED

DETAILED DESCRIPTION

ORIGINATOR: _____ DATE: _____

PROJECT CHEMIST(s): _____ NOTIFIED BY: _____ DATE: _____

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 Chemist 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:

Customer Notified by ☐ Telephone ☐ Fax ☐ E-mail ☐ Narrative ☐ Not notified
Project Chemist Verification and Approval of Corrective Action _____ Date: _____
Comments: (Retain record)

Page 1 of 1
Original: Customer File Photocopies: Supervisor and QA PM N&CA Report 2004.doc 3/26/2004

16.0 QUALITY ASSURANCE REPORTS

Quality assurance requires an active, ongoing commitment by CAS personnel at all levels of the organization. Information flow and feedback mechanisms are designed so that analysts, supervisors and managers are aware of quality assurance issues in the laboratory. Analysts performing routine testing are responsible for generating a Data Quality Report (DQR), or similar form, with every analytical batch they process. This report contains explicit documentation of the various controls that must be met during the analysis. This report also allows the analyst to provide appropriate notes and/or a case 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 corrected if possible.

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. The project chemist must also review the entire body of data to ensure that any and all client-specified objectives were successfully achieved. A case narrative may be 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.

Any operational or quality assurance problems noted by the Laboratory Director are then addressed during the senior staff operations meetings with all appropriate department managers. The Laboratory Director also performs an annual documented review of the laboratory quality system to identify any necessary changes or improvements to the quality system or quality assurance policies.

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 CAS are evaluated, in part, against the appropriate technical description.

Training begins the first day of employment at CAS 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 CAS. 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).

Each employee participates in Ethics training, which is part of the CAS Improper Practices Prevention Program. CAS 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.

A training plan is developed for each Standard Operating Procedure. The training plan includes a description of the step-by-step process for training an employee and for initial demonstration of proficiency. Where the analyst performs the entire procedure, a generic training plan may be used. In cases where work cells are used, a training plan specific to the work cell is established.

17.1 Initial Demonstration of Capability

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. (For use of this option, LCSs must be from "second-source" standard materials independent of the calibration standards materials.).

- 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 PT sample analyses using the test method, or a similar test method using the same technology.

- Performing Initial Demonstration of Capability as described above, with acceptable levels of precision and accuracy.

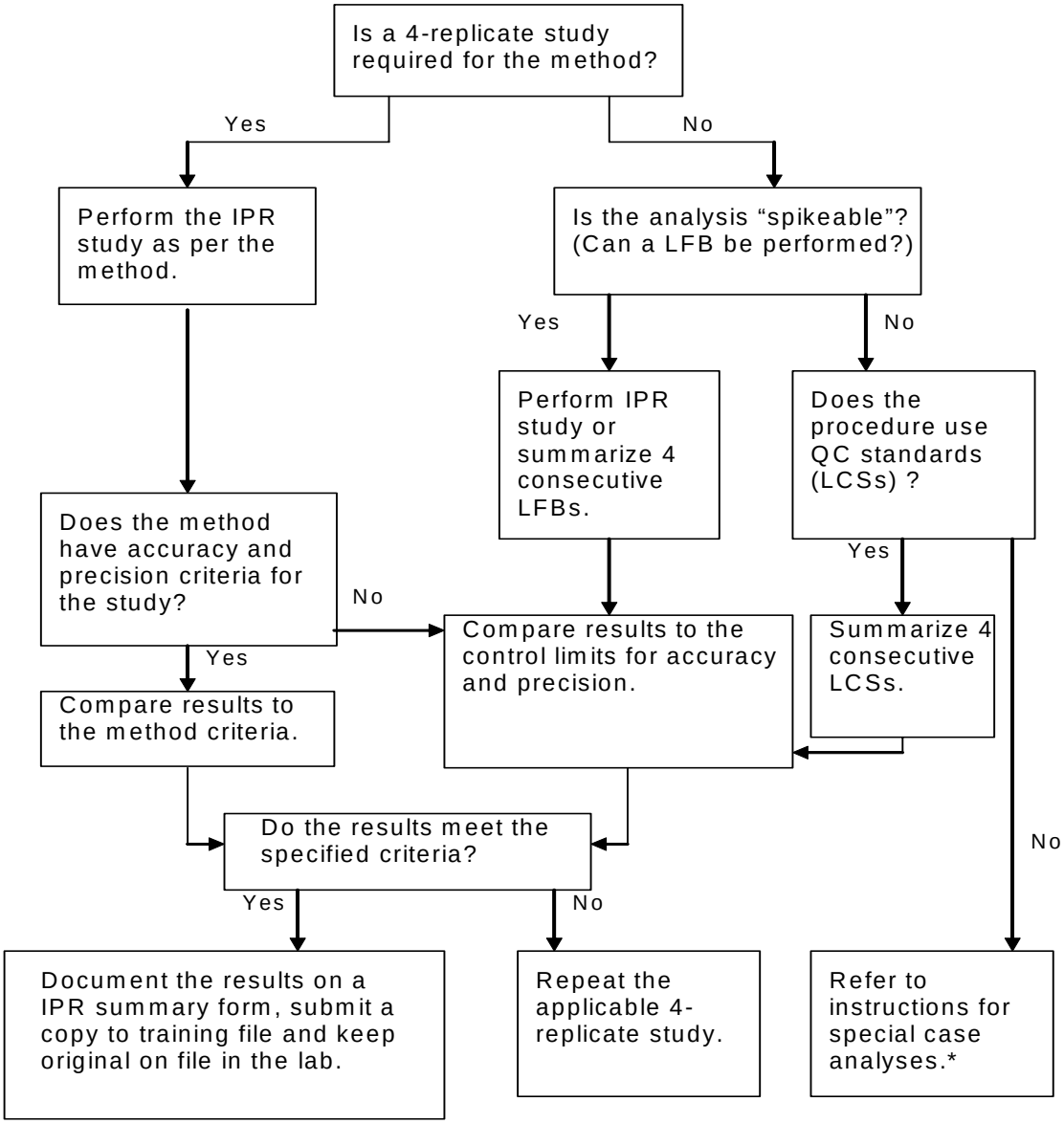
- Analysis of at least 4 consecutive LCSs with acceptable levels of accuracy and precision from in-control analytical batches.

- 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 CAS resumes. A database is used to record the various technical skills and training acquired while employed by CAS. 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 NELAC 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, etc.): Supervisor acknowledgement of training and approval.

18.0 REFERENCES FOR ANALYTICAL PROCEDURES

The analytical methods used at CAS 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 CAS are taken from the following references:

- *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), Proposed Update IV, and updates posted online at http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm. See Chapters 1, 2, 3, and 4. Unless otherwise specified, the most current published version is used.

- *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*, 16th Edition (1985); 17th Edition (1989); 18th Edition (1992); 19th Edition (1995). 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. OLM01.8, OLM02.0, 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).
- *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).
- National Environmental Laboratory Accreditation Conference (NELAC), 2003 Quality Standards.
- *Department of Defense Quality Systems Manual for Environmental Laboratories*, Final Version 2 (June 2002) and Final Version 3 (January 2006).

APPENDIX A

LIST of QA PROGRAM DOCUMENTS

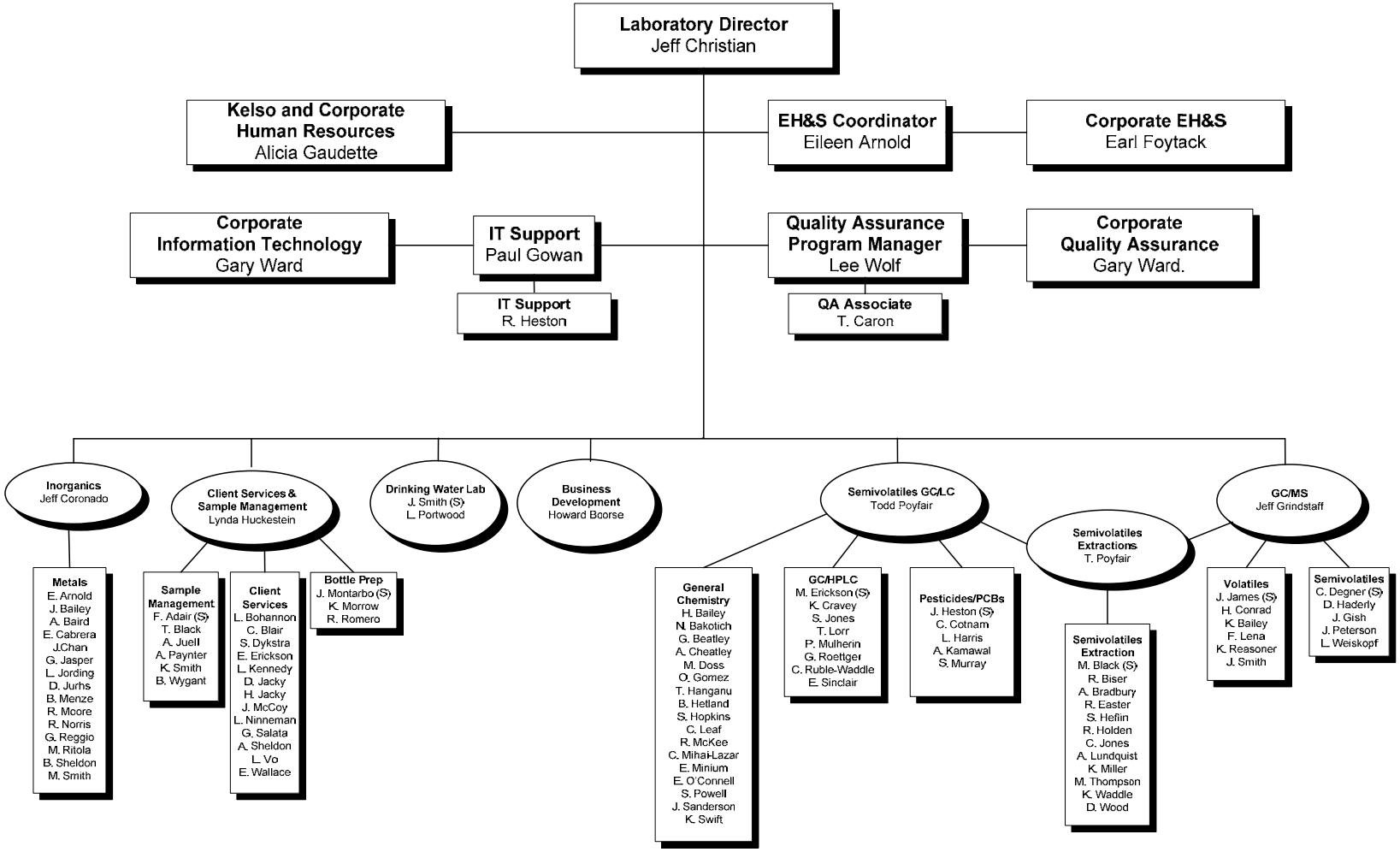
Quality Assurance Manual	1/10/07
Software Quality Assurance Plan	7/11/05
CAS-Kelso Certifications/Accreditations	Cert_kel.xls
Columbia Analytical Services MDL Tracking Spreadsheet	Mdl_list.xls
Technical Training Summary Database	TrainDat.mdb
Approved Signatories List	AppSignatories.pdf
Personnel resumes/qualifications	HR Department
Personnel Job Descriptions	HR Department
Quality Control Acceptance Criteria	Qclimits.xls
Master Logbook of Laboratory Logbooks	Masterlog-001
TECHNICAL STANDARD OPERATING PROCEDURES	
SOP TABLE OF CONTENTS	SOPLIST.DOC
ADMINISTRATIVE STANDARD OPERATING PROCEDURES	
ADMINISTRATIVE - CORPORATE	**FILE NAME**
CHAIN OF CUSTODY FOR SAMPLE TRANSFER BETWEEN LABORATORIES	ADM-COC
CHECKING NEW LOTS OF CHEMICALS FOR CONTAMINATION	ADM-CTMN
CONTROL LIMITS	ADM-CTRL_LIM
DEALING WITH COMPLAINTS	ADM-CMPLT
DOCUMENT CONTROL	ADM-DOCCTRL
DOCUMENTATION OF TRAINING	ADM-TRANDOC
ELECTRONIC DATA AUDITING	ADM-E_DATAUDIT
MAKING ENTRIES INTO LOGBOOKS AND ONTO BENCHSHEETS	ADM-DATANTRY
MANUAL INTEGRATION OF CHROMATOGRAPHIC PEAKS	ADM-INT
NONCONFORMITY AND CORRECTIVE ACTION	ADM-NCAR
PREPARATION OF STANDARD OPERATING PROCEDURES	ADM-SOP
PURCHASING THROUGH CAS PURCHASING AGENT	ADM-PUR
QUALIFICATION OF SUBCONTRACT LABORATORIES	ADM-SUBLAB
SAMPLE BATCHES	ADM-BATCH
SIGNIFICANT FIGURES	ADM-SIG.FIG
THE DETERMINATION OF METHOD DETECTION LIMITS AND LODs	ADM-MDL

ADMINISTRATIVE – LOCAL LABORATORY	**FILE NAME**
ARMY CORPS OF ENGINEERS HTRW PROJECT MANAGEMENT	ADM-HTRW
CHECKING PIPET 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
ELECTRONIC DATA BACKUP AND ARCHIVING	ADM-EBACKUP
FACILITY AND LABORATORY CLEANING	ADM-FACL
INTERNAL QUALITY ASSURANCE AUDITS	ADM-IAUD
LABORATORY DATA REVIEW PROCESS	ADM-DREV
METHOD DETECTION LIMIT DOCUMENTATION AND CONTROL	ADM-MDLC
PROJECT MANAGEMENT	ADM-PCM
REAGENT LOGIN AND TRACKING	ADM-RLT
SUPPORT EQUIPMENT MONITORING AND CALIBRATION	ADM-SEMC
THERMOMETER CALIBRATION	ADM-TCAL
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

APPENDIX B

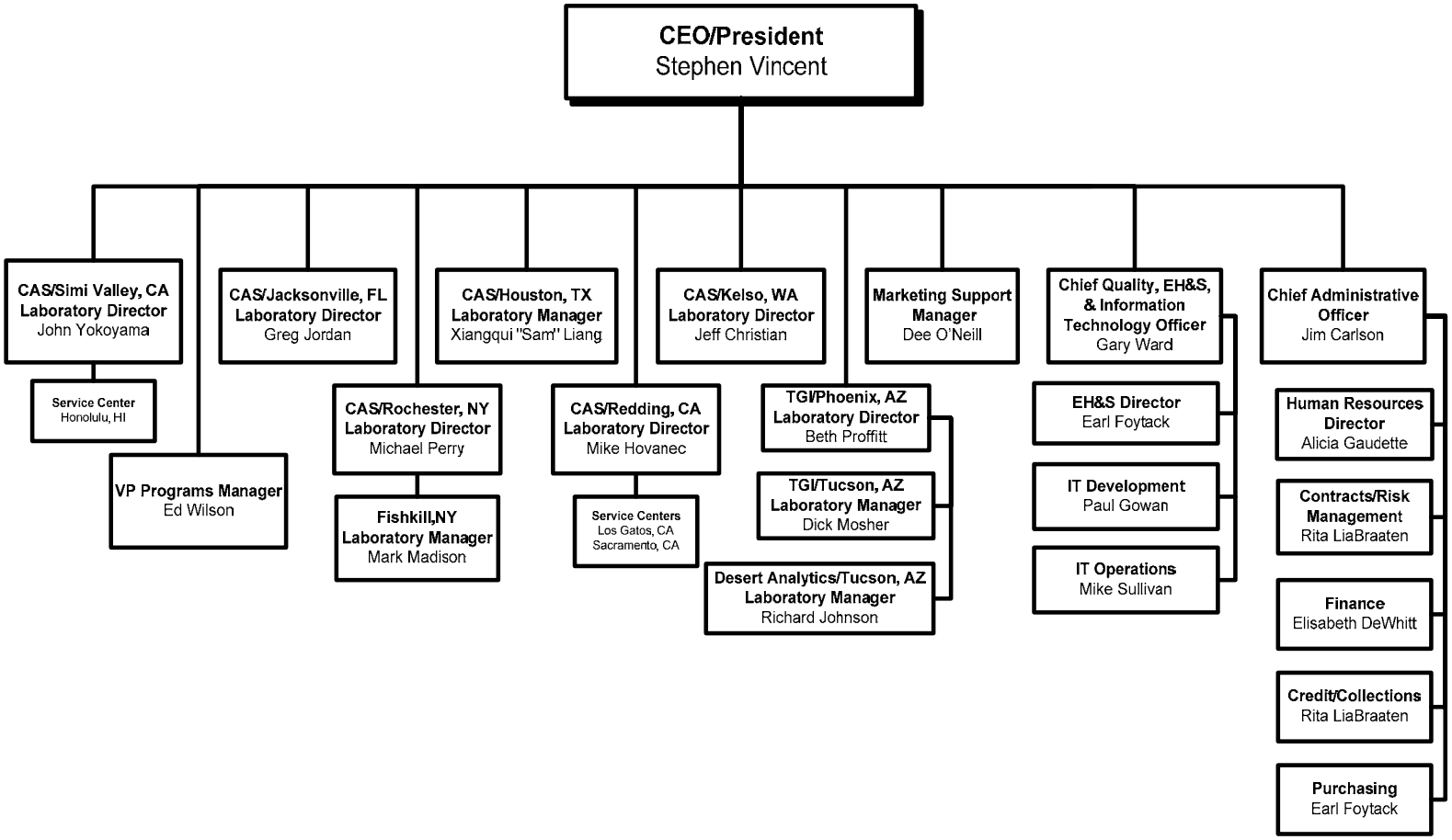
ORGANIZATIONAL CHARTS and RESUMES OF KEY PERSONNEL

Columbia Analytical Services, Inc.
Kelso, Washington Laboratory Organization



Revised 1/12/07

Columbia Analytical Services, Inc.
Laboratory Division Organization



JEFFREY D. CHRISTIAN

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, 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) and Redding (CA) facilities, 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

On request.

LEE E. WOLF

1988 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

TECHNICAL MANAGER IV, KELSO QUALITY ASSURANCE MANAGER – 2002 to Present

Responsibilities

Responsible for the overall coordination of the laboratory QA program, and for ensuring that established quality objectives are met. Responsible for Quality Assurance function, including the Quality Assurance Manual, certifications, documenting SOPs, and maintaining performance evaluation 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. Provides audit responses and initiates any changes in procedures resulting from an audit. Coordinate the analysis of performance evaluation samples required for certification/accreditation programs. Report and review results for these analyses. Conduct internal audits and make recommendations for corrective action.

Experience

Scientist IV, Kelso Quality Assurance Manager, *Columbia Analytical Services, Inc., Kelso, Washington,* 1996-2002. Duties primarily as listed above.

Project Chemist/Principal Organic Scientist, *Columbia Analytical Services, Inc., Kelso, Washington,* 1994-1996. Responsibilities included GC and GC/MS method development and special projects coordination. Acts as technical advisor to the GC and GC/MS laboratories and GC/MS interpretation specialist and CLP organics specialist. Also responsible for Project Chemist functions, including management and coordination of projects for clients, identifying client needs, and preparation of data reports.

Semi-VOA Department Manager, *Columbia Analytical Services,* 1988-1994. Responsibilities included overall management of the Semi-VOA department. Oversee the operation of Semi-VOA GC/MS, data review and reporting and related QA/QC function. Also responsible for supervision of staff, including training, scheduling, and other personnel issues. Beginning in 1992, increased responsibilities to include Project Chemist functions for organics EPA-SAS and other clients. This involved scheduling projects for clients, identifying client needs, and preparing data reports.

GC/MS Chemist, *U.S. Testing Co., Richland, Washington,* 1985-1988. Responsibilities included GC and GC/MS analysis of water and soil samples for volatiles and Semi-VOA by EPA protocol, including Methods 8240, 8270 and CLP. Coordinated extraction and GC-GC/MS areas to manage sample/data flow through the lab. Experience also with pesticide/PCB analysis by EPA Methods 8080 and CLP. Responsible for development of analysis methods for non-routine pesticides and herbicides and performed HPLC analysis.

Laboratory Assistant, *Eastern Washington University, Cheney, Washington,* 1985. Responsibilities included supervision and instruction of organic chemistry labs. Experience with GC and IR operation. Responsible for lab safety.

Chemist Assistant, *Spokane County Air Pollution Control Authority, Spokane, Washington,* 1984. Responsibilities included gathering and analyzing air samples for CO content using IR equipment.

Education

Documenting Your Quality System, *A2LA Short Course, Las Vegas, Nevada,* 1998.
Internal Laboratory Audits, *A2LA Short Course, Las Vegas, Nevada,* 1998.
Mass Spectra Interpretation, *ACS Short Course, Denver, Colorado,* 1992.
BS, Chemistry, Minor in Geology, *Eastern Washington University, Cheney, Washington,* 1985.

Publications/ Presentations

On Request.

Affiliations

American Chemical Society.

LYNDA A. HUCKESTEIN

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, 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 to 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.

JEFFREY A. CORONADO

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

TECHNICAL MANAGER IV, INORGANICS DEPARTMENT MANAGER – 2001 to Present

Responsibilities

Primary responsibilities include management of the Metals laboratory department. Responsible for training oversight, data review, report accuracy and timeliness QA/QC implementation, tracking department workload, and scheduling and performance of the Metals department. Also responsible for departmental budgets, method development efforts, 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.

JEFFREY A. GRINDSTAFF

1991 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

TECHNICAL MANAGER III, GC/MS VOA AND SEMI-VOA LABORATORIES – 1997 to Present

Responsibilities

Primary responsibilities include management of the GC/MS SemiVoa and VOA laboratory departments. Responsible for training oversight, data review, report accuracy and timeliness QA/QC implementation, tracking department workload, and scheduling and performance of the GC/MS departments. Also responsible for departmental budgets, method development efforts, and resource allocation. Also performs GC/MS maintenance and troubleshooting.

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 and instrument calibration, tuning, and maintenance. Also implemented and further developed EPA methods for quantitative analysis of pesticides and priority pollutants..

Education

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

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, Florida. 1996

Affiliations

American Chemical Society. 1989

TODD N. POYFAIR

1991 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

TECHNICAL MANAGER III, SVG LABORATORIES – 2001 to Present

Responsibilities

Primary responsibilities include management of the GC, HPLC, and General Chemistry laboratory departments. Responsible for training oversight, data review, report accuracy and timeliness QA/QC implementation, tracking department workload, and scheduling and performance of the these departments. Also responsible for departmental budgets, method development efforts, and resource allocation.

Documentation of Demonstration of Capabilities is available for review.

Experience

Supervisor/Manager, General Chemistry Department, *Columbia Analytical Services, Inc., Kelso, Washington,* 1995-2001. Responsibilities included supervision, management, and training of General Chemistry staff. Also responsible for workload coordination, data review, reporting, and instrument maintenance within the General Chemistry department.

Project Chemist, Client Services Group, *Columbia Analytical Services, Inc., Kelso, Washington,* 1993-1995. Responsibilities included technical project management and customer service. Responsible for meeting the clients' needs of timely and appropriate analyses, and to acted as liaison for all client-related activities within CAS.

Scientist II, General Chemistry Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-1993. Responsibilities included the review and summarization of pH, alkalinity, conductivity, turbidity, hardness, and CODs.

Scientist I, **General Chemistry Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1992. Responsibilities included analysis of Total Organic Halogens, Chemical Oxygen Demand, Sulfides, Ammonia, TKN, Nitrate/Nitrite by Lachat, and Cyanide.

Analyst III, **General Chemistry Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1991-1992. Responsibilities included analysis of pH, Conductivity, Alkalinity, Turbidity, and Oil and Grease.

Education

BS, Chemistry, *Portland State University, Portland, Oregon,* 1991.
BA, German, *Portland State University, Portland, Oregon,* 1990.
COURSEWORK, *Brigham Young University, Provo, Utah.* 1982-1983 & 1985-1986.

JAMES R. "JIM" SMITH

2001 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

TECHNICAL MANAGER I, DRINKING WATER LABORATORY SUPERVISOR – 2002 to Present

Responsibilities

Primary responsibilities include management of the Semivolatiles Drinking Water laboratory department. Responsible for training oversight, data review, report accuracy and timeliness QA/QC implementation, tracking department workload, and scheduling and performance of the department. Also responsible for departmental budgets, method development efforts, and resource allocation. Project management of Drinking water accounts. Operation of Varian GC/MS, Agilant GC/ECD and Agilant HPLC.

Documentation of Demonstration of Capabilities is available for review.

Experience

Project Manager III, *Columbia Analytical Services, Inc., Kelso, Washington,* 2001-2002. 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. Extensive technical experience with the various GC and GC/MS method allow for detailed technical review of organics projects. Also has extensive experience coordinating drinking water and sediment projects. Currently responsible for coordination of approximately $200,000 of analyses in the laboratory on a monthly basis. Current large clients include Bechtel (Navy work), which involves numerous groundwater-monitoring projects, the Port of Seattle and URS. Also responsible for various storm water studies for Bremerton Naval Shipyard.

Director, Trace Organics and Project Manager, *Amtest, Inc., Redmond, Washington,* 1987-2001. Responsible for project management, client contact, data review, and writing reports. Additional responsibilities pertained to supervision of the trace organics department and running the GC/MS system. Performed various methods by GC/MS for volatiles, semi-volatiles and by GC for pesticides, PCBs, herbicides, and fuels (LUST). Also performed hazardous waste characterization including completion of waste profile forms.

Education

BS, Chemistry, *Rochester Institute of Technology, Rochester, New York,* 1985.

EILEEN M. ARNOLD

1987 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, 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 Services 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 within 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, maintenance 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 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.

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.

PAUL GOWAN

1994 TO PRESENT

CAS Holdings Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

IT MANAGER II, 2002 to Present

Responsibilities

Identify and plan IT requirements by interacting with management personnel to identify current and long-term user objectives. Assist in developing and tracking the IT computer capital budget. Provide electronic data deliverable (EDD) and LIMS system guidance to management personnel throughout the company. Ensure the effective utilization of computer systems. Train users as required. Establish IT policies, standards, practices, and specifications. Participate in the LIMS planning group.

Plan and supervise IT department staffing, organization, hardware and software acquisitions to meet requirements. Acquire, develop, and maintain a skilled staff through effective performance review and career development programs. Develop and maintain relationships with local and national vendors of computer hardware, software, and telephone equipment.

Experience

KELSO IT MANAGER, *Columbia Analytical Services, Inc., Kelso, Washington,* 1999-2002. Responsible for all IT efforts related to the CAS laboratory in Kelso, WA, including, but not excluded to computing equipment specification, purchasing and maintenance; network infrastructure; software development; strategic planning for future IT initiatives; budget preparation performance reviews, and career growth planning for IT staff.

TECHNICAL INFORMATION SPECIALIST, *CAS Holdings,Inc., Kelso, Washington,* 1994-1999. Primary responsibilities included support automation of HP chemstation and Enviroquant; support continued development of unified organic laboratories; support and development of project-specific data deliverables, technical assistance in Information Technology at Kelso to meet CAS IT objectives as related to LIMS.

Organics Section Manager, *Anametrix, Inc., San Jose, California,* 1992-1994. Responsibilities included managing the GC/MS and GC/Pesticide Departments, following protocols of the Department of Defense NEESA contracts (demanding stringent QA/QC and "Level D" data package submittals). Primary responsibilities included supervisor training and development, budget preparation and maintenance, performance reviews, data review, continuing research on environmental trends, SOP generation and updates, method development, capital equipment evaluation for laboratories, and project management.

GC/MS Program Manager, *Anametrix, Inc., San Jose, California,* 1988-1992. Responsibilities included supervision of five chemists, two Finnigan 4000 GC/MS Systems, and three HP 5971 GC/MS Systems. Primary responsibilities were to maintain high productivity and insure that the GC/MS department generated legally defensible data. Also responsible for sample scheduling and tracking, instrument maintenance and troubleshooting, analyst training and review, client interfacing, and purchasing.

GC/MS Analyst, *Anametrix, Inc., San Jose, California,* 1986-1988. Responsibilities included analyzing for VOA and Semi-VOA priority pollutants using EPA Methods 624/625 and 8240/8370.

Education

BA, Biochemistry, *San Jose State University, San Jose, California,* 1986.

GARY K. WARD

2001 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

VICE PRESIDENT, CHIEF QUALITY, SAFETY, AND ETHICS OFFICER – 2001 to Present

Responsibilities

Responsibilities include directing and managing the overall corporate-wide quality systems, ethics and safety programs for all CAS facilities, as well as strategic planning, marketing, business development, and information technology. Responsible for all interaction and liaison with government entities involving quality, technical and operational issues.

Experience

Deputy Director, Laboratory Standards, *Intertek Testing Services, Houston, Texas,* 1998-2001. Responsibilities included professional standards/quality assurance for 240 laboratories in 93 countries, involving laboratory tests ranging from petroleum products and environmental samples to toys, textiles, and building products. Resolution of issues with a variety of governments, agiences, and companies with particular focus on interactions with the US EPA. Was previously responsible for all operations of over 100 labs in the Americas, ranging from Canada to South America, including duties to improve quality, raise profits and revenues, and implement a LIMS.

Director, Technical Operations, *Environmental Health Laboratories, South Bend, Indiana,* 1995-1998. Responsibilities included operations and quality assurance of the laboratory. Directed, administered and coordinated activities of the lab in accordance with goals and objectives of the company. Responsible for the R&D program, laboratory throughput and financial performance, and implementation of the new LIMS system.

Executive Scientist, *Quanterra (Enseco), Arvada, Colorado,* 1987-1995. Responsibilities included providing expertise and experience in laboratory analysis and operations to the entire laboratory system. Duties included implementation of network-wide LIMS as well as coordination of the Technology, QA, IS, and Operations groups. As Director of Technology and Quality Assurance was responsible for management of the R&D program, Quality Assurance program, and Environment, Health and Safety program throughout the Enseco lab system. Direct reports were all QA managers, safety managers, and chief scientists from each of the 13 laboratories.

Deputy Branch Chief, *U.S. Environmental Protection Agency*, 1983-1987. Responsibilities included providing expertise to entire Superfund program ranging from lab analytical services to sampling. Duties involved managing the CLP program as well as the Superfund R&D program. As CLP National Program Manager was responsible for development and implementation of CLP analytical protocols, administration of contracts for over 100 laboratories throughout the country, and liasion with contract divisions, other EPA programs, and enforcement. Responsible for development and implementation of disk deliverables, automated contract screening, as well as writing new protocols for specific methods such as ICP/MS and for EPA methods such as included in SW846, 3rd Edition. Duties also included coordination of the annual CLP conferences.

Education

MS, Chemical Oceanography, *RSMAS, University of Miami, Miami, Florida,* 1973.
BS, Chemistry, *Loyola University, Los Angeles, California,* 1970.

Publications, Presentations. And Affiliations

Mr. Ward has a number of publications and presentations, and is affiliated with several professional organizations. For a list of these, please contact CAS.

STEPHEN W. VINCENT

1986 TO PRESENT

Columbia Analytical Services, Inc., 1317 S. 13th Avenue, Kelso, WA 98626 (360) 577-7222

Current Position

PRESIDENT, CAS HOLDINGS INC. – 1986 to Present

Responsibilities

Responsible for the overall growth and profitability of the CAS laboratory network. This includes establishing and implementing long-range objectives, plans, and policies, and representing the company with its major customers, technical community, and the public.

Experience

Laboratory Manager, *Weyerhaeuser Company, Federal Way, Washington,* 1979-1986. Responsibilities involved all phases of technical and administrative management. This included management of organic, inorganic, and microbiological analyses and management of capital; an annual operating budget of approximately $2 million; management of thirty staff members; contract procurement, and project management. Projects included an EPA Inorganic CLP contract; an EPA acid rain deposition contract; a contract with the Fish and Wildlife Service to measure trace organic contaminants in animal tissues; and others.

Analytical Chemist, *Weyerhaeuser Company, Longview, Washington,* 1975-1979. Responsibilities: Method development, routine analysis and supervision for the Weyerhaeuser Multi-Region Support Lab. Responsible for setting up a company-wide laboratory audit, round robin, and quality assurance program.

Education

Market Strategy for Technology Based Companies, *Executives Program, Stanford University*. 1994.
Advanced Technical Management Program, *University of California at Los Angeles, Department of Business, Engineering and Management,* 1991.

Completion of Coursework for MS, Pulp and Paper Technology, *University of Washington, Seattle, Washington,* 1984.
Post Graduate Coursework, Engineering and Management, *University of California at Los Angeles, Graduate School of Engineering and Applied Science, Los Angeles, California,* 1981.
BS, Oceanography, *University of Washington, Seattle, Washington,* 1974.

Publications/ Presentations

Mr. Vincent has a number of publications and presentations. For a list of these publications and presentations, please contact CAS.

Affiliations

American Chemical Society.
Technical Association of the Pulp and Paper Industry.

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 (9):			
Precisa and Mettler models	1988-2000	MM	15
Autoclave - Market Forge Sterilmatic	1988	LM	5
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	3
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):			
Lachat Quik-Chem AE	1990	LM	5
Bran-Leubbe	2002	LM	4
Ion Chromatographs (3)			
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
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
Microscopes (2):			
Bausch & Lomb	1988	LM	1
Swift	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 (2):			
Hitachi 100-40 Single Beam	1986	LM	5
Beckman-Coulter DU520	2005	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)			
Various Mettler AE 200 analytical balance	1990	MM	12
Various Mettler 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) - Thermo Jarrell Ash Model 61E	1988	LM	4
Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES): Thermo Jarrell Ash, Model IRIS	2000	MM	4
Inductively Coupled Plasma Mass Spectrometers (ICP-MS):			
VG PQ-S	1997	MM	3
VG Excell	2001	MM	3
Thermo X-Series	2006	MM	3
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**
Accelerated Solvent Extractor - Dionex ASE 200	1996	LM	5
Analytical Balance (3)			
Mettler BB240	1987	MM	12
Satorious B610 (2)	1999, 2000	MM	12
Aspirator pumps - Labconco Cole Parmer (1)	1994	LM	7
Centrifuges (2):			
Adams Model DYNAC	1986	LM	12
Sorvall Model GLC-1	1988	LM	12
Drying Oven - Fisher Model 655 G	1991	LM	7
Evaporators (10):			
Organomation N-Evap (5)	1989-90,1998-2001	LM	12
Organomation S-Evap (5)	1989-1991	LM	12
Extractors: Lab-Line Multi-Unit Extraction Heaters (60)	1987-1992	LM	12
Extractors (64):			
Continuous Liquid/Liquid Extractors (24)	1991	LM	10
Branson Model 450 Sonifier (2)	1991	LM	4
Tekmar Sonifier (2)	1994	LM	4
Soxhtherm (36)	2000	LM	5
Gel Permeation Chromatography (GPC) (4)			
ABC single column (2)	1998, 1999	LM	4
ABC Autoprep 1000	1995	LM	4
J2 Scientific	2005	LM	4
Muffle Furnace - Parflow MIC 6000	1994	LM	12
Solid Phase Extractors (8) – Dionex SPE-Dex 4790	2003, 2006	LM	6
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	5
Chromatography Data Systems (12)			
HP Enviroquant (8)	1994-2002	LM	5
Thruput Target (4)	1998-2000	LM	5
Gas Chromatographs (13):			
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual ECD Detectors (7)	1990 – 1995	LM	5
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual FPD Detectors	1991	LM	5
Agilent 6890 GC with Agilent 7683 Autosampler and Dual ECD Detectors (4)	2001, 2005	LM	5
Agilent 6890 GC with Agilent 7683 Autosampler and Dual FPD Detectors	2003	MM	5

GC/MS SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
HP Enviroquant Chromatography Data Systems (9)	1994-2002	LM	6
Gas Chromatograph: Hewlett-Packard 5890 with HP 7673 autosampler and FID Detector	1994	LM	2
Semivolatile GC/MS Systems (8):			
Agilent 6890/5973 with ATAS Optic2 LVI and HP 7673 Autosampler (2)	1997, 2001	MM	4
Agilent 5890/5970 with ATAS Optic2 LVI and HP 7673 Autosampler (2)	1990,1994	MM	4
Agilent 5890/5972 with ATAS Optic2 LVI and HP 7673 Autosampler (3)	1993, 1994, 1998	MM	4
Agilent 6890/5973 with ATAS Optic3 LVI and 7683 Autosampler (1)	2004	MM	4

PETROLEUM HYDROCARBONS GC/HPLC LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
SAMPLE PREPARATION			
Analytical Balance (3)			
Mettler BB300	1991	MM	6
Mettler BB240	1994	MM	6
Mettler AE166	1994	MM	6
Aspirator pumps – GAST (2)	2002, 2004	LM	6
Drying Oven - Fisher Model 630F	1991	LM	6
Evaporators (2):			
Organomation N-Evap	1990	LM	6
Organomation S-Evap	1991	LM	6
Extractors (4):			
Sonic Horns (2): Branson, Ultrasonics, Fisher Models	1991-1994	LM	6
INSTRUMENTATION			
HP Enviroquant Chromatography Data Systems (10)	1994-2002	LM	6
Gas Chromatographs (5):			
Hewlett-Packard 5890 Series II with PID/PID/FID:	1991	LM	3
Tekmar LSC-2000 Purge and Trap Concentrator	1991	LM	3
Dynatech Archon 5100 Autosampler	1992	LM	3
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual FID Detectors (2)	1990 - 1995	LM	3
Agilent 6890 with Dual FID Detectors and Agilent 7873 Autosampler (2)	2001, 2005	LM	3
High-Performance Liquid Chromatographs (2):			
HP 1090M Series II with Diode Array UV Detector	1999	LM	3
HP 1050/1100 Series with Fluorescence & Diode Array UV Detectors	2004	LM	3
High-Performance Liquid Chromatograph/Mass Spectrometer - Thermo Electron TSQ Quantum LC/MS/MS with Thermo Surveyor HPLC and Autosampler	2005	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	6
Baxter Vortex Mixer	1989	LM	6
Extractors (10):			
Millipore TCLP Zero Headspace Extractors (10)	1987-1992	LM	2
TCLP Extractor - Tumbler (12 position)	1989	LM	2
HP Enviroquant Chromatography Data Systems (10)	1994-2002	LM	6
Drying Ovens (2):			
Narco 420	1989	LM	6
VWR 1305 U	1991	LM	6
Sonic Water Bath - Branson Model 2200	1989	LM	6
Volatile GC/MS Systems (8):			
Agilent 5890/5970 (2)	1989	MM	4
Tekmar 3000 Purge and Trap Concentrator	1995	LM	4
Dynatech ARCHON 5100 Autosampler	1996	LM	4
Agilent 5890/5970	1999	MM	4
EST Encon Purge and Trap Concentrator	2002	LM	4
Dynatech ARCHON 5100 Autosampler	1999	LM	4
Agilent 5890/5971	1991	MM	4
Tekmar 3000 Purge and Trap Concentrator	2001	LM	4
Dynatech ARCHON 5100 Autosampler	1995	LM	4
Agilent 5890/5972A	1993	MM	4
Tekmar 3000 Purge and Trap Concentrator	1995	LM	4
Dynatech ARCHON 5100 Autosampler	1996	LM	4
Agilent 6890/5973 (2)	2001, 2007	MM, LM	4
Tekmar 3100 Purge and Trap Concentrator	2001, 2007	LM	4
Varian Archon Autosampler	2001, 2007	LM	4
Agilent 6890/5973	2005	MM	4
Tekmar Velocity Purge and Trap Concentrator	2005	LM	4
Tekmar Aquatech Autosampler	2005	LM	4

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 (5) – Horizon SPE-DEX Solid Phase Extractor	2003	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/ECD system: Dual micro-ECD detectors Agilent autosampler	2003 2003 2003	LM LM LM	2 2 2
Varian Ion trap GC/MS: Varian 3800 GC w/CP8400 autosampler Varian 3900 GC Varian Saturn 2100T mass spectrometer	2003 2006 2003 2003	LM LM LM LM	2 2 2 2

PHARMACEUTICAL TESTING LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Thermo High-Performance Liquid Chromatograph with Diode Array UV Detector	2005	MM	2
Thermo FTIR	2005	MM	4
Viscometer	2006	LM	1
Analytical Balance - Mettler AB104-5 and AT-250	2004	MM	4
Incubator – VWR 1510E	2004	LM	2
Karl Fisher Titrators (2) – Mettler DL38 and DL39	2004	MM	3
Melting point apparatus – Optimelt	2004	LM	3
Refractometer – Reichert Abbe Mark II	2004	LM	2
Rotary evaporator – Labonco/Cole-Parmer aspirator pump	2004	LM	3
Rotary shakers – Thermolyne Rotomix (2)	2004	LM	2
Vacuum oven – Precision Model 19 w/GAST aspirator	2004	LM	3
Water baths (3) – VWR Models 1212, 1235 and Labline 18002	2004	LM	2
Gas Chromatographs (2): Hewlett-Packard 5890 Series II with TCD/FID detector Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual FID Detectors	 1988 1990	 LM LM	 3 3

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
2 - Network Servers 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.	1994 - 2004	LM	NA
Approximately 50+ HP and Dell Laserjet printers (various types including IIIs, 4s, 5s, 8150s, 4000s, 4050s, 4250, 8150s, W5300s)	1991 - 2004	LM	NA
Approximately 130 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 9600 - 33600 Baud, (2) Brother 4750e, 15 ppm; (1) Brother MFC 1970MC, 2ppm; (1) Canon CFX-L4000	1991 - 2004	LM	NA
Copiers/Scanners (2) Konica 7085, 85ppm, 20GB; (2) Konica 7155; (1) Konica 7035, BizHub. The 7085s and one 7155 are accessible via LAN for network scanning.	2000 - 2004	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
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

SOP LIST AND LIST OF NELAC ACCREDITED METHODS

COLUMBIA ANALYTICAL SERVICES, INC. , KELSO, WA.
STANDARD OPERATING PROCEDURES TABLE OF CONTENTS
January 11, 2006

SOP NAME	FILE NAME	REV #
ARMY CORPS OF ENGINEERS HTRW PROJECT MANAGEMENT	ADM-HTRW	1
CHECKING PIPETTE CALIBRATION	ADM-CPIP	4
CONTINGENCY PLAN FOR LABORATORY EQUIPMENT FAILURE	ADM-ECP	0
CONTROL CHARTING QUALITY CONTROL DATA	ADM-CHRT	1
DATA ARCHIVING	ADM-ARCH	3
DATA REPORTING AND REPORT GENERATION	ADM-RG	5
DEPARTMENT OF DEFENSE PROJECTS LABORATORY PRACTICES AND PROJECT MANAGEMENT	ADM-DOD	0
ELECTRONIC DATA BACKUP AND ARCHIVING	ADM-EBACKUP	0
FACILITY AND LABORATORY CLEANING	ADM-FACL	0
INTERNAL QUALITY ASSURANCE AUDITS	ADM-IAUD	5
LABORATORY DATA REVIEW PROCESS	ADM-DREV	4
METHOD DETECTION LIMIT DOCUMENTATION AND CONTROL	ADM-MDLC	2
PROJECT MANAGEMENT	ADM-PCM	6
REAGENT LOGIN AND TRACKING	ADM-RLT	2
SUPPORT EQUIPMENT MONITORING AND CALIBRATION	ADM-SEMC	8
COLIFORM, FECAL	BIO-9221FC	5
COLIFORM, FECAL (MEMBRANE FILTER PROCEDURE)	BIO-9222D	1
COLIFORM, TOTAL	BIO-9221TC	4
COLIFORM, TOTAL (DRINKING WATER)	BIO-9221DW	3
COLILERT COMPLETED TEST VERIFICATION OF E. COLI IN MUG CULTURES	BIO-CCT	0
COLILERT P/A	BIO-COLI	2
ENTEROLERT	BIO-ENT	0
FECAL STREPTOCOCCUS/ENTEROCOCCUS	BIO-9230B	5
HEPTEROTROPHIC PLATE COUNT	BIO-HPC	3
MICROBIOLOGY QUALITY ASSURANCE AND QUALITY CONTROL	BIO-QAQC	10
SHEEN SCREEN/OIL DEGRADING MICROORGANISMS	BIO-SHEEN	0

EPA CLP ORGANICS ANALYSES	CLP_ORGA	1
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AUTOMATED SOXHLET EXTRACTION	EXT-3541	3
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FLORISIL CLEANUP	EXT-FLOR	1
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MEASURING SAMPLE WEIGHTS AND VOLUMES FOR ORGANIC ANALYSIS	EXT-WVOL	2
PREPARATION OF ANHYDROUS SODIUM SULFATE , MARTIX SAND, SODIUM CHLORIDE, AND POTASSIUM CARBONATE	EXT-SULF	2
PRESSURIZED FLUID EXTRACTION	EXT-3545	5
SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION	EXT-3510	7
SOLID PHASE DISPERSION IN TISSUES	EXT-SPD	0
SOLID PHASE EXTRACTION	EXT-3535	2
SOXHLET EXTRACTION	EXT-3540	8
ULTRASONIC EXTRACTION	EXT-3550	9
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ALKALINITY TOTAL	GEN-310.1	5
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AMMONIA BY FLOW INJECTION ANALYSIS	GEN-350.1	6
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FLASHPOINT DETERMINATION - SETAFLASH	GEN-1020	5
FLUORIDE BY ION SELECTIVE ELECTRODE	GEN-FISE	5
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OPERATION, CALIBRATION, AND MAINTENANCE OF THE HEWLETT PACKARD 1050 SERIES HPLC	PHM-IN05	1
OPERATION, CALIBRATION, AND MAINTENANCE OF THE METTLER TOLEDO DL39 COULOMETRIC TITRATOR	PHM-IN06	0
OPERATION, CALIBRATION, AND MAINTENANCE OF THE SRS MPA100 AUTOMATED MELTING POINT SYSTEM	PHM-IN07	2
OPERATION, CALIBRATION, AND MAINTENANCE OF THE SUN INSTRUMENTS TYPE WXG-D DISK POLARIMETER	PHM-IN08	1
OPERATION, CALIBRATION, AND MAINTENANCE OF THE REICHERT ABBE MARK II MODEL 10480 REFRACTOMETER	PHM-IN09	0
OPERATION, CALIBRATION, AND MAINTENANCE OF THE LAB-LINE MODEL 18002 UTILITY HEATED WATER BATH	PHM-IN10	0
OPERATION, CALIBRATION, AND MAINTENANCE OF THE Brookfield DV-III Ultra Programmable Rheometer	PHM-IN11	1
OPERATION, CALIBRATION, AND MAINTENANCE OF THE THERMOE ELECTRON NICOLET 4700 FTIR	PHM-IN12	1
OPERATION, CALIBRATION, AND MAINTENANCE OF THE METTLER TOLEDO AT250 ANALYTICAL BALANCE	PHM-IN13	0
OPERATION, CALIBRATION, AND MAINTENANCE OF The PERKIN ELMER AANALYST 200 FLAME ATOMIC ABSORPTION SPECTROMETER (Flame Only)	PHM-IN14	1
OPERATION, CALIBRATION, AND MAINTENANCE OF THE VARIAN SPECTRA AA 220ZGFAA SPECTROMETER	PHM-IN15	1
OPERATION, CALIBRATION, AND MAINTENANCE OF THE THERMO IRIS ADVANTAGE ICP-AES	PHM-IN17	1
OPERATION, CALIBRATION, AND MAINTENANCE OF The PERKIN ELMER AANALYST 200 FLAME AA AND FIAS 100 FOR CVAA MERCURY	PHM-IN18	0
OPERATION, CALIBRATION, AND MAINTENANCE OF The VWR 1540 INCUBATOR	PHM-IN19	1
OPERATION, CALIBRATION, AND MAINTENANCE of the BECKMAN COULTER DU500 UV/Vis SPECTROPHOMETER	PHM-IN20	0
OPERATION, CALIBRATION, AND MAINTENANCE OF The FISHER ISOTEMP INCUBATOR	PHM-IN21	0
OPERATION, CALIBRATION, AND MAINTENANCE OF The NAPCO 6300 INCUBATOR	PHM-IN23	0
OPERATION, CALIBRATION, AND MAINTENANCE OF The VWR 2020 INCUBATOR	PHM-IN24	0
OPERATION, CALIBRATION, AND MAINTENANCE OF The HANNA AUTOTITRATOR	PHM-IN25	0
OPERATION, CALIBRATION, AND MAINTENANCE OF THE METTLER TOLEDO SEVENMULTI ELECTROCHEMICAL ANALYTICAL METER	PHM-IN26	0
PHARMACEUTICAL QUALITY ASSURANCE SOPs		
CONTROLLED SUBSTANCES	PHM-CSUB	1
VALIDATION OF PHARMACEUTICAL AND NUTRACEUTICAL TESTING METHODS	PHM-QA01	0
INSTRUMENT QUALIFICATION AND VERIFICATION	PHM-QA02	3
PHARMACEUTICAL TESTING DOCUMENTATION AND RECORDKEEPING	PHM-QA03	3
DETERMINATION OF TEST SPECIFICATIONS	PHM-QA04	0
EVALUATING AND SELECTING TEST METHODS FOR PHARMACEUTICAL AND NUTRACEUTICAL TESTING	PHM-QA05	2

OUT OF SPECIFICATION (OOS) INVESTIGATION AND RETESTING	PHM-QA06	2
CHANGE CONTROL	PHM-QA07	2
DATA MANAGEMENT OF PHARMACEUTICAL TESTING RECORDS	PHM-QA08	0
PHARMACEUTICAL PERSONNEL TRAINING	PHM-QA09	0
VALIDATION OF SOFTWARE USED FOR PHARMACEUTICAL TESTING	PHM-QA10	0
DATA SYSTEMS ACCESS AND AUDIT TRAILS	PHM-QA11	0
QUALITY CONTROL OF PHARMCEUTICAL TESTING DATA	PHM-QA12	0
SOP NAME - CORPORATE ADMINISTRATIVE SOPS MAINTAINED BY CORPORATE QA	**FILE NAME**	**REV #**
CHAIN OF CUSTODY FOR SAMPLE TRANSFER BETWEEN LABORATORIES	ADM-COC	1
CHECKING NEW LOTS OF CHEMICALS FOR CONTAMINATION	ADM-CTMN	2
CONTROL LIMITS	ADM-CTRL_LIM	4
DEALING WITH COMPLAINTS	ADM-CMPLT	3
DETERMINATION OF METHOD DETECTION LIMITS AND LIMITS OF DETECTION	ADM-MDL	6
DOCUMENT CONTROL	ADM-DOCCTRL	5
DOCUMENTATION OF TRAINING	ADM-TRANDOC	9
ELECTRONIC DATA AUDITING	ADM-E_DATAAUDIT	1
ESTIMATION OF UNCERTAINTY OF MEASUREMENTS	ADM-UNCERT	2
MAKING ENTRIES INTO LOGBOOKS AND ONTO BENCHSHEETS	ADM-DATANTRY	6
MANAGERIAL REVIEW OF THE LABORATORY"S QUALITY SYSTEM	ADM-MGMTRVW	1
MANUAL INTEGRATION OF CHROMATOGRAPHIC PEAKS	ADM-INT	2
NONCONFORMITY AND CORRECTIVE ACTION	ADM-NCAR	4
PREPARATION OF ELECTRONIC DATA FOR ORGANIC ANALYSES ELECTRONIC DATA AUDITS	ADM-EDATA	2
PREPARATION OF STANDARD OPERATING PROCEDURES	ADM-SOP	6
PROFICIENCY TESTING SAMPLE ANALYSIS	ADM-PTS	0
PURCHASING THROUGH CAS PURCHASING AGENT	ADM-PUR	1
QUALIFICATION OF SUBCONTRACT LABORATORIES OUTSIDE OF CAS NETWORK	ADM-SUBLAB	2
SAMPLE BATCHES	ADM-BATCH	7
SIGNIFICANT FIGURES	ADM-SIG.FIG	6

Laboratory Scope of Accreditation

Attachment to Certificate #: E87412-05, expiration date June 30, 2007. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87412 EPA Lab Code: WA00035 (360) 577-7222

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,1,1-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,1,2,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,1,2-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,1-Dichloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,1-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,1-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,2,3-Trichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,2,3-Trichloropropane	EPA 504.1	Group II Unregulated Contaminants	NELAP	7/17/2003
1,2,3-Trichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,2,4-Trichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,2,4-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,2-Dibromo-3-chloropropane (DBCP)	EPA 504.1	Synthetic Organic Contaminants	NELAP	7/17/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 504.1	Synthetic Organic Contaminants	NELAP	7/17/2003
1,2-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,2-Dichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,2-Dichloropropane	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
1,3,5-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,3-Dichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,3-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
1,4-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
2,2-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
2,4,5-T	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
2,4-D	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
2-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
3-Hydroxycarbofuran	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/17/2003
4,4'-DDD	EPA 508.1	Group I Unregulated Contaminants	NELAP	7/17/2003
4,4'-DDD	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
4,4'-DDE	EPA 508.1	Group I Unregulated Contaminants	NELAP	7/17/2003
4,4'-DDE	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
4,4'-DDT	EPA 508.1	Group I Unregulated Contaminants	NELAP	7/17/2003
4,4'-DDT	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
4-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/17/2003
4-Isopropyltoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001

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Columbia Analytical Services, Inc. - WA
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Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Acetochlor	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Acifluorfen	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
Alachlor	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Aldicarb (Temik)	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Aldicarb sulfone	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Aldicarb sulfoxide	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Aldrin	EPA 508.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Aldrin	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Alkalinity as CaCO3	SM 2320 B	Primary Inorganic Contaminants	NELAP	10/8/2001
Aluminum	EPA 200.7	Secondary Inorganic Contaminants	NELAP	10/8/2001
Aluminum	EPA 200.8	Secondary Inorganic Contaminants	NELAP	10/8/2001
Antimony	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Antimony	EPA 200.9	Primary Inorganic Contaminants	NELAP	10/8/2001
Arsenic	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Arsenic	EPA 200.9	Primary Inorganic Contaminants	NELAP	10/8/2001
Atrazine	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Barium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Barium	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Benzene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Benzo(a)pyrene	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Beryllium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Beryllium	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Bromate	EPA 300.1	Primary Inorganic Contaminants	NELAP	7/17/2003
Bromide	EPA 300.1	Primary Inorganic Contaminants	NELAP	7/17/2003
Bromoacetic acid	EPA 552.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Bromobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Bromochloroacetic acid	EPA 552.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Bromochloromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/17/2003
Bromodichloromethane	EPA 524.2	Other Regulated Contaminants,Group II Unregulated Contaminants	NELAP	10/8/2001
Bromoform	EPA 524.2	Other Regulated Contaminants,Group II Unregulated Contaminants	NELAP	10/8/2001
Butachlor	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Butyl benzyl phthalate	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
Cadmium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Cadmium	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Calcium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Carbaryl (Sevin)	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Carbofuran (Furaden)	EPA 531.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Carbon tetrachloride	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Chlorate	EPA 300.1	Secondary Inorganic Contaminants	NELAP	12/23/2005
Chlordane (tech.)	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Chloride	EPA 300.0	Secondary Inorganic Contaminants	NELAP	10/8/2001
Chlorite	EPA 300.1	Primary Inorganic Contaminants	NELAP	7/17/2003
Chloroacetic acid	EPA 552.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Chlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Chloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Chloroform	EPA 524.2	Other Regulated Contaminants,Group II Unregulated Contaminants	NELAP	10/8/2001
Chromium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Chromium	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
cis-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
cis-1,3-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Color	SM 2120 B	Secondary Inorganic Contaminants	NELAP	7/17/2003
Conductivity	SM 2510 B	Primary Inorganic Contaminants	NELAP	10/8/2001
Copper	EPA 200.7	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	10/8/2001
Copper	EPA 200.8	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	10/8/2001
Copper	EPA 200.9	Primary Inorganic Contaminants	NELAP	10/8/2001
Cyanide	EPA 335.4	Primary Inorganic Contaminants	NELAP	10/8/2001
Dacthal (DCPA)	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Dalapon	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
DCPA di acid degradate	EPA 515.4	Group I Unregulated Contaminants	NELAP	7/17/2003
DCPA mono acid degradate	EPA 515.4	Group I Unregulated Contaminants	NELAP	7/17/2003
Di(2-ethylhexyl)adipate	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Dibromoacetic acid	EPA 552.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Dibromochloromethane	EPA 524.2	Other Regulated Contaminants,Group II Unregulated Contaminants	NELAP	10/8/2001
Dibromomethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Dicamba	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
Dichloroacetic acid	EPA 552.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Dichlorodifluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Dichloromethane (DCM, Methylene chloride)	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Dieldrin	EPA 508.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Dieldrin	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Diethyl phthalate	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
Dimethyl phthalate	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
Di-n-butyl phthalate	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
Di-n-octyl phthalate	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
Diquat	EPA 549.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Endothall	EPA 548.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Endrin	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Endrin	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
EPTC (Eptam, s-ethyl-dipropyl thio carbamate)	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Ethylbenzene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Fluoride	EPA 300.0	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	10/8/2001
Fluoride	SM 4500 F-C	Secondary Inorganic Contaminants,Primary Inorganic Contaminants	NELAP	10/8/2001
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Glyphosate	EPA 547	Synthetic Organic Contaminants	NELAP	7/17/2003
Heptachlor	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Heptachlor	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Heptachlor epoxide	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Heptachlor epoxide	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Heterotrophic plate count	SM 9215 B	Microbiology	NELAP	7/17/2003
Hexachlorobenzene	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Hexachlorobutadiene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Hexachlorocyclopentadiene	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Iron	EPA 200.7	Secondary Inorganic Contaminants	NELAP	10/8/2001
Isophorone	EPA 525.2	Group III Unregulated Contaminants	NELAP	7/17/2003
Isopropylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001

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Lead	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Lead	EPA 200.9	Primary Inorganic Contaminants	NELAP	10/8/2001
Magnesium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Manganese	EPA 200.7	Secondary Inorganic Contaminants	NELAP	10/8/2001
Manganese	EPA 200.8	Secondary Inorganic Contaminants	NELAP	10/8/2001
Mercury	EPA 245.1	Primary Inorganic Contaminants	NELAP	10/8/2001
Methomyl (Lannate)	EPA 531.1	Group I Unregulated Contaminants	NELAP	7/17/2003
Methoxychlor	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Methoxychlor	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Methyl bromide (Bromomethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Methyl chloride (Chloromethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Methyl tert-butyl ether (MTBE)	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Metolachlor	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Metribuzin	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Molinate	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Naphthalene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
n-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Nickel	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Nickel	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Nitrate	EPA 300.0	Primary Inorganic Contaminants	NELAP	10/8/2001
Nitrate	EPA 353.2	Primary Inorganic Contaminants	NELAP	10/8/2001
Nitrite	EPA 300.0	Primary Inorganic Contaminants	NELAP	10/8/2001
Nitrite	EPA 353.2	Primary Inorganic Contaminants	NELAP	10/8/2001
n-Propylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Orthophosphate as P	SM 4500-P F	Primary Inorganic Contaminants	NELAP	10/8/2001
Oxamyl	EPA 531.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Paraquat	EPA 549.2	Synthetic Organic Contaminants	NELAP	12/23/2005
PCBs	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
Pentachlorophenol	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
Pentachlorophenol	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Perchlorate	EPA 314.0	Primary Inorganic Contaminants	NELAP	7/17/2003
pH	EPA 150.1	Secondary Inorganic Contaminants,Primary Inorganic Contaminants	NELAP	10/8/2001
Picloram	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
Propachlor (Ramrod)	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
sec-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Selenium	EPA 200.8	Primary Inorganic Contaminants	NELAP	10/8/2001
Selenium	EPA 200.9	Primary Inorganic Contaminants	NELAP	10/8/2001
Silica as SiO2	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Silver	EPA 200.7	Secondary Inorganic Contaminants	NELAP	10/8/2001
Silver	EPA 200.8	Secondary Inorganic Contaminants	NELAP	10/8/2001
Silvex (2,4,5-TP)	EPA 515.4	Synthetic Organic Contaminants	NELAP	7/17/2003
Simazine	EPA 525.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Sodium	EPA 200.7	Primary Inorganic Contaminants	NELAP	10/8/2001
Styrene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Sulfate	EPA 300.0	Secondary Inorganic Contaminants,Primary Inorganic Contaminants	NELAP	10/8/2001
Terbacil	EPA 525.2	Group I Unregulated Contaminants	NELAP	7/17/2003
tert-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Tetrachloroethylene (Perchloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Thallium	EPA 200.8	Primary Inorganic Contaminants	NELAP	11/18/2004
Thallium	EPA 200.9	Primary Inorganic Contaminants	NELAP	10/8/2001
Toluene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Total coliforms & E. coli	SM 9223 B	Microbiology	NELAP	10/8/2001
Total dissolved solids	SM 2540 C	Secondary Inorganic Contaminants	NELAP	10/8/2001
Total haloacetic acids	EPA 552.2	Synthetic Organic Contaminants	NELAP	7/17/2003
Total nitrate-nitrite	EPA 300.0	Primary Inorganic Contaminants	NELAP	10/8/2001
Total nitrate-nitrite	EPA 353.2	Primary Inorganic Contaminants	NELAP	10/8/2001
Total trihalomethanes	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Toxaphene (Chlorinated camphene)	EPA 508.1	Synthetic Organic Contaminants	NELAP	7/17/2003
trans-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
trans-1,3-Dichloropropylene	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Trichloroacetic acid	EPA 552.2	Group I Unregulated Contaminants	NELAP	7/17/2003
Trichloroethene (Trichloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Trichlorofluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	10/8/2001
Turbidity	EPA 180.1	Secondary Inorganic Contaminants	NELAP	10/8/2001
Vinyl chloride	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Xylene (total)	EPA 524.2	Other Regulated Contaminants	NELAP	10/8/2001
Zinc	EPA 200.7	Secondary Inorganic Contaminants	NELAP	10/8/2001
Zinc	EPA 200.8	Secondary Inorganic Contaminants	NELAP	10/8/2001

Laboratory Scope of Accreditation

Attachment to Certificate #: E87412-05, expiration date June 30, 2007. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87412 EPA Lab Code: WA00035 (360) 577-7222

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,1-Trichloroethane	EPA 624	Volatile Organics	NELAP	10/8/2001
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,2,2-Tetrachloroethane	EPA 624	Volatile Organics	NELAP	10/8/2001
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1,2-Trichloroethane	EPA 624	Volatile Organics	NELAP	10/8/2001
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethane	EPA 624	Volatile Organics	NELAP	10/8/2001
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethylene	EPA 624	Volatile Organics	NELAP	10/8/2001
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2,4-Trichlorobenzene	EPA 625	Extractable Organics	NELAP	10/8/2001
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	10/8/2001
1,2-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	10/8/2001
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2-Dichloroethane	EPA 624	Volatile Organics	NELAP	10/8/2001
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichloropropane	EPA 624	Volatile Organics	NELAP	10/8/2001
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	10/8/2001
1,3-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	10/8/2001
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003

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E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	7/1/2003
1,4-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	10/8/2001
1,4-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	10/8/2001
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,4-Dioxane (1,4-Diethyleneoxide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ 183)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ 187)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,5,5'-Hexachlorobiphenyl (BZ 141)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,5,5',6-Hexachlorobiphenyl (BZ 151)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
2,3,3',4',6-Pentachlorobiphenyl (BZ 110)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3,4,6-Tetrachlorophenol	EPA 1653	Extractable Organics	NELAP	10/8/2001
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,3-Dichlorobiphenyl (BZ 5)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4',5-Trichlorobiphenyl (BZ 31)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4,5-Trichlorophenol	EPA 1653	Extractable Organics	NELAP	10/8/2001
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trichlorophenol	EPA 1653	Extractable Organics	NELAP	10/8/2001

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E87412
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1317 South 13th Avenue
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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,4,6-Trichlorophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4-Dichlorophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dimethylphenol	EPA 625	Extractable Organics	NELAP	10/8/2001
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 625	Extractable Organics	NELAP	10/8/2001
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 625	Extractable Organics	NELAP	10/8/2001
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Chlorobiphenyl (BZ 1)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2-Chloroethyl vinyl ether	EPA 624	Volatile Organics	NELAP	10/8/2001
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Chloronaphthalene	EPA 625	Extractable Organics	NELAP	10/8/2001
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chlorophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Hexanone	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Methyl-4,6-dinitrophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitrophenol	EPA 625	Extractable Organics	NELAP	10/8/2001

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E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/1/2003
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Sec-butyl-4,6-dinitrophenol (DNBP, Dinoseb)	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,3'-Dichlorobenzidine	EPA 625	Extractable Organics	NELAP	10/8/2001
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3,4,5-Trichlorocatechol	EPA 1653	Extractable Organics	NELAP	10/8/2001
3,4,5-Trichloroguaiacol	EPA 1653	Extractable Organics	NELAP	10/8/2001
3,4,6-Trichlorocatechol	EPA 1653	Extractable Organics	NELAP	10/8/2001
3,4,6-Trichloroguaiacol	EPA 1653	Extractable Organics	NELAP	10/8/2001
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/1/2003
4,4'-DDD	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDE	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDT	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,5,6-Trichloroguaiacol	EPA 1653	Extractable Organics	NELAP	10/8/2001
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/1/2003
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Bromophenyl phenyl ether	EPA 625	Extractable Organics	NELAP	10/8/2001
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloro-3-methylphenol	EPA 625	Extractable Organics	NELAP	10/8/2001
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chlorophenyl phenylether	EPA 625	Extractable Organics	NELAP	10/8/2001
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitrophenol	EPA 625	Extractable Organics	NELAP	10/8/2001

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/1/2003
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthene	EPA 625	Extractable Organics	NELAP	10/8/2001
Acenaphthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Acenaphthylene	EPA 625	Extractable Organics	NELAP	10/8/2001
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthylene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Acetone	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acetonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acetophenone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acrolein (Propenal)	EPA 624	Volatile Organics	NELAP	7/17/2003
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acrylonitrile	EPA 624	Volatile Organics	NELAP	7/17/2003
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Adsorbable organic halogens (AOX)	EPA 1650	General Chemistry	NELAP	10/8/2001
Aldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Alkalinity as CaCO3	EPA 310.1	General Chemistry	NELAP	10/8/2001
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	12/23/2005
Aluminum	EPA 200.7	Metals	NELAP	10/8/2001
Aluminum	EPA 200.8	Metals	NELAP	10/8/2001
Aluminum	EPA 6010	Metals	NELAP	7/1/2003
Aluminum	EPA 6020	Metals	NELAP	7/1/2003
Aluminum	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Amenable cyanide	EPA 335.1	General Chemistry	NELAP	10/8/2001
Ammonia as N	EPA 350.1	General Chemistry	NELAP	10/8/2001
Ammonia as N	EPA 350.3	General Chemistry	NELAP	10/8/2001
Aniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
Anthracene	EPA 625	Extractable Organics	NELAP	10/8/2001

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Anthracene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Antimony	EPA 200.7	Metals	NELAP	10/8/2001
Antimony	EPA 200.8	Metals	NELAP	10/8/2001
Antimony	EPA 6010	Metals	NELAP	7/1/2003
Antimony	EPA 6020	Metals	NELAP	7/1/2003
Antimony	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Aramite	EPA 8270	Extractable Organics	NELAP	7/1/2003
Aroclor-1016 (PCB-1016)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1221 (PCB-1221)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1232 (PCB-1232)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1242 (PCB-1242)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1248 (PCB-1248)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1254 (PCB-1254)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1260 (PCB-1260)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Arsenic	EPA 200.7	Metals	NELAP	10/8/2001
Arsenic	EPA 200.8	Metals	NELAP	10/8/2001
Arsenic	EPA 200.9	Metals	NELAP	10/8/2001
Arsenic	EPA 6010	Metals	NELAP	10/8/2001
Arsenic	EPA 6020	Metals	NELAP	7/1/2003
Arsenic	EPA 7060	Metals	NELAP	10/8/2001
Arsenic	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Azinphos-methyl (Guthion)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Barium	EPA 200.7	Metals	NELAP	10/8/2001
Barium	EPA 200.8	Metals	NELAP	10/8/2001
Barium	EPA 6010	Metals	NELAP	7/1/2003
Barium	EPA 6020	Metals	NELAP	7/1/2003
Barium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Benzene	EPA 624	Volatile Organics	NELAP	10/8/2001

Laboratory Scope of Accreditation

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State Laboratory ID: E87412 EPA Lab Code: WA00035 (360) 577-7222

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Benzene	EPA 8021	Volatile Organics	NELAP	7/1/2003
Benzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Benzidine	EPA 625	Extractable Organics	NELAP	10/8/2001
Benzo(a)anthracene	EPA 625	Extractable Organics	NELAP	10/8/2001
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)anthracene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Benzo(a)pyrene	EPA 625	Extractable Organics	NELAP	10/8/2001
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)pyrene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Benzo(b)fluoranthene	EPA 625	Extractable Organics	NELAP	10/8/2001
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(b)fluoranthene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Benzo(g,h,i)perylene	EPA 625	Extractable Organics	NELAP	10/8/2001
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(g,h,i)perylene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Benzo(k)fluoranthene	EPA 625	Extractable Organics	NELAP	10/8/2001
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(k)fluoranthene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Benzoic acid	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Beryllium	EPA 200.7	Metals	NELAP	10/8/2001
Beryllium	EPA 200.8	Metals	NELAP	10/8/2001
Beryllium	EPA 6010	Metals	NELAP	7/1/2003
Beryllium	EPA 6020	Metals	NELAP	7/1/2003
Beryllium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
beta-BHC (beta-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
beta-Naphthylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Biochemical oxygen demand	EPA 405.1	General Chemistry	NELAP	10/8/2001
bis(2-Chloroethoxy)methane	EPA 625	Extractable Organics	NELAP	10/8/2001
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroethyl) ether	EPA 625	Extractable Organics	NELAP	10/8/2001
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 625	Extractable Organics	NELAP	10/8/2001
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	7/1/2003

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bis(2-Ethylhexyl) phthalate (DEHP)	EPA 625	Extractable Organics	NELAP	10/8/2001
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	7/1/2003
Bolstar (Sulprofos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Boron	EPA 200.7	Metals	NELAP	10/8/2001
Bromide	EPA 300.0	General Chemistry	NELAP	10/8/2001
Bromobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromodichloromethane	EPA 624	Volatile Organics	NELAP	10/8/2001
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromoform	EPA 624	Volatile Organics	NELAP	10/8/2001
Bromoform	EPA 8260	Volatile Organics	NELAP	7/1/2003
Butyl benzyl phthalate	EPA 625	Extractable Organics	NELAP	10/8/2001
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Cadmium	EPA 200.7	Metals	NELAP	10/8/2001
Cadmium	EPA 200.8	Metals	NELAP	10/8/2001
Cadmium	EPA 6010	Metals	NELAP	10/8/2001
Cadmium	EPA 6020	Metals	NELAP	7/1/2003
Cadmium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Calcium	EPA 200.7	Metals	NELAP	10/8/2001
Calcium	EPA 6010	Metals	NELAP	10/8/2001
Calcium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Carbazole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	7/1/2003
Carbon tetrachloride	EPA 624	Volatile Organics	NELAP	10/8/2001
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chemical oxygen demand	EPA 410.1	General Chemistry	NELAP	10/8/2001
Chemical oxygen demand	EPA 410.2	General Chemistry	NELAP	12/23/2005
Chlordane (tech.)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chloride	EPA 300.0	General Chemistry	NELAP	10/8/2001
Chloride	EPA 325.3	General Chemistry	NELAP	10/8/2001
Chlorobenzene	EPA 624	Volatile Organics	NELAP	10/8/2001
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chloroethane	EPA 624	Volatile Organics	NELAP	10/8/2001
Chloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003

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Chloroform	EPA 624	Volatile Organics	NELAP	10/8/2001
Chloroform	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chloroprene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Chlorpyrifos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chromium	EPA 200.7	Metals	NELAP	10/8/2001
Chromium	EPA 200.8	Metals	NELAP	10/8/2001
Chromium	EPA 6010	Metals	NELAP	7/1/2003
Chromium	EPA 6020	Metals	NELAP	7/1/2003
Chromium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Chromium VI	EPA 7195	Metals	NELAP	7/1/2003
Chromium VI	EPA 7196	General Chemistry	NELAP	7/1/2003
Chrysene	EPA 625	Extractable Organics	NELAP	10/8/2001
Chrysene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Chrysene	EPA 8310	Extractable Organics	NELAP	7/1/2003
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
cis-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	10/8/2001
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
cis-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Cobalt	EPA 200.7	Metals	NELAP	10/8/2001
Cobalt	EPA 200.8	Metals	NELAP	10/8/2001
Cobalt	EPA 6010	Metals	NELAP	7/1/2003
Cobalt	EPA 6020	Metals	NELAP	7/1/2003
Cobalt	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Color	EPA 110.2	General Chemistry	NELAP	10/8/2001
Conductivity	EPA 120.1	General Chemistry	NELAP	10/8/2001
Copper	EPA 200.7	Metals	NELAP	10/8/2001
Copper	EPA 200.8	Metals	NELAP	10/8/2001
Copper	EPA 6010	Metals	NELAP	10/8/2001
Copper	EPA 6020	Metals	NELAP	7/1/2003
Copper	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Coumaphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
delta-BHC	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Demeton-o	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Diazinon	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dibenz(a,h) anthracene	EPA 625	Extractable Organics	NELAP	10/8/2001
Dibenz(a,h) anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibenz(a,h) anthracene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibromochloromethane	EPA 624	Volatile Organics	NELAP	10/8/2001
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dibromomethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dichlorovos (DDVP, Dichlorvos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dieldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Diesel range organics (DRO)	CA-LUFT	Extractable Organics	NELAP	7/1/2003
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	7/28/2003
Diesel range organics (DRO)	NWTPH-Dx	Extractable Organics	NELAP	7/1/2003
Diethyl phthalate	EPA 625	Extractable Organics	NELAP	10/8/2001
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dimethyl phthalate	EPA 625	Extractable Organics	NELAP	10/8/2001
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-butyl phthalate	EPA 625	Extractable Organics	NELAP	10/8/2001
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-octyl phthalate	EPA 625	Extractable Organics	NELAP	10/8/2001
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Disulfoton	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Disulfoton	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan I	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan II	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan sulfate	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin aldehyde	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Ethanol	EPA 8015	Volatile Organics	NELAP	7/1/2003
Ethoprop	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Ethylbenzene	EPA 624	Volatile Organics	NELAP	10/8/2001
Ethylbenzene	EPA 8021	Volatile Organics	NELAP	7/1/2003
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ethylene glycol	EPA 8015	Volatile Organics	NELAP	7/1/2003
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Fecal coliforms	SM 9221 E	Microbiology	NELAP	10/8/2001
Fensulfothion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Fenthion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Fluoranthene	EPA 625	Extractable Organics	NELAP	10/8/2001
Fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluoranthene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Fluorene	EPA 625	Extractable Organics	NELAP	10/8/2001
Fluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluorene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Fluoride	EPA 300.0	General Chemistry	NELAP	10/8/2001
Fluoride	EPA 340.2	General Chemistry	NELAP	10/8/2001
Formaldehyde	EPA 8315	Extractable Organics	NELAP	7/1/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	12/23/2005
Gasoline range organics (GRO)	CA-LUFT	Extractable Organics	NELAP	7/1/2003
Gasoline range organics (GRO)	EPA 8015	Volatile Organics	NELAP	7/17/2003
Gasoline range organics (GRO)	NWTPH-Gx	Extractable Organics	NELAP	7/1/2003
Hardness	EPA 130.2	General Chemistry	NELAP	10/8/2001
Heptachlor	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Heptachlor epoxide	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Hexachlorobenzene	EPA 625	Extractable Organics	NELAP	10/8/2001
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorobutadiene	EPA 625	Extractable Organics	NELAP	10/8/2001
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorocyclopentadiene	EPA 625	Extractable Organics	NELAP	10/8/2001
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachloroethane	EPA 625	Extractable Organics	NELAP	10/8/2001
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorophene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Ignitability	EPA 1020	General Chemistry	NELAP	7/1/2003
Indeno(1,2,3-cd)pyrene	EPA 625	Extractable Organics	NELAP	10/8/2001
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Indeno(1,2,3-cd)pyrene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Iron	EPA 200.7	Metals	NELAP	10/8/2001
Iron	EPA 6010	Metals	NELAP	7/1/2003
Iron	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Isodrin	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Isophorone	EPA 625	Extractable Organics	NELAP	10/8/2001
Isophorone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Isosafrole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Kepone	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Kjeldahl nitrogen - total	EPA 351.4	General Chemistry	NELAP	10/8/2001
Lead	EPA 200.7	Metals	NELAP	10/8/2001
Lead	EPA 200.8	Metals	NELAP	10/8/2001
Lead	EPA 200.9	Metals	NELAP	10/8/2001
Lead	EPA 6010	Metals	NELAP	10/8/2001
Lead	EPA 6020	Metals	NELAP	7/1/2003
Lead	EPA 7421	Metals	NELAP	10/8/2001
Lead	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Magnesium	EPA 200.7	Metals	NELAP	10/8/2001
Magnesium	EPA 6010	Metals	NELAP	7/1/2003

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Magnesium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Malathion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Manganese	EPA 200.7	Metals	NELAP	10/8/2001
Manganese	EPA 200.8	Metals	NELAP	10/8/2001
Manganese	EPA 6010	Metals	NELAP	7/1/2003
Manganese	EPA 6020	Metals	NELAP	7/1/2003
Manganese	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Mercury	EPA 1631	Metals	NELAP	10/8/2001
Mercury	EPA 245.1	Metals	NELAP	10/8/2001
Mercury	EPA 7470	Metals	NELAP	10/8/2001
Mercury	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Merphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methanol	NCASI 94.03	Volatile Organics	NELAP	10/8/2001
Methanol	NCASI 99.01	Volatile Organics	NELAP	10/8/2001
Methapyrilene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Methyl bromide (Bromomethane)	EPA 624	Volatile Organics	NELAP	10/8/2001
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl chloride (Chloromethane)	EPA 624	Volatile Organics	NELAP	10/8/2001
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl parathion (Parathion, methyl)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methylene chloride	EPA 624	Volatile Organics	NELAP	10/8/2001
Methylene chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Mevinphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Molybdenum	EPA 200.7	Metals	NELAP	10/8/2001
Molybdenum	EPA 200.8	Metals	NELAP	10/8/2001
Molybdenum	EPA 6010	Metals	NELAP	10/8/2001
Molybdenum	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Naphthalene	EPA 625	Extractable Organics	NELAP	10/8/2001
Naphthalene	EPA 8260	Volatile Organics	NELAP	7/1/2003

Laboratory Scope of Accreditation

Attachment to Certificate #: E87412-05, expiration date June 30, 2007. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87412 EPA Lab Code: WA00035 (360) 577-7222

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Naphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Naphthalene	EPA 8310	Extractable Organics	NELAP	7/1/2003
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Nickel	EPA 200.7	Metals	NELAP	10/8/2001
Nickel	EPA 200.8	Metals	NELAP	10/8/2001
Nickel	EPA 6010	Metals	NELAP	10/8/2001
Nickel	EPA 6020	Metals	NELAP	7/1/2003
Nickel	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Nitrate as N	EPA 300.0	General Chemistry	NELAP	10/8/2001
Nitrate as N	EPA 353.2	General Chemistry	NELAP	7/17/2003
Nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	10/8/2001
Nitrite as N	EPA 300.0	General Chemistry	NELAP	10/8/2001
Nitrite as N	EPA 353.2	General Chemistry	NELAP	7/17/2003
Nitrobenzene	EPA 625	Extractable Organics	NELAP	10/8/2001
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	7/1/2003
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodimethylamine	EPA 625	Extractable Organics	NELAP	10/8/2001
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodi-n-propylamine	EPA 625	Extractable Organics	NELAP	10/8/2001
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiphenylamine	EPA 625	Extractable Organics	NELAP	10/8/2001
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	7/1/2003
Oil & Grease	EPA 1664	General Chemistry	NELAP	10/8/2001
Orthophosphate as P	EPA 365.3	General Chemistry	NELAP	10/8/2001
o-Toluidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Parathion, ethyl	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Parathion, ethyl	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Pentachloronitrobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pentachlorophenol	EPA 1653	Extractable Organics	NELAP	10/8/2001
Pentachlorophenol	EPA 625	Extractable Organics	NELAP	10/8/2001
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
pH	EPA 150.1	General Chemistry	NELAP	10/8/2001
pH	EPA 9040	General Chemistry	NELAP	7/1/2003
Phenacetin	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenanthrene	EPA 625	Extractable Organics	NELAP	10/8/2001
Phenanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenanthrene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Phenol	EPA 625	Extractable Organics	NELAP	10/8/2001
Phenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phorate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Phosphorus, total	EPA 365.3	General Chemistry	NELAP	10/8/2001
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Potassium	EPA 200.7	Metals	NELAP	10/8/2001
Potassium	EPA 6010	Metals	NELAP	10/8/2001
Potassium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Pronamide (Kerb)	EPA 8270	Extractable Organics	NELAP	7/1/2003
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Pyrene	EPA 625	Extractable Organics	NELAP	10/8/2001
Pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pyrene	EPA 8310	Extractable Organics	NELAP	7/1/2003
Pyridine	EPA 8270	Extractable Organics	NELAP	7/1/2003
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	7/1/2003
Residual free chlorine	EPA 330.4	General Chemistry	NELAP	10/8/2001
Residue-filterable (TDS)	EPA 160.1	General Chemistry	NELAP	10/8/2001
Residue-nonfilterable (TSS)	EPA 160.2	General Chemistry	NELAP	10/8/2001
Residue-settleable	EPA 160.5	General Chemistry	NELAP	10/8/2001
Residue-total	EPA 160.3	General Chemistry	NELAP	10/8/2001
Residue-volatile	EPA 160.4	General Chemistry	NELAP	10/8/2001
Ronnel	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Safrole	EPA 8270	Extractable Organics	NELAP	7/1/2003
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Selenium	EPA 200.7	Metals	NELAP	10/8/2001

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Selenium	EPA 200.8	Metals	NELAP	10/8/2001
Selenium	EPA 200.9	Metals	NELAP	10/8/2001
Selenium	EPA 6010	Metals	NELAP	10/8/2001
Selenium	EPA 7740	Metals	NELAP	10/8/2001
Selenium	EPA 7742	Metals	NELAP	7/17/2003
Selenium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Silver	EPA 200.7	Metals	NELAP	10/8/2001
Silver	EPA 200.8	Metals	NELAP	10/8/2001
Silver	EPA 6010	Metals	NELAP	7/1/2003
Silver	EPA 6020	Metals	NELAP	7/1/2003
Silver	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Sodium	EPA 200.7	Metals	NELAP	10/8/2001
Sodium	EPA 6010	Metals	NELAP	7/1/2003
Sodium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Stirofos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Styrene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Sulfate	EPA 300.0	General Chemistry	NELAP	10/8/2001
Sulfide	EPA 376.1	General Chemistry	NELAP	10/8/2001
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Tetrachlorocatechol	EPA 1653	Extractable Organics	NELAP	10/8/2001
Tetrachloroethylene (Perchloroethylene)	EPA 624	Volatile Organics	NELAP	10/8/2001
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Tetrachloroguaiacol	EPA 1653	Extractable Organics	NELAP	10/8/2001
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	7/1/2003
Thallium	EPA 200.7	Metals	NELAP	10/8/2001
Thallium	EPA 200.8	Metals	NELAP	10/8/2001
Thallium	EPA 200.9	Metals	NELAP	10/8/2001
Thallium	EPA 6020	Metals	NELAP	7/1/2003
Thallium	EPA 7841	Metals	NELAP	7/1/2003
Thallium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Thionazin (Zinophos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Tin	EPA 200.7	Metals	NELAP	7/17/2003
Titanium	EPA 200.7	Metals	NELAP	7/17/2003
Tokuthion (Prothiophos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Toluene	EPA 624	Volatile Organics	NELAP	10/8/2001

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Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Toluene	EPA 8021	Volatile Organics	NELAP	7/1/2003
Toluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Total coliforms	SM 9221 B	Microbiology	NELAP	10/8/2001
Total cyanide	EPA 335.4	General Chemistry	NELAP	7/17/2003
Total cyanide	EPA 9012	General Chemistry	NELAP	12/23/2005
Total cyanide	ILM04.1-Exhibit D	General Chemistry	NELAP	7/1/2003
Total hardness as CaCO3	EPA 200.7	Metals	NELAP	10/8/2001
Total organic carbon	EPA 415.1	General Chemistry	NELAP	10/8/2001
Total organic carbon	EPA 9060	General Chemistry	NELAP	7/1/2003
Total organic halides (TOX)	EPA 9020	General Chemistry	NELAP	7/1/2003
Total Petroleum Hydrocarbons (TPH)	EPA 1664	General Chemistry	NELAP	10/8/2001
Total Petroleum Hydrocarbons (TPH)	EPA 8015	Extractable Organics	NELAP	7/1/2003
Total Petroleum Hydrocarbons (TPH)	NWTPH-HCID	Extractable Organics	NELAP	7/1/2003
Total phenolics	EPA 420.1	General Chemistry	NELAP	10/8/2001
Toxaphene (Chlorinated camphene)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
trans-1,2-Dichloroethylene	EPA 624	Volatile Organics	NELAP	10/8/2001
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,3-Dichloropropylene	EPA 624	Volatile Organics	NELAP	10/8/2001
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichloroethene (Trichloroethylene)	EPA 624	Volatile Organics	NELAP	10/8/2001
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	10/8/2001
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichloronate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Trichlorosyringol	EPA 1653	Extractable Organics	NELAP	10/8/2001
Turbidity	EPA 180.1	General Chemistry	NELAP	10/8/2001
Uranium	EPA 200.8	Metals	NELAP	10/8/2001
Vanadium	EPA 200.7	Metals	NELAP	10/8/2001
Vanadium	EPA 200.8	Metals	NELAP	10/8/2001
Vanadium	EPA 6010	Metals	NELAP	7/1/2003
Vanadium	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Vinyl chloride	EPA 624	Volatile Organics	NELAP	10/8/2001
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003

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Columbia Analytical Services, Inc. - WA
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Kelso, WA 98626

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Xylene (total)	EPA 624	Volatile Organics	NELAP	10/8/2001
Xylene (total)	EPA 8021	Volatile Organics	NELAP	7/1/2003
Xylene (total)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Zinc	EPA 200.7	Metals	NELAP	10/8/2001
Zinc	EPA 200.8	Metals	NELAP	10/8/2001
Zinc	EPA 6010	Metals	NELAP	10/8/2001
Zinc	EPA 6020	Metals	NELAP	7/1/2003
Zinc	ILM04.1-Exhibit D	Metals	NELAP	7/1/2003

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Kelso, WA 98626

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2,4,5-Tetrachlorobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8270	Extractable Organics	NELAP	7/17/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	10/8/2001
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	10/8/2001
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,4-Naphthoquinone	EPA 8270	Extractable Organics	NELAP	10/8/2001
1,4-Phenylenediamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
1-Chlorohexane	EPA 8260	Volatile Organics	NELAP	7/17/2003
1-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	7/17/2003
1-Naphthylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001

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Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ 183)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ 187)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,4,5,5'-Hexachlorobiphenyl (BZ 141)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,5,5',6-Hexachlorobiphenyl (BZ 151)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	10/8/2001
2,3,3',4',6-Pentachlorobiphenyl (BZ 110)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,3,4,6-Tetrachlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,3-Dichlorobiphenyl (BZ 5)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,4',5-Trichlorobiphenyl (BZ 31)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	10/8/2001
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	10/8/2001
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	10/8/2001
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	10/8/2001
2-Acetylaminofluorene	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	10/8/2001
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	10/8/2001

Laboratory Scope of Accreditation

Attachment to Certificate #: E87412-05, expiration date June 30, 2007. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87412 EPA Lab Code: WA00035 (360) 577-7222

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Chlorobiphenyl (BZ 1)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	10/8/2001
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	10/8/2001
2-Hexanone	EPA 8260	Volatile Organics	NELAP	10/8/2001
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Nitropropane	EPA 8260	Volatile Organics	NELAP	7/17/2003
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	10/8/2001
2-Picoline (2-Methylpyridine)	EPA 8270	Extractable Organics	NELAP	10/8/2001
2-Sec-butyl-4,6-dinitrophenol (DNBP, Dinoseb)	EPA 8270	Extractable Organics	NELAP	10/8/2001
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	10/8/2001
3,3'-Dimethylbenzidine	EPA 8270	Extractable Organics	NELAP	10/8/2001
3-Methylcholanthrene	EPA 8270	Extractable Organics	NELAP	10/8/2001
3-Methylphenol (m-Cresol)	EPA 8270	Extractable Organics	NELAP	7/17/2003
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	10/8/2001
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	10/8/2001
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	10/8/2001
4-Aminobiphenyl	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	10/8/2001
4-Dimethyl aminoazobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	10/8/2001
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	10/8/2001
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001

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4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	10/8/2001
5-Nitro-o-toluidine	EPA 8270	Extractable Organics	NELAP	10/8/2001
7,12-Dimethylbenz(a) anthracene	EPA 8270	Extractable Organics	NELAP	10/8/2001
a-a-Dimethylphenethylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
Acenaphthene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Acenaphthene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Acenaphthylene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Acetone	EPA 8260	Volatile Organics	NELAP	10/8/2001
Acetonitrile	EPA 8260	Volatile Organics	NELAP	10/8/2001
Acetophenone	EPA 8270	Extractable Organics	NELAP	10/8/2001
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	10/8/2001
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	10/8/2001
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Aluminum	EPA 6010	Metals	NELAP	10/8/2001
Aluminum	EPA 6020	Metals	NELAP	10/8/2001
Aluminum	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Aniline	EPA 8270	Extractable Organics	NELAP	10/8/2001
Anthracene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Anthracene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Antimony	EPA 6010	Metals	NELAP	10/8/2001
Antimony	EPA 6020	Metals	NELAP	10/8/2001
Antimony	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Aramite	EPA 8270	Extractable Organics	NELAP	10/8/2001
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Arsenic	EPA 6010	Metals	NELAP	7/1/2003
Arsenic	EPA 6020	Metals	NELAP	10/8/2001

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Arsenic	EPA 7060	Metals	NELAP	10/8/2001
Arsenic	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Azinphos-methyl (Guthion)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Barium	EPA 6010	Metals	NELAP	10/8/2001
Barium	EPA 6020	Metals	NELAP	10/8/2001
Barium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Benzene	EPA 8021	Volatile Organics	NELAP	10/8/2001
Benzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Benzo(a)anthracene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Benzo(a)pyrene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Benzo(b)fluoranthene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Benzo(g,h,i)perylene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Benzo(k)fluoranthene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Benzoic acid	EPA 8270	Extractable Organics	NELAP	10/8/2001
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	10/8/2001
Beryllium	EPA 6010	Metals	NELAP	10/8/2001
Beryllium	EPA 6020	Metals	NELAP	10/8/2001
Beryllium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
beta-Naphthylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	10/8/2001
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	10/8/2001
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	10/8/2001
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	10/8/2001
Bolstar (Sulprofos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Bromobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Bromoform	EPA 8260	Volatile Organics	NELAP	10/8/2001
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	10/8/2001
Cadmium	EPA 6010	Metals	NELAP	10/8/2001

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Cadmium	EPA 6020	Metals	NELAP	10/8/2001
Cadmium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Calcium	EPA 6010	Metals	NELAP	10/8/2001
Calcium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Carbazole	EPA 8270	Extractable Organics	NELAP	10/8/2001
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	10/8/2001
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	10/8/2001
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Chloride	EPA 300.0	General Chemistry	NELAP	2/17/2006
Chloride	EPA 9056	General Chemistry	NELAP	7/17/2003
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Chlorobenzilate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Chloroethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Chloroform	EPA 8260	Volatile Organics	NELAP	10/8/2001
Chloroprene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Chlorpyrifos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Chromium	EPA 6010	Metals	NELAP	10/8/2001
Chromium	EPA 6020	Metals	NELAP	10/8/2001
Chromium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Chromium VI	EPA 7195	Metals	NELAP	10/8/2001
Chromium VI	EPA 7196	General Chemistry	NELAP	10/8/2001
Chrysene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Chrysene	EPA 8310	Extractable Organics	NELAP	10/8/2001
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	10/8/2001
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	10/8/2001
cis-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Cobalt	EPA 6010	Metals	NELAP	10/8/2001
Cobalt	EPA 6020	Metals	NELAP	10/8/2001
Cobalt	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Copper	EPA 6010	Metals	NELAP	10/8/2001
Copper	EPA 6020	Metals	NELAP	10/8/2001
Copper	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Corrosivity (pH)	EPA 1110	General Chemistry	NELAP	10/8/2001
Coumaphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Demeton-o	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Demeton-s	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Diallate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Diazinon	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dibenz(a,h) anthracene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Dibenz(a,h) anthracene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	10/8/2001
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Dibromomethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dichlorovos (DDVP, Dichlorvos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Diesel range organics (DRO)	CA-LUFT	Extractable Organics	NELAP	10/8/2001
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	7/17/2003
Diesel range organics (DRO)	NWTPH-Dx	Extractable Organics	NELAP	10/8/2001
Diethyl ether	EPA 8260	Volatile Organics	NELAP	7/17/2003
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	10/8/2001
Dimethoate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Dimethoate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	10/8/2001
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	10/8/2001
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	10/8/2001
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Disulfoton	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Disulfoton	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
EPN	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Ethoprop	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Ethyl acetate	EPA 8260	Volatile Organics	NELAP	7/17/2003
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	10/8/2001

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Ethyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	10/8/2001
Ethylbenzene	EPA 8021	Volatile Organics	NELAP	10/8/2001
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Ethylene glycol	EPA 8015	Volatile Organics	NELAP	10/8/2001
Famphur	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Fensulfothion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Fenthion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Fluoranthene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Fluoranthene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Fluorene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Fluorene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Fluoride	EPA 300.0	General Chemistry	NELAP	2/17/2006
Fluoride	EPA 9056	General Chemistry	NELAP	7/17/2003
Formaldehyde	EPA 8315	Extractable Organics	NELAP	10/8/2001
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Gasoline range organics (GRO)	CA-LUFT	Extractable Organics	NELAP	10/8/2001
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	7/17/2003
Gasoline range organics (GRO)	NWTPH-Gx	Extractable Organics	NELAP	10/8/2001
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	10/8/2001
Hexachlorophene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Hexachloropropene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Ignitability	EPA 1020	General Chemistry	NELAP	10/8/2001
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Indeno(1,2,3-cd)pyrene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Iron	EPA 6010	Metals	NELAP	10/8/2001
Iron	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Isobutyl alcohol (2-Methyl-1-propanol)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Isodrin	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001

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Isophorone	EPA 8270	Extractable Organics	NELAP	10/8/2001
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Isosafrole	EPA 8270	Extractable Organics	NELAP	10/8/2001
Kepone	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Lead	EPA 6010	Metals	NELAP	10/8/2001
Lead	EPA 6020	Metals	NELAP	10/8/2001
Lead	EPA 7421	Metals	NELAP	10/8/2001
Lead	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Magnesium	EPA 6010	Metals	NELAP	10/8/2001
Magnesium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Malathion	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Manganese	EPA 6010	Metals	NELAP	10/8/2001
Manganese	EPA 6020	Metals	NELAP	10/8/2001
Manganese	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
MCPA	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
MCPP	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Mercury	EPA 7470	Metals	NELAP	10/8/2001
Mercury	EPA 7471	Metals	NELAP	10/8/2001
Mercury	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Merphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	10/8/2001
Methapyrilene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	10/8/2001
Methyl methanesulfonate	EPA 8270	Extractable Organics	NELAP	7/17/2003
Methyl parathion (Parathion, methyl)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Methyl parathion (Parathion, methyl)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Methylene chloride	EPA 8260	Volatile Organics	NELAP	10/8/2001
Mevinphos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Molybdenum	EPA 6010	Metals	NELAP	10/8/2001
Molybdenum	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Naphthalene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Naphthalene	EPA 8270	Extractable Organics	NELAP	10/8/2001

Laboratory Scope of Accreditation

Attachment to Certificate #: E87412-05, expiration date June 30, 2007. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87412 EPA Lab Code: WA00035 (360) 577-7222

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Naphthalene	EPA 8310	Extractable Organics	NELAP	10/8/2001
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Nickel	EPA 6010	Metals	NELAP	10/8/2001
Nickel	EPA 6020	Metals	NELAP	10/8/2001
Nickel	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Nitrate	EPA 9056	General Chemistry	NELAP	7/17/2003
Nitrite	EPA 9056	General Chemistry	NELAP	7/17/2003
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	10/8/2001
Nitroglycerin	EPA 8332	Extractable Organics	NELAP	7/17/2003
Nitroquinoline-1-oxide	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosodiethylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitroso-di-n-butylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosomethylethylamine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosomorpholine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosopiperidine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Nitrosopyrrolidine	EPA 8270	Extractable Organics	NELAP	10/8/2001
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
o,o,o-Triethyl phosphorothioate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	10/8/2001
Oil & Grease	EPA 1664	General Chemistry	NELAP	10/8/2001
Oil & Grease	EPA 9071	General Chemistry	NELAP	10/8/2001
o-Toluidine	EPA 8270	Extractable Organics	NELAP	10/8/2001
Parathion, ethyl	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Parathion, ethyl	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
p-Dioxane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Pentachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/17/2003
Pentachloronitrobenzene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
pH	EPA 9040	General Chemistry	NELAP	10/8/2001
pH	EPA 9045	General Chemistry	NELAP	7/17/2003
Phenacetin	EPA 8270	Extractable Organics	NELAP	10/8/2001
Phenanthrene	EPA 8270	Extractable Organics	NELAP	10/8/2001

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Phenanthrene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Phenol	EPA 8270	Extractable Organics	NELAP	10/8/2001
Phorate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Phorate	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Potassium	EPA 6010	Metals	NELAP	10/8/2001
Potassium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Pronamide (Kerb)	EPA 8270	Extractable Organics	NELAP	10/8/2001
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Pyrene	EPA 8270	Extractable Organics	NELAP	10/8/2001
Pyrene	EPA 8310	Extractable Organics	NELAP	10/8/2001
Pyridine	EPA 8270	Extractable Organics	NELAP	10/8/2001
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	10/8/2001
Ronnel	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Safrole	EPA 8270	Extractable Organics	NELAP	10/8/2001
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Selenium	EPA 6010	Metals	NELAP	7/17/2003
Selenium	EPA 7740	Metals	NELAP	10/8/2001
Selenium	EPA 7742	Metals	NELAP	7/17/2003
Selenium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Silver	EPA 6010	Metals	NELAP	10/8/2001
Silver	EPA 6020	Metals	NELAP	10/8/2001
Silver	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Sodium	EPA 6010	Metals	NELAP	10/8/2001
Sodium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Stirofos	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Styrene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Sulfate	EPA 300.0	General Chemistry	NELAP	2/17/2006
Sulfate	EPA 9056	General Chemistry	NELAP	7/17/2003
Sulfide	EPA 9030/9034	General Chemistry	NELAP	7/17/2003
Sulfotepp	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	7/17/2003
Synthetic Precipitation Leaching Procedure	EPA 1312	General Chemistry	NELAP	7/17/2003
tert-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	7/17/2003
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	10/8/2001

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Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	10/8/2001
Thallium	EPA 6010	Metals	NELAP	7/17/2003
Thallium	EPA 6020	Metals	NELAP	10/8/2001
Thallium	EPA 7841	Metals	NELAP	10/8/2001
Thallium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Thionazin (Zinophos)	EPA 8270	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Tokuthion (Prothiophos)	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Toluene	EPA 8021	Volatile Organics	NELAP	10/8/2001
Toluene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Total cyanide	EPA 9012	General Chemistry	NELAP	12/23/2005
Total cyanide	ILM04.1-Exhibit D	General Chemistry	NELAP	10/8/2001
Total organic carbon	EPA 9060	General Chemistry	NELAP	10/8/2001
Total organic halides (TOX)	EPA 9020	General Chemistry	NELAP	10/8/2001
Total Petroleum Hydrocarbons (TPH)	NWTPH-HCID	Extractable Organics	NELAP	10/8/2001
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Toxicity Characteristic Leaching Procedure	EPA 1311	General Chemistry	NELAP	10/8/2001
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	10/8/2001
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	10/8/2001
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	10/8/2001
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	10/8/2001
Trichloronate	EPA 8141	Pesticides-Herbicides-PCB's	NELAP	10/8/2001
Vanadium	EPA 6010	Metals	NELAP	10/8/2001
Vanadium	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	10/8/2001
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	10/8/2001
Xylene (total)	EPA 8021	Volatile Organics	NELAP	10/8/2001
Xylene (total)	EPA 8260	Volatile Organics	NELAP	10/8/2001
Zinc	EPA 6010	Metals	NELAP	10/8/2001
Zinc	EPA 6020	Metals	NELAP	10/8/2001
Zinc	ILM04.1-Exhibit D	Metals	NELAP	10/8/2001

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E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3,5-Trinitrobenzene (1,3,5-TNB)	EPA 8330	Extractable Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3-Dinitrobenzene (1,3-DNB)	EPA 8330	Extractable Organics	NELAP	7/1/2003
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ 180)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ 183)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,4',5'-Hexachlorobiphenyl (BZ 138)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ 187)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,5,5'-Hexachlorobiphenyl (BZ 141)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,4,5'-Pentachlorobiphenyl (BZ 87)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,5,5',6-Hexachlorobiphenyl (BZ 151)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',3,5'-Tetrachlorobiphenyl (BZ 44)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',4,4',5,5'-Hexachlorobiphenyl (BZ 153)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',4,5,5'-Pentachlorobiphenyl (BZ 101)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',5,5'-Tetrachlorobiphenyl (BZ 52)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,2',5-Trichlorobiphenyl (BZ 18)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3,3',4',6-Pentachlorobiphenyl (BZ 110)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3',4,4'-Tetrachlorobiphenyl (BZ 66)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,3-Dichlorobiphenyl (BZ 5)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4',5-Trichlorobiphenyl (BZ 31)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trinitrotoluene (2,4,6-TNT)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dinitrotoluene (2,6-DNT)	EPA 8330	Extractable Organics	NELAP	7/1/2003

Laboratory Scope of Accreditation

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E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Amino-4,6-dinitrotoluene (2-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/1/2003
2-Chlorobiphenyl (BZ 1)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/1/2003
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
3-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/1/2003
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
4-Amino-2,6-dinitrotoluene (4-am-dnt)	EPA 8330	Extractable Organics	NELAP	7/1/2003
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Methylphenol (p-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitrotoluene	EPA 8330	Extractable Organics	NELAP	7/1/2003
Acenaphthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aluminum	EPA 6010	Metals	NELAP	7/1/2003
Aluminum	EPA 6020	Metals	NELAP	7/1/2003
Aniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
Anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Antimony	EPA 6010	Metals	NELAP	7/1/2003
Antimony	EPA 6020	Metals	NELAP	7/1/2003

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Analyte	Method/Tech	Category	Certification Type	Effective Date
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Arsenic	EPA 6010	Metals	NELAP	7/1/2003
Arsenic	EPA 6020	Metals	NELAP	7/1/2003
Arsenic	EPA 7060	Metals	NELAP	7/1/2003
Barium	EPA 6010	Metals	NELAP	7/1/2003
Barium	EPA 6020	Metals	NELAP	7/1/2003
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzoic acid	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Beryllium	EPA 6010	Metals	NELAP	7/1/2003
Beryllium	EPA 6020	Metals	NELAP	7/1/2003
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	7/1/2003
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Cadmium	EPA 6010	Metals	NELAP	7/1/2003
Cadmium	EPA 6020	Metals	NELAP	7/1/2003
Carbazole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chromium	EPA 6010	Metals	NELAP	7/1/2003
Chromium	EPA 6020	Metals	NELAP	7/1/2003
Chromium VI	EPA 7196	Metals	NELAP	7/1/2003
Chrysene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Cobalt	EPA 6010	Metals	NELAP	7/1/2003

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Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
Cobalt	EPA 6020	Metals	NELAP	7/1/2003
Copper	EPA 6010	Metals	NELAP	7/1/2003
Copper	EPA 6020	Metals	NELAP	7/1/2003
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dibenz(a,h) anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	7/1/2003
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Iron	EPA 6010	Metals	NELAP	7/1/2003
Isophorone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Lead	EPA 6010	Metals	NELAP	7/1/2003
Lead	EPA 6020	Metals	NELAP	7/1/2003
Lead	EPA 7421	Metals	NELAP	7/1/2003
Manganese	EPA 6010	Metals	NELAP	7/1/2003
Manganese	EPA 6020	Metals	NELAP	7/1/2003
Mercury	EPA 7471	Metals	NELAP	7/1/2003

Laboratory Scope of Accreditation

Attachment to Certificate #: E87412-05, expiration date June 30, 2007. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87412 EPA Lab Code: WA00035 (360) 577-7222

E87412
Columbia Analytical Services, Inc. - WA
1317 South 13th Avenue
Kelso, WA 98626

Matrix: Biological Tissue

Analyte	Method/Tech	Category	Certification Type	Effective Date
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Molybdenum	EPA 6010	Metals	NELAP	7/1/2003
Naphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Nickel	EPA 6010	Metals	NELAP	7/1/2003
Nickel	EPA 6020	Metals	NELAP	7/1/2003
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Nitrobenzene	EPA 8330	Extractable Organics	NELAP	7/1/2003
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	EPA 8330	Extractable Organics	NELAP	7/1/2003
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pyridine	EPA 8270	Extractable Organics	NELAP	7/1/2003
RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)	EPA 8330	Extractable Organics	NELAP	7/1/2003
Selenium	EPA 6010	Metals	NELAP	7/1/2003
Selenium	EPA 7740	Metals	NELAP	7/1/2003
Selenium	EPA 7742	Metals	NELAP	7/1/2003
Silver	EPA 6010	Metals	NELAP	7/1/2003
Silver	EPA 6020	Metals	NELAP	7/1/2003
Tetryl (methyl-2,4,6-trinitrophenylnitramine)	EPA 8330	Extractable Organics	NELAP	7/1/2003
Thallium	EPA 6020	Metals	NELAP	7/1/2003
Thallium	EPA 7841	Metals	NELAP	7/1/2003
Total cyanide	EPA 9012	General Chemistry	NELAP	12/23/2005
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Vanadium	EPA 6010	Metals	NELAP	7/1/2003
Zinc	EPA 6010	Metals	NELAP	7/1/2003
Zinc	EPA 6020	Metals	NELAP	7/1/2003

APPENDIX F

ADDITIONAL AGENCY-SPECIFIC DOCUMENTS